

# OPERATOR'S MANUAL

## V-2000 Photometer

(with 45+ factory calibrations)



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# Chapter 1

## Introduction

### 1-1 Battery Installation

The V-2000 requires 4 AA alkaline or lithium batteries. With alkaline batteries, the expected life is 2,500 hours of operation. With lithium batteries, the expected life is 10,000 hours of operation. The V-2000 has a battery saving auto-shutoff feature that turns the instrument off after 20 minutes of non-use. Remove batteries when photometer is not in use.

To install batteries, carefully loosen the 2 captive screws on the battery cover (back of instrument). Remove the cover, and insert the batteries with the correct orientation as illustrated in the battery compartment. Replace the battery cover and replace the captive screws.

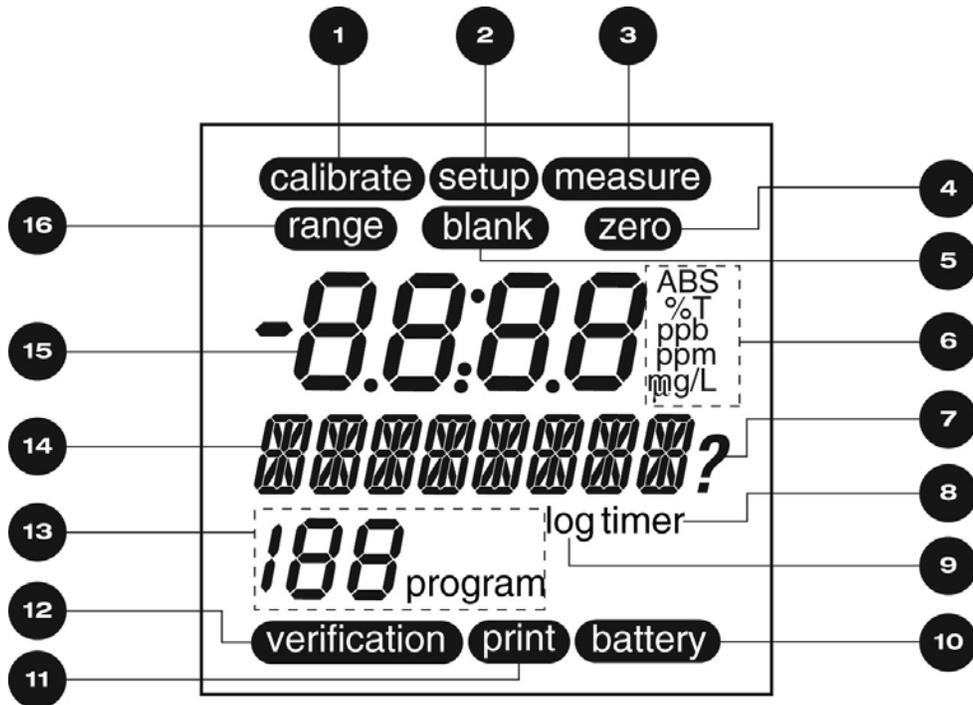
### 1-2 Sample Cell Adapters

The V-2000 comes with 2 sample cell adapters: 13 mm adapter for Vacu-vials; 16 mm adapter for COD Vials. The adapters ensure correct alignment of the ampoule or vial being used. If the ampoule or vial is not aligned correctly (i.e. wrong adapter or no adapter is used), measurement errors will occur.

To insert an adapter, the male tabs on the adapter (left and right sides) should be matched up with the alignment slots to the left and right of the sample cell compartment. Insert the adapter with the correct alignment and push down firmly until it snaps into place.

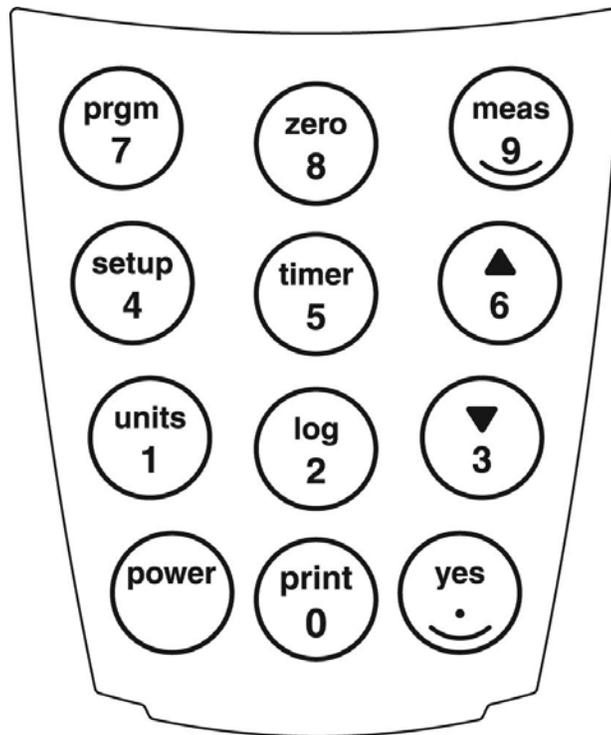
**Important Note:** Above the sample cell compartment, the instrument says “**cuvette adapter ◀ lock-unlock ▶**”. Disregard this message! There is no lock, unlock feature to the current cell adapters. They go straight in and out. DO NOT attempt to turn the adapter from left to right when inserting it or removing it.

## 1-3 LCD Display



- |    |                     |   |
|----|---------------------|---|
| 1  | <b>calibrate</b>    | Displays when the instrument is in the calibration mode           |
| 2  | <b>setup</b>        | Displays when the instrument is in the setup mode                 |
| 3  | <b>measure</b>      | Displays when the instrument is taking a measurement              |
| 4  | <b>zero</b>         | Displays when the instrument is zeroing                           |
| 5  | <b>blank</b>        | Displays when a blank is active for a selected method             |
| 6  | <b>units</b>        | Displays the chosen unit of measure                               |
| 7  | <b>?</b>            | Displays when the instrument is prompting the user for a response |
| 8  | <b>timer</b>        | Displays when the timer is active                                 |
| 9  | <b>log</b>          | Displays if data is stored in the log                             |
| 10 | <b>battery</b>      | Displays if battery is low  |
| 11 | <b>print</b>        | Displays when the instrument is printing (or downloading)         |
| 12 | <b>verification</b> | Displays when the instrument is in the verification mode          |
| 13 | <b>XXX program</b>  | Displays the active program number                                |
| 14 | <b>⊗⊗⊗⊗⊗⊗</b>       | Alphanumeric display  |
| 15 | <b>-8.8:8.8</b>     | Numeric display   |
| 16 | <b>range</b>        | Displays to indicate over range condition                         |

## 1-4 Keypad



### KEY

### FUNCTION

---

<b>power</b>	Turns the instrument on or off
<b>print / 0</b>	Initiates print mode or inputs a 0
<b>yes / .</b>	Confirms a selection, functions as Enter key, or inputs a decimal point
<b>units / 1</b>	Allows selection of unit of measure or inputs a 1
<b>log / 2</b>	Initiates data log mode or inputs a 2
<b>▼ / 3</b>	Scroll down or inputs a 3
<b>setup / 4</b>	Allows access to user selectable functions (Blank, Verify, Digits, Clock, Set Baud, Print, AutoPrint, Auto Log, Self-Test, Upload, User Prgm, Cal User) or inputs a 4
<b>timer / 5</b>	Initiates timer, allows access to time/date and stopwatch or inputs a 5
<b>▲ / 6</b>	Scroll up or inputs a 6
<b>prgm / 7</b>	Initiates program number input or inputs a 7
<b>zero / 8</b>	Initiates a zero measurement or inputs an 8
<b>meas / 9</b>	Initiates a measurement or inputs a 9

### 1-5 List of Direct Read Programs (Factory Calibrations)

Analyte	Prog. #	Kit Cat. #	Instrument Display	Cell Size, mm	Blank Y/N	Wave-length	Range ppm	Method
Aluminum	9	K-0603	ALUMINUM	13	Y	520	0 - 0.25	Eriochrome Cyanine R
Ammonia	12	K-1413	AMMONIA	13	N	610	0 - 3.00 (as N)	Hydroxybenzyl Alcohol
Ammonia	13	K-1413	AMMONIA	13	N	610	0 - 60.0 (as N)	Hydroxybenzyl Alcohol
Ammonia	15	K-1503	AMMONIA	13	N	420	0 - 7.00 (as N)	Direct Nesslerization
Ammonia	16	K-1523	AMMONIA	13	N	420	0 - 14.0 (as N)	Direct Nesslerization
Chloride	26	K-2103	CHLORIDE	13	Y	420	0 - 40.0	Ferric Thiocyanate
Chlorine	32	K-2513	CHLORINE	13	N	520	0 - 5.00	DPD
Chlorine	32	K-2523	CHLORINE	13	N	520	0 - 5.00	DPD
Chlorine Dioxide	37	K-2703	CLORDIOX	13	N	520	0 - 11.0	DPD
Chromate	42	K-2803	CHROMATE	13	N	520	0 - 3.50 (as CrO <sub>4</sub> )	Diphenylcarbazide
Copper	55	K-3503	COPPER	13	N	420	0 - 12.00	Bathocuproine
Cyanide	60	K-3803	CYANIDE	13	Y	610	0 - 0.400	Isonicotinic/Barbituric Acid
DEHA	64	K-3903	DEHA	13	N	580	0 - 2.00	PDTS
Hydrazine	89	K-5003	HYDRAZ	13	Y	420	0 - 1.20	PDMAB
Peroxide	93	K-5513	PEROXIDE	13	N	520	0 - 3.00	DPD
Peroxide	95	K-5543	PEROXIDE	13	N	520	0 - 6.00	Ferric Thiocyanate
Iron	100	K-6003	IRON	13	N	520	0 - 6.00	Phenanthroline
Iron	101	K-6013	IRON	13	N	420	0 - 25.0	Phenanthroline
Iron	102	K-6023	IRON	13	N	580	0 - 2.50	PDTS
Iron	100	K-6203	IRON	13	N	520	0 - 6.00	Phenanthroline

Analyte	Prog. #	Kit Cat. #	Instrument Display	Cell Size, mm	Blank Y/N	Wave-length	Range ppm	Method
Manganese	110	K-6503	MN	13	N	520	0 - 30.0	Periodate
Molybdate	115	K-6703	MOLYB	13	N	420	0 - 25.0 (as Mo)	Catechol
Nitrate	119	K-6903	NITRATE	13	N	520	0 - 1.50 (as N)	Cadmium Reduction
Nitrate	122	K-6913	NITRATE	13	N	520	0 - 1.50 (as N)	Zinc Reduction
Nitrate	120	K-6923	NITRATE	13	N	520	0 - 7.5 (as N)	Cadmium Reduction
Nitrate	121	K-6933	NITRATE	13	N	520	0 - 50.0 (as NO <sub>3</sub> )	Cadmium Reduction
Nitrite	125	K-7003	NITRITE	13	N	520	0 - 1.00 (as N)	Azo Dye Formation
COD LR, EPA Accepted	48	K-7350S K-7355	COD LR	16	Y	420	0 - 150	Dichromate Reactor Digestion
COD LR, Mercury Free	48	K-7351S K-7356	COD LR	16	Y	420	0 - 150	Dichromate Reactor Digestion
COD HR, EPA Accepted	49	K-7360S K-7365	COD HR	16	Y	610	0 - 1500	Dichromate Reactor Digestion
COD HR, Mercury Free	49	K-7361S K-7366	COD HR	16	Y	610	0 - 1500	Dichromate Reactor Digestion
COD HR+, Not EPA Accepted	49	K-7370S K-7375	COD HR	16	Y	610	0 - 15,000	Dichromate Reactor Digestion
COD HR+, Mercury Free	49	K-7371S K-7376	COD HR	16	Y	610	0 - 15,000	Dichromate Reactor Digestion
Ozone	133	K-7423	OZONE	13	N	520	0 - 5.00	DPD
Oxygen	140	K-7503	OXYGEN	13	N	610	0 - 2.00	Indigo Carmine
Oxygen	141	K-7513	OXYGEN	13	N	520	0 - 15.0	Indigo Carmine
Oxygen	142	K-7553	OXYGEN	13	N	520	0 - 1.000	Rhodazine D™
Peracetic Acid	148	K-7913	PERACET	13	N	520	0 - 5.00	DPD
Phenols	152	K-8003	PHENOLS	13	N	520	0 - 8.00	4-Aminoantipyrine
Phenols	153	K-8023	PHENOLS	13	N	520	0 - 20.0	4-Aminoantipyrine
Phosphate	158	K-8503	PHOS PO4	13	N	420	0 - 80.0 (as PO <sub>4</sub> )	Vanadomolybdo-phosphoric Acid
Phosphate	159	K-8513	PHOS PO4	13	N	610	0 - 8.00 (as PO <sub>4</sub> )	Stannous Chloride

Analyte	Prog. #	Kit Cat. #	Instrument Display	Cell Size, mm	Blank Y/N	Wave-length	Range ppm	Method
Phosphate	160	K-8513	PHOS P	13	N	610	0 - 2.64 (as P)	Stannous Chloride
Silica	168	K-9003	SILICA	13	N	610	0 - 10.00 (as SiO <sub>2</sub> )	Heteropoly Blue
Sulfate	174	K-9203	SULFATE	13	N	420	0 - 100.0	Turbidimetric
Sulfide	179	K-9503	SULFIDE	13	N	610	0 - 3.00	Methylene Blue
Sulfide	180	K-9523	SULFIDE	13	N	610	0 - 6.00	Methylene Blue
Zinc	187	K-9903	ZINC	13	Y	610	0 - 3.00	Zincon
Zinc	188	K-9923	ZINC	13	Y	610	0 - 15.0	Zincon

# Chapter 2

## Setup Menu Functions

To view the Setup menu functions, press the **setup** key and use the ▲ or ▼ keys to scroll through the list of functions.

### 2-1 Blank

The blank function is employed for specific methods that require the generation and use of a reagent blank ampoule or vial. See table in section 1-5 and the product specific instructions for methods that require the use of a reagent blank.

The procedure for using the blank function is found in Chapter 3, Section 3-3 Procedure B - Zeroing, Program Selection, Setting Reagent Blank and Measuring. Detailed directions for generating a reagent blank are in the product specific instructions.

- The “blank” icon is displayed in the upper center of the LCD display (see Chapter 1, Section 1-3) whenever a blank value is stored and active for a particular program.
- If a blank value has accidentally been stored for a method that does not employ a reagent blank (blank icon is displayed), it is important to clear the blank before running the method.

To Clear Blank:

1. Press the **setup** key and use the ▲ or ▼ keys until “BLANK” is displayed, then press the **yes** key.
2. “SET BLNK?” will be displayed.
3. Use the ▲ or ▼ keys to toggle from “SET BLNK?” to “CLR BLNK”. When “CLR BLNK” is displayed, press the **yes** key.
4. “CLEARED” will be displayed.
5. Press the **meas** key to exit the setup menu.

- The V-2000 stores the last reagent blank set, even when the unit powers off.

### 2-2 Verify

Disregard this function. It offers no added value to the instrument user.

### 2-3 Digits

The digits function allows the user to select the resolution of the reading (the number of significant digits in the displayed test result): 0.000, 0.00, 0.0, and 0. The default setting for this function is 0.000, however the test range on the parameter specific test kit instructions and in the List of Supported Parameters in Chapter 9 of this manual is displayed with the suggested number of significant digits for each particular method.

**NOTE:** The digits resolution setting on the V-2000 is not method specific, and therefore must be reset as needed when moving from one preprogrammed method to another.

1. Press the **setup** key and use the ▲ or ▼ keys until “DIGITS” is displayed, then press the **yes** key.
2. Use the ▲ or ▼ keys until the desired resolution is displayed, then press the **yes** key.
3. Press the **meas** key to exit the setup menu.

## 2-4 Clock

The clock function allows the user to set the time and date.

1. Press the **setup** key and use the ▲ or ▼ keys until “CLOCK” is displayed, then press the **yes** key.
2. “20\_\_” will be displayed. Enter the year.
3. “\_\_Month” will be displayed. Enter the month.
4. “\_\_Day” will be displayed. Enter the day.
5. “\_\_:\_\_ (24) hour” will be displayed. Enter the time.
6. Press the **meas** key to exit the setup menu.

## 2-5 Set Baud

The set baud function allows the user to select the baud rate:1200, 2400, 4800, and 9600. The default setting for this function is 1200.

1. Press the **setup** key and use the ▲ or ▼ keys until “SET BAUD” is displayed, then press the **yes** key.
2. Use the ▲ or ▼ keys until the desired baud rate is displayed, then press the **yes** key.
3. Press the **meas** key to exit the setup menu.

## 2-6 Print

The print function allows the user to select the printout format. The user can select between a standard printout (used if printing directly to a printer) and a comma delimited format (used for importing data into a spreadsheet).

1. Press the **setup** key and use the ▲ or ▼ keys until “PRINT” is displayed, then press the **yes** key.
2. Use the ▲ or ▼ keys to toggle between “STND PRN” and “CMA DELM”. When the desired printout format is displayed, press the **yes** key.
3. Press the **meas** key to exit the setup menu.

## 2-7 Auto Print

The auto print function allows the user to automatically send readings to a printer.

1. Press the **setup** key and use the ▲ or ▼ keys until “AUTO PRT” is displayed, then press the **yes** key.
2. Use the ▲ or ▼ keys to toggle between “AUTO OFF” and “AUTO ON”. When the desired printing function is displayed, press the **yes** key.
3. Press the **meas** key to exit the setup menu.

## 2-8 Auto Log

The auto log function allows the user to automatically save **up to** 100 test results in the photometer’s memory. The procedure for using the auto log function is found in Chapter 4, Section 4-2 Logging Data Automatically.

## 2-9 Self-Test

The self-test function puts the photometer into self-diagnostic mode. It is not recommended as a routine test, but rather as a troubleshooting tool to be used if the unit gives an error message, produces a suspect test result or in any way malfunctions. If an instrument fails any portion of the self-test, contact CHEMetrics Technical Services Department.

1. Before initiating the self-test, the sample cell adapter must be removed and the light shield must be in place.
2. Press the **setup** key and use the ▲ or ▼ keys until “SELFTEST” is displayed, then press the **yes** key.
3. The instrument will display “KBD TEST”. Then it will prompt the user to press each of the keys beginning with the **7** key.
4. When the test is complete, the instrument should display “UNIT OK”.
5. Press any key to view the complete LCD display capabilities. Compare the instrument display to the display graphic in Chapter 1, Section 1-3 LCD Display.
6. Press any key to exit the self-test.
7. Press the **meas** key to exit the setup menu.

## 2-10 Upload

The upload function allows the user to update the V-2000 to the most current method version. The V-2000 is updated by logging on to the CHEMetrics website, [www.chemetrics.com](http://www.chemetrics.com) and clicking on “V-2000 New Method Update” under the Support tab. To ensure that the most current program calibrations are being used, it is recommended that every V-2000 user check the CHEMetrics website frequently. For more information, see Chapter 5, Section 5-3 Uploading Method Revisions.

# Chapter 3

## Setup and Measurement Procedures

### 3-1 General Operating Instructions

The following guidelines can be considered general operating instructions for the V-2000 photometer. More parameter specific information is provided in the kit instructions supplied with each Vacu-vial or COD test kit.

- The V-2000 must be zeroed prior to each series of measurements. Additionally, if the ampoule/vial adapter has been removed or comes loose during operation, the V-2000 should be re-zeroed.
- For best results, always cover the ampoule or vial with the light shield prior to zeroing the instrument, setting a reagent blank value or measuring a sample.
- For COD testing, LED based photometers do not produce accuracy, precision and sensitivity equivalent to that attainable from spectrophotometers. For NPDES reporting purposes for COD, a spectrophotometer is the preferred method of measurement.
- Range and accuracy claims for CHEMetrics instrumental products on different instrument platforms are available on the CHEMetrics website.
- A 13 mm sealed ampoule containing distilled water is supplied in each Vacu-vials Test Kit for use as a ZERO vial for Vacu-vial test procedures.
- A 16 mm screw cap vial containing distilled water is supplied with the V-2000 Photometer for use as a zeroing vial for the COD test procedure.
- Methods that do not specifically call for the generation of a reagent blank should follow **Procedure A - Zeroing, Program Selection and Measuring** below.
- Methods that do specifically call for the generation of a reagent blank should follow **Procedure B - Zeroing, Program Selection, Setting Reagent Blank and Measuring** below.

### 3-2 Procedure A - Zeroing, Program Selection and Measuring

1. Install the appropriate sample cell adapter into the photometer.
2. Turn the photometer on by pressing the **power** key.

3. Insert the ZERO vial into the V-2000, cover the ZERO vial with the light shield, and press the **zero** key. "Wait" is displayed, then the result is displayed as "0.000".
4. Press the **prgm** key, enter the appropriate program number, then press the **yes** key. The instrument will display the appropriate method name and program number.  
**NOTE:** If the program number is a 3 digit number, the **yes** key does not have to be pressed.
5. Follow the parameter specific Test Procedure supplied with the Test Kit on the sample to be tested and insert the resulting test vial into the V-2000. Cover the test vial with the light shield.
6. Press **meas** key. If a color development wait time is specified in the parameter specific Test Procedure, the V-2000 timer will begin to countdown. The V-2000 will automatically proceed to the measure mode when wait time is complete. The instrument will read the test vial and display the test result. The test result can either be recorded manually, logged manually or logged automatically. The instrument displays under and over-range results only very briefly. Under and over-range results cannot be logged.  
**NOTE:** To bypass the timer feature, simply press the **timer** key. The instrument will immediately initiate measurement mode.

### 3-3 Procedure B - Zeroing, Program Selection, Setting Reagent Blank and Measuring

1. Install the appropriate sample cell adapter into the photometer.
2. Turn the photometer on by pressing the **power** key.
3. Insert the zeroing vial into the V-2000, cover the zeroing vial with the light shield, and press the **zero** key. "Wait" is displayed, then the result is displayed as "0.000".
4. Press the **prgm** key, enter the appropriate program number, then press the **yes** key. The instrument will display the appropriate method name and program number.  
**NOTE:** If the program number is a 3 digit number, the **yes** key does not have to be pressed.
5. Press the **setup** key and use the **▲** or **▼** keys until "BLANK" is displayed, then press the **yes** key.
6. "SET BLNK?" will be displayed, press the **yes** key.
7. "SAMPLE?" will be displayed.
8. Follow the parameter specific instructions supplied with the test kit for Generating a Reagent Blank. Insert the Reagent Blank into the V-2000. Cover the Reagent Blank with the light shield, then press the **yes** key. The instrument will read the blank, display an absorbance value momentarily, and then move to the next setup function.
9. Press the **meas** key to exit the setup menu. The instrument display will return to the appropriate method name and program number.
10. Follow the parameter specific Test Procedure supplied with the test kit on the sample to be tested, and insert the resulting test ampoule or vial into the V-2000. Cover the test ampoule with the light shield.
11. Press the **meas** key. If a color development wait time is specified in the parameter specific Test Procedure, the V-2000 timer will begin to countdown. The V-2000 will automatically proceed to the measure mode when wait time is complete. The instrument will read the test ampoule or vial and display the test result. The test result can either be recorded manually, logged manually or logged automatically. The instrument displays under and over-range results only very briefly. Under and over-range results cannot be logged.  
**NOTE:** To bypass the timer feature, simply press the **timer** key. The instrument will immediately initiate measurement mode.

# Chapter 4

## Data Logging

The V-2000 allows the user to store **up to** 100 test results in the photometer's memory.

### 4-1 Logging Data Manually

When a test result is being displayed by the instrument, press the **log** key. The log icon will appear in the display. The V-2000 will display "1 LOGGED". As new test results are logged, the number preceding LOGGED will append to reflect the number of saved test results.

### 4-2 Logging Data Automatically

Auto log automatically saves test results to the photometer's memory.

1. Press the **setup** key and use the ▲ or ▼ keys until "AUTO LOG" is displayed, then press the **yes** key.
2. Use the ▲ or ▼ keys to toggle between "AUTO OFF" and "AUTO ON". When the desired function is displayed, press the **yes** key.
3. Press the **meas** key to exit the setup menu.
4. With auto log turned on, the log icon will appear in the display. After the first data point is logged, the V-2000 will display "1 LOGGED". As new test results are logged, the number preceding LOGGED will append to reflect the number of saved test results.

**NOTE:** Under and over-range results are not logged.

### 4-3 Displaying the Log

1. Press and hold the **log** key for approximately 4 seconds.
2. "DISPLAY" will be displayed and the last test result saved into the log will be displayed.
3. Use ▲ or the ▼ keys to scroll through the log test results.
4. Press the **meas** key to escape log display.

### 4-4 Clearing the Log

1. Press and hold the **log** key for approximately 4 seconds.
2. Press the **zero** key; "CLR LOG?" will be displayed.  
**NOTE:** It is recommended to print or download the log prior to clearing.
3. Press the **yes** key to clear the log. "DELETED" will be displayed and the V-2000 will return to the measurement mode.

# Chapter 5

## Use with Printers and Computers

The V-2000 photometer allows communication directly to a printer or bidirectional communication with a computer.

Connecting to a printer or a computer requires the use of the supplied RS-232 serial cable. This cable has a special 3-pin connector on one end for the V-2000. The RS-232 port is on the underside of the instrument. When connecting the RS-232 cable to a computer, a serial connector may be required (a 25 pin to 9 pin adapter is included with the V-2000). If your computer does not have a serial port, you will need to purchase a separate adapter that will allow you to connect the V-2000 RS-232 serial cable to the USB port on your computer. See Section 9-1 for ordering information.

### 5-1 Printing Logged Data Directly to a Printer

1. With the V-2000 power off, connect the V-2000 to a printer using the supplied RS-232 serial cable.
2. Press the **power** key.
3. Press the **setup** key and use the ▲ or ▼ keys until "PRINT" is displayed, then press the **yes** key.
4. Use the ▲ or ▼ keys to toggle between "STND PRN" and "CMA DELM". When "STND PRN" is displayed, press the **yes** key.
5. Press the **meas** key to exit the setup menu.
6. Press and hold the **log** key for approximately 4 seconds.
7. Press and hold the **print** key for approximately 4 seconds to download all saved data.

### 5-2 Downloading Logged Data to a Computer

Logged data can be sent to a terminal emulator program and then exported to a spreadsheet or database software application. The instructions below are specific to the terminal emulator program HyperTerminal.

1. Create a text file (\*.txt) to receive the V-2000 logged data. Close this file.
2. With the V-2000 power off, connect the V-2000 to a computer using the supplied RS-232 serial cable.
3. Press the **power** key.
4. Press the **setup** key and use the ▲ or ▼ keys until "PRINT" is displayed, then press the **yes** key.
5. Use the ▲ or ▼ keys to toggle between "STND PRN" and "CMA DELM". When "CMA DELM" is displayed, press the **yes** key.
6. Press the **meas** key to exit the setup menu.
7. Open HyperTerminal by going to the Start button, then clicking on Programs/Accessories/Communications/HyperTerminal.
8. A "New Connection" window will open. Enter a name to call the terminal program, for example, "V2000". Select an icon to represent the terminal program.
9. A "Connect To" window will open. Select the Com (serial) port to which V-2000 is connected.

10. Enter the port transmission settings:

Baud Rate	1200, 2400, 4800, 9600
Data Bits	8
Parity	None
Stop Bit	1
Start Bit	1
Flow Control	Hardware

NOTE: The baud rate selected in Hyper Terminal must match the V-2000 baud rate. See Chapter 2, Section 2-5 Baud Rate.

11. From the HyperTerminal menu, select Transfer. Click on Capture Text. Enter the path and name of the file created in Step 1. Click on Start.
12. Make sure that the desired V-2000 printout format has been chosen as outlined in Chapter 2, Section 2-6 Print.
13. Press and hold the **log** key for approximately 4 seconds.
14. Press and hold the **print** key for approximately 4 seconds to download all saved data.
15. Log #, test results, program #, parameter, and date/time tag should appear in the Hyper Terminal window. When transmission is complete, click the Disconnect icon. Click on Transfer/Capture Text and Stop. Exit the HyperTerminal program.
16. The text file which captured the V-2000 data can now be opened in the appropriate software application. Data may be reformatted as needed for spreadsheet or database manipulation.

### 5-3 Uploading Method Revisions

The V-2000 method version can be updated by logging onto the CHEMetrics website, [www.chemetrics.com](http://www.chemetrics.com) and clicking on "V-2000 Upload New Method Update" under the Support tab. The CHEMetrics website will inform customers when new programs are available or existing programs have been updated. Also, the product specific test kits will have information about method updates as they occur. To ensure that the most current program calibrations are being used, it is recommended that every V-2000 user check the CHEMetrics website frequently.

To determine which method revision is currently present on your V-2000 instrument, simply turn the instrument on. On start up, the V-2000 briefly displays the current method revision.

Notes: Uploading a new method revision does not overwrite any user generated custom methods or discontinued methods.

#### Method Revision Update Instructions:

1. With the V-2000 power off, connect the V-2000 to a computer using the supplied RS-232 serial cable.
2. Press the power key to turn the V-2000 on.
3. Then press the setup key.
4. Press the ▲ or ▼ keys until "UPLOAD" is displayed, then press the yes key to accept.
5. V-2000 display should read "WAITING", indicating V-2000 is ready for the Method Revision Update.

NOTE: To abort the upload process, press yes key again.

6. Click on the link provided on the CHEMetrics website and open the program.
7. Follow the instructions provided in the file.

8. When the program is complete, the V-2000 will briefly display “ALL DONE” along with the new method revision number.

NOTE: The Method Revision Update should take approximately 1 minute or less to complete. The update may not progress if the procedure is not followed precisely, if any of the connections between the V-2000 and computer are not firmly intact, or if the wrong “<COM> port” number is entered. The message “Looking for V-2000 photometer” displayed in the black DOS window on the computer monitor for an extended time indicates that the process has stalled. If this occurs, abort the process by pressing any key on the computer keyboard, then power the V-2000 off and reinitiate the update. If additional attempts are unsuccessful, contact CHEMetrics Technical Support at 540-788-9026 or [technical@chemetrics.com](mailto:technical@chemetrics.com).

# Chapter 6

## Error Messages

<b>Error Code</b>	<b>Cause</b>	<b>Required Action</b>
E11, 420 LOW, 420 HIGH	420 nm LED Failure	Contact technical@chemetrics.com
E12, 520 LOW, 520 HIGH	520 nm LED Failure	Contact technical@chemetrics.com
E13, 580 LOW, 580 HIGH	580 nm LED Failure	Contact technical@chemetrics.com
E14, 610 LOW, 610 HIGH	610 nm LED Failure	Contact technical@chemetrics.com
E19	LCD Driver Error	Contact technical@chemetrics.com
KBD ERR	Keyboard Failure	Contact technical@chemetrics.com
OVERRNG	Over-range: Test result is more than 110% above claimed operational range	Dilute the sample and repeat the test
UNDRRNG	Under-range: Test result is less than zero.	No action required

# Chapter 7

## Custom User Programs

The V-2000 offers the user the option of taking readings in absorbance or % Transmittance at each of the 4 available wavelengths (program numbers 1 - 4). Absorbance readings can then be used to generate a user specific custom method.

The V-2000 offers the user the ability to store up 10 user specific custom methods.

Program Number	Designation
0	manual zeroing; equivalent to using the <b>zero</b> key
1	420 nm absorbance or % Transmittance
2	520 nm absorbance or % Transmittance
3	580 nm absorbance or % Transmittance
4	610 nm absorbance or % Transmittance
190-199	user generated custom methods

### 7-1 Creating a Custom User Program

1. Install the appropriate sample cell adapter into the photometer.
2. Turn the photometer on by pressing the power key.
3. Insert the zeroing vial into the V-2000, cover the zeroing vial with the light shield, and press the zero key. "Wait" is displayed, then the result is displayed as "0.000".
4. Press the prgm key, enter the desired program number (190 -199 only), The instrument will display "No User X" if no user program is currently stored at that location.
5. Press the setup key and use the ▲ or ▼ keys until "USR PRGM" is displayed, then press the yes key.
6. "WAVELENGTH?" will be displayed. Use the ▲ or ▼ keys to select the desired wavelength. When the desired wavelength is displayed, press the yes key.
7. "UNITS" will be displayed. Use the ▲ or ▼ keys to select the desired units. When the desired units are displayed, press the yes key.
8. "TIMER 1" will be displayed. Press the yes key to set the first timer, then use the ▲ or ▼ keys to select the desired length of time (10 second intervals). When the desired length of time is displayed, press the yes key.
9. "TIMER 2" will be displayed. Press the yes key to set the second timer, then use the ▲ or ▼ keys to select the desired length of time (10 second intervals). When the desired length of time is displayed, press the yes key.
10. "SAVE USR ?" will be displayed. Press the yes key to save the new user program. "—SAVED—" will be displayed. The instrument will automatically proceed to "CAL USR".

## 7-2 Calibrating a Custom User Program

A calibration must be entered to use a new custom user program. Calibrating a user program requires a minimum of 2 calibration standards, selected to bracket the anticipated sample concentration. However, the V-2000 will accept up to 5 calibration standards which cover the expected range of the test.

1. Follow the parameter specific Test Procedure to generate 2 to 5 fully reacted test vials for the selected standard concentrations.
2. While "CAL USR" is displayed, press the yes key.
3. "3 CAL PTS?" will be displayed. Use the ▲ or ▼ keys until the desired number of calibration points is displayed, then press the yes key.
4. "PT1 CONC?" will be displayed. Insert the first reacted standard into the V-2000 and cover it with the light shield. Enter the value for the first standard using the numeric keypad. Press the yes key to accept the value entered. The V-2000 will read the first standard and automatically proceed to the next standard.

NOTE: If no decimal point is used in the concentration value, press the yes key again to accept the value.

5. Repeat Step 4 for each of the remaining standard concentrations.
6. "CAL OK" will be displayed at the completion of the calibration.

## 7-3 Replacing or Reviewing a Custom User Program

1. Install the appropriate sample cell adapter into the photometer.
2. Turn the photometer on by pressing the power key.
3. Insert the zeroing vial into the V-2000, cover the zeroing vial with the light shield, and press the zero key. "Wait" is displayed, then the result is displayed as "0.000".
4. Press the prgm key, enter the desired program number (190 -199 only), The instrument will display "No User X" if no user program is currently stored at that location or "\*\*\*USR X" if a program is stored at that location.
5. Press the setup key and use the ▲ or ▼ keys until "USR PRGM" is displayed, then press the yes key.
6. If a stored user program exists, "REPL OLD?" will be displayed. Use the ▲ or ▼ keys to toggle between "REPL OLD?" and "REVIEW?". To view program settings, press the yes key to select "REVIEW?". To replace the existing program or change certain settings, press the yes key to select "REPL OLD?".

# Chapter 8

## Specifications & Features

Direct Read Programs	50+
Wavelength (nm)	420, 520, 580, 610
Wavelength Accuracy	± 2 nm
Wavelength Selection	Automatic
Photometric Reproducibility	± 0.005A (0-1A)
Photometric Accuracy	± 0.005A @ 1.0 Abs Nominal
Photometric Range	0 - 2 A
Light Source	Light Emitting Diode (LED)
Detector	Photodiode
Bandwidth	10 ± 2 nm
Operating Temperature	0.0 to 45.0 °C
Humidity	90% at 50.0 °C max
Waterproof	IP67
Cell Adapter(s)	16 mm, 13 mm
Output Units	mg/L, ppm, µg/L, ppb, g/L, Absorbance, or % Transmittance
Datalogging	100 points, Date and Time Tag
Download Capability	Data to Spreadsheet and Printer, RS232 Output
Upload Capability	Methods Update, RS232 Input
Power Supply	4 AA Alkaline Batteries
Compliance	European CE Mark
Timing Capability	Built-in Timer

# Chapter 9

## Product Information

### 9-1 Instrument & Accessories

Catalog Number	Description
V-2000	V-2000 Photometer, comes with 13 mm sample cell adapter (installed), 16 mm sample cell adapter, COD ZEROING Vial, RS-232 cable, serial connector, light shield, operator's manual, 4 alkaline AA batteries.
A-0182	V-2000 Carrying case, hard plastic kit case with handle and foam insert that holds V-2000 Photometer and up to 4 Vacu-vials Test Kits.
A-0183	COD ZEROING Vial, package of one
A-0188	Sample Dilution Kit, to extend the kit ranges on the V-2000, equipment to perform 30 sample dilutions (10X, 125X, 250X, 500X, 1000X and 5000X). Not applicable to Oxygen or Ozone analysis.
A-0190	Canvas Tote Bag, soft canvas bag with shoulder strap and multiple pockets that hold V-2000 Photometer and up to 4 Vacu-vials Test Kits.
A-0307	RS232 (9 pin) to USB cable adapter, package of one.

### 9-2 Warranty Information

The V-2000 Photometer, other than its expendable components (sample cell adapters, light shield, RS-232 cable and serial connector), carries an unconditional guarantee of freedom from defects in material and workmanship for a period of two years from date of shipment. User access to the interior of the instrument may impair its function and will void this warranty. Improper use, application or servicing also voids this warranty. Manufacturer's liability shall not exceed cost of replacement of the photometer.

### 9-3 European Union's WEEE Directive 2002/96/EC

This product is required to comply with European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the WEEE symbol. For information on recycling this instrument, contact the instrument distributor.



Marking of electrical and electronic equipment, which applies to electrical and electronic equipment falling under the Directive 2002/96/EC (WEEE) and the equipment that has been put on the market after 13 August 2005.



# DR/890 COLORIMETER PROCEDURES MANUAL





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# INTRODUCTION

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This manual is divided into five sections:

## **Section 1 Chemical Analysis Information**

This section applies to all the procedures. It provides background information and reference/review material for the technician or chemist. Commonly used techniques are explained in detail.

## **Section 2 Sample Pretreatment**

This section provides a brief overview of sample pretreatment and two USEPA digestions. A brief discussion of the Hach Digesdahl Digestion Apparatus and the Hach Distillation Apparatus is included.

## **Section 3 Waste Management and Safety**

Section 3 includes information on waste management, regulations, waste disposal and resources on waste management. The Safety portion covers reading an MSDS and general safety guidelines.

## **Section 4 Procedures**

Section 4 contains step-by-step illustrated instructions for measuring parameters. The steps also include helpful notes. Each procedure contains information on sample collection, storage and preservation, accuracy checks, possible interferences, summary of method and a list of the reagents and apparatus necessary to run the test.

## **Section 5 Ordering Information**

This section provides information needed for ordering, shipping, return of items and Hach trademarks.

**Before attempting the analysis procedures the analyst should read the instrument manual to learn about the colorimeter's features and operation.**



# Sample Procedure Explained

**Range with units of measure**

**Approval of method by United States EPA if applicable**

**Types of samples analyzed**

**Procedure Name**

**Name of method used**

**Procedure Identification Number**

**Procedure step**

**Keystrokes required**

**Instrument Display**

**Illustration of procedure steps and instrument keystrokes required**

**Additional information that may be applicable**

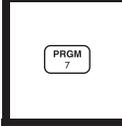
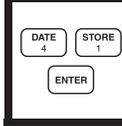
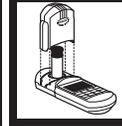
**Reference for method used**

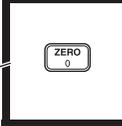
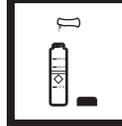
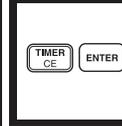
**Reference for EPA approval**

Method 8034  
For water and wastewater

**MANGANESE, HR (0 to 20.0 mg/L)**

**Periodate Oxidation Method<sup>\*</sup>; USEPA approved for reporting wastewater analysis (digestion is required; see Section 1)<sup>\*\*</sup>**

			
<p><b>1.</b> Enter the stored program number for manganese, periodate oxidation method.</p> <p>Press: <b>PRGM</b></p> <p>The display will show:</p> <p style="text-align: center;"><b>PRGM ?</b></p> <p><i>Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).</i></p>	<p><b>2.</b> Press: <b>41 ENTER</b></p> <p>The display will show <b>mg/L, Mn</b> and the <b>ZERO</b> icon.</p> <p><i>Note: For alternate forms (KMnO4, MnO4), press the <b>CONC</b> key.</i></p>	<p><b>3.</b> Fill a sample cell with 10 mL of sample (the blank).</p> <p><i>Note: For Total manganese determination perform a digestion (see Section 1).</i></p> <p><i>Note: Adjust the pH of stored samples before analysis.</i></p>	<p><b>4.</b> Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.</p>

			
<p><b>5.</b> Press: <b>ZERO</b></p> <p>The cursor will move to the right, then the display will show:</p> <p style="text-align: center;"><b>0.0 mg/L Mn</b></p>	<p><b>6.</b> Remove the cell from the instrument. Add the contents of one Buffer Powder Pillow, citrate type, to the cell. Cap the cell and invert until the powder is dissolved. Remove cap.</p>	<p><b>7.</b> Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap the sample cell. Invert for 10 seconds to mix.</p>	<p><b>8.</b> Press: <b>TIMER ENTER</b></p> <p>A two-minute reaction period will begin.</p> <p><i>Note: A violet color will form if manganese is present.</i></p>

<sup>\*</sup> Adapted from *Standard Methods for the Examination of Water and Wastewater*.  
<sup>\*\*</sup> *Federal Register*, 44 (116) 34193 (June 14, 1979).

## Sample Procedure Explained, continued

Specific sampling and storage information for this test

Confirm accuracy with these steps (in addition, may also be used to troubleshoot a test, improve technique, check reagents and to assure cleanliness of glassware)

### MANGANESE, HR, continued

---



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L manganese will be displayed.  
*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

---

**Sampling and Storage**  
Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1 for more information. If only dissolved Mn is to be determined, filter before acid addition.

**Accuracy Check**

**Standard Additions Method**

- Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.
- Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.
- Transfer only 10 mL of each solution to the 10-mL sample cells.
- Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* in Section 1 for troubleshooting information.

# Sample Procedure Explained, continued

Expected  
repeatability and  
estimated detection  
limit of the  
procedure

Levels of common  
sample substances  
or conditions that  
will cause  
inaccurate results

Concise  
explanation of  
method

**MANGANESE, HR, continued**

---

**Standard Solution Method**  
Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or CLASS A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.

**Method Performance**

**Precision**  
In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.18$  mg/L Mn.

**Estimated Detection Limit**  
The estimated detection limit for program 41 is 0.12 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

**Interferences**  
The following may interfere when present in concentrations exceeding those listed below:

Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

**Summary of Method**  
Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.

## Sample Procedure Explained, continued

**Lists all reagents and standards required for the procedure**

**Items needed to perform the procedure**

**Supplemental reagents and apparatus mentioned in notes or after the procedure**

**Amount of reagents and apparatus needed to perform the procedure**

**Use this phone number to obtain technical assistance**

MANGANESE, HR, continued			
REQUIRED REAGENTS			
High Range Manganese Reagent Set (100 tests) 10 mL			Cat. No. 24300-00
Description	Quantity Required Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows for manganese	.1 pillow	100/pkg	21077-69
REQUIRED APPARATUS			
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
OPTIONAL REAGENTS			
Hydrochloric Acid, 6 N	500 mL		884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL		12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg		14258-10
Nitric Acid, ACS	500 mL		152-49
Nitric Acid Solution 1:1	500 mL		2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB		2450-32
Water, deionized	4 L		272-56
OPTIONAL APPARATUS			
Ampule Breaker Kit	each		21968-00
Clippers, for opening powder pillows	each		968-00
Flask, erlenmeyer, 250 mL	each		505-46
Flask, volumetric, Class A, 50 mL	each		14574-41
Flask, volumetric, Class A, 500 mL	each		26366-49
Flask, volumetric, Class A, 100 mL	each		14574-42
Flask, volumetric, Class A, 1000 mL	each		14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg		391-33
pH Meter, EC10, portable	each		50050-00
Pipet, serological, 1mL	each		532-35
Pipet, serological, 5 mL	each		532-37
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each		19700-10
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg		21997-96
Pipet, volumetric, Class A, 5.00 mL	each		14515-37
Pipet, volumetric, Class A, 1.00 mL	each		14515-35
Pipet Filler, safety bulb	each		14651-00
<i>For Technical Assistance, Price and Ordering</i>			
In the U.S.A.—Call 800-227-4224			
Outside the U.S.A.—Contact the Hach office or distributor serving you.			

## SECTION 1 CHEMICAL ANALYSIS INFORMATION

### Abbreviations

The following abbreviations are used throughout the text of the procedure section:

Abbreviation	Definition	Abbreviation	Definition
°C	degree(s) Celsius (Centigrade)	MDL	Method detection limit
°F	degree(s) Fahrenheit	MDB	marked dropping bottle
ACS	American Chemical Society reagent grade purity	mg/L	milligrams per liter (ppm)
APHA Standard Methods	<i>Standard Methods for the Examination of Water and Wastewater.</i> <sup>1</sup>	µg/L	micrograms per liter (ppb)
AV	AccuVac	mL	(milliliter)-approximately the same as a cubic centimeter (cc) or 1/1000 of a liter. Also known as a "cc".
conc	concentrated	MR	medium range
CFR	Code of Federal Regulations	NIPDWR	National Interim Primary Drinking Water Regulations
DB	dropping bottle	NPDES	National Pollutant Discharge Elimination System
EDL	Estimated detection limit	PCB	Poly chlorinated biphenyl
FAU	Formazin Attenuation Units. Turbidity unit of measure based on a Formazin stock suspension.	SCDB	self-contained dropping bottle
g	grams	TNT	Test 'N Tube™
gr/gal	grains per gallon (1 gr/gal = 17.12 mg/L)	TPH	Total petroleum hydrocarbons
HR	high range	TPTZ	(2,4,6-Tri-(2-Pyridyl)-1,3,5-Triazine)
L	Liter. Volume equal to one cubic decimeter (dm <sup>3</sup> )	ULR	Ultra low range
LR	low range	USEPA	United States Environmental Protection Agency

<sup>1</sup> Published jointly by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Environment Federation (WEF). Order from Hach requesting Cat. No. 22708-00 or from the Publication Office of the American Public Health Association. This book is the standard reference work for water analysis. Many procedures contained in this manual are based on *Standard Methods*.

## CHEMICAL ANALYSIS INFORMATION, continued

### Converting Chemical Species

Species conversion factors for many commonly used substances are pre-programmed into the instrument (see *Table 1*). Conversions are method specific and are viewable after taking the reading by pressing **CONC**.

**Table 1 Conversion Factors**

To Convert From...	To...	Multiply By...
mg/L Al	mg/L Al <sub>2</sub> O <sub>3</sub>	1.8895
mg/L Ca-CaCO <sub>3</sub>	mg/L Ca	0.4004
mg/L CaCO <sub>3</sub>	mg/L Ca	0.4004
mg/L CaCO <sub>3</sub>	mg/L Mg	0.2428
µg/L Carbohydrazide	µg/L Hydroquinone	1.92
µg/L Carbohydrazide	µg/L ISA	2.69
µg/L Carbohydrazide	µg/L MEKO	3.15
mg/L Cr <sup>6+</sup>	mg/L CrO <sub>4</sub> <sup>2-</sup>	2.231
mg/L Cr <sup>6+</sup>	mg/L Na <sub>2</sub> CrO <sub>4</sub>	3.115
mg/L Mg-CaCO <sub>3</sub>	mg/L Mg	0.2428
mg/L Mn	mg/L KMnO <sub>4</sub>	2.876
mg/L Mn	mg/L MnO <sub>4</sub> <sup>-</sup>	2.165
mg/L Mo <sup>6+</sup>	mg/L MoO <sub>4</sub> <sup>2-</sup>	1.667
mg/L Mo <sup>6+</sup>	mg/L Na <sub>2</sub> MoO <sub>4</sub>	2.146
mg/L N	mg/L NH <sub>3</sub>	1.216
mg/L N	mg/L NO <sub>3</sub> <sup>-</sup>	4.427
mg/L Na <sub>2</sub> CrO <sub>4</sub>	mg/L Cr <sup>6+</sup>	0.321
mg/L Na <sub>2</sub> CrO <sub>4</sub>	mg/L CrO <sub>4</sub> <sup>2-</sup>	0.72
mg/L NH <sub>2</sub> Cl-N	mg/L Cl <sub>2</sub>	5.0623
mg/L NH <sub>2</sub> Cl-N	mg/L NH <sub>2</sub> Cl	3.6750
mg/L NH <sub>3</sub> -N	mg/L NH <sub>3</sub>	1.216
mg/L NH <sub>3</sub> -N	mg/L NH <sub>4</sub> <sup>+</sup>	1.288
mg/L NO <sub>2</sub> <sup>-</sup>	mg/L NaNO <sub>2</sub>	1.5
mg/L NO <sub>2</sub> <sup>-</sup>	mg/L NO <sub>2</sub> <sup>-</sup> -N	0.3045
mg/L NO <sub>2</sub> <sup>-</sup> -N	mg/L NaNO <sub>2</sub>	4.926
µg/L NO <sub>2</sub> <sup>-</sup> -N	µg/L NaNO <sub>2</sub>	4.926
mg/L NO <sub>2</sub> <sup>-</sup> -N	mg/L NO <sub>2</sub> <sup>-</sup>	3.284
µg/L NO <sub>2</sub> <sup>-</sup> -N	µg/L NO <sub>2</sub> <sup>-</sup>	3.284
mg/L NO <sub>3</sub> <sup>-</sup> -N	mg/L NO <sub>3</sub> <sup>-</sup>	4.427
mg/L PO <sub>4</sub> <sup>3-</sup>	mg/L P	0.3261
µg/L PO <sub>4</sub> <sup>3-</sup>	µg/L P	0.3261
mg/L PO <sub>4</sub> <sup>3-</sup>	mg/L P <sub>2</sub> O <sub>5</sub>	0.7473
µg/L PO <sub>4</sub> <sup>3-</sup>	µg/L P <sub>2</sub> O <sub>5</sub>	0.7473
mg/L SiO <sub>2</sub>	mg/L Si	0.4674
µg/L SiO <sub>2</sub>	µg/L Si	0.4674

## CHEMICAL ANALYSIS INFORMATION, continued

### Hardness Conversion

Table 2 lists the factors for converting one unit of measure for hardness to another unit of measure. For example, to convert mg/L CaCO<sub>3</sub> to German parts/100,000 CaO, multiply the value in mg/L x 0.056.

**Table 2 Hardness Conversion Factors**

Units of Measure	mg/L CaCO <sub>3</sub>	British gr/gal (Imperial) CaCO <sub>3</sub>	American gr/gal (US) CaCO <sub>3</sub>	French parts/100,000 CaCO <sub>3</sub>	German Parts/100,000 CaO	meq/L <sup>1</sup>	g/L CaO	lbs./cu ft CaCO <sub>3</sub>
mg/L CaCO <sub>3</sub>	1.0	0.07	0.058	0.1	0.056	0.02	5.6x10 <sup>-4</sup>	6.23x10 <sup>-5</sup>
English gr/gal CaCO <sub>3</sub>	14.3	1.0	0.83	1.43	0.83	0.286	8.0x10 <sup>-3</sup>	8.9x10 <sup>-4</sup>
US gr/gal CaCO <sub>3</sub>	17.1	1.2	1.0	1.72	0.96	0.343	9.66x10 <sup>-3</sup>	1.07x10 <sup>-3</sup>
Fr. p/100,000 CaCO <sub>3</sub>	10.0	0.7	0.58	1.0	0.56	0.2	5.6x10 <sup>-3</sup>	6.23x10 <sup>-4</sup>
Ger. p/100,000 CaO	17.9	1.25	1.04	1.79	1.0	0.358	1x10 <sup>-2</sup>	1.12x10 <sup>-3</sup>
meq/L	50.0	3.5	2.9	5.0	2.8	1.0	2.8x10 <sup>-2</sup>	3.11x10 <sup>-2</sup>
g/L CaO	1790.0	125.0	104.2	179.0	100.0	35.8	1.0	0.112
lbs./cu ft CaCO <sub>3</sub>	16,100.0	1,123.0	935.0	1,610.0	900.0	321.0	9.0	1.0

<sup>1</sup> 'epm/L, or 'mval/L'

Note: 1 meq/L = 1N/1000

## CHEMICAL ANALYSIS INFORMATION, continued

### Dissolved Oxygen

Table 3 lists the mg/L dissolved oxygen in water at saturation for various temperatures and atmospheric pressures. The table was formulated in a laboratory using pure water. The values given are only approximations for estimating the oxygen content of a particular body of surface water.

**Table 3 Dissolved Oxygen Saturation In Water**

Temp		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
°F	°C	inches							
		30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
32.0	0	14.9	14.6	14.4	13.9	13.5	12.9	12.5	12.0
33.8	1	14.5	14.2	14.1	13.6	13.1	12.6	12.2	11.7
35.6	2	14.1	13.9	13.7	13.2	12.9	12.3	11.8	11.4
37.4	3	13.8	13.5	13.3	12.9	12.4	12.0	11.5	11.1
39.2	4	13.4	13.2	13.0	12.5	12.1	11.7	11.2	10.8
41.0	5	13.1	12.8	12.6	12.2	11.8	11.4	10.9	10.5
42.8	6	12.7	12.5	12.3	11.9	11.5	11.1	10.7	10.3
44.6	7	12.4	12.2	12.0	11.6	11.2	10.8	10.4	10.0
46.4	8	12.1	11.9	11.7	11.3	10.9	10.5	10.1	9.8
48.2	9	11.8	11.6	11.5	11.1	10.7	10.3	9.9	9.5
50.0	10	11.6	11.3	11.2	10.8	10.4	10.1	9.7	9.3
51.8	11	11.3	11.1	10.9	10.6	10.2	9.8	9.5	9.1
53.6	12	11.1	10.8	10.7	10.3	10.0	9.6	9.2	8.9
55.4	13	10.8	10.6	10.5	10.1	9.8	9.4	9.1	8.7
57.2	14	10.6	10.4	10.2	9.9	9.5	9.2	8.9	8.5
59.0	15	10.4	10.2	10.0	9.7	9.3	9.0	8.7	8.3
60.8	16	10.1	9.9	9.8	9.5	9.1	8.8	8.5	8.1
62.6	17	9.9	9.7	9.6	9.3	9.0	8.6	8.3	8.0
64.4	18	9.7	9.5	9.4	9.1	8.8	8.4	8.1	7.8
66.2	19	9.5	9.3	9.2	8.9	8.6	8.3	8.0	7.6
68.0	20	9.3	9.2	9.1	8.7	8.4	8.1	7.8	7.5
69.8	21	9.2	9.0	8.9	8.6	8.3	8.0	7.7	7.4
71.6	22	9.0	8.8	8.7	8.4	8.1	7.8	7.5	7.2
73.4	23	8.8	8.7	8.5	8.2	8.0	7.7	7.4	7.1

## CHEMICAL ANALYSIS INFORMATION, continued

**Table 3 Dissolved Oxygen Saturation In Water (continued)**

		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
Temp		inches							
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
75.2	24	8.7	8.5	8.4	8.1	7.8	7.5	7.2	7.0
77.0	25	8.5	8.4	8.3	8.0	7.7	7.4	7.1	6.8
78.8	26	8.4	8.2	8.1	7.8	7.6	7.3	7.0	6.7
80.6	27	8.2	8.1	8.0	7.7	7.4	7.1	6.9	6.6
82.4	28	8.1	7.9	7.8	7.6	7.3	7.0	6.7	6.5
84.2	29	7.9	7.8	7.7	7.4	7.2	6.9	6.6	6.4
86.0	30	7.8	7.7	7.6	7.3	7.0	6.8	6.5	6.2
87.8	31	7.7	7.5	7.4	7.2	6.9	6.7	6.4	6.1
89.6	32	7.6	7.4	7.3	7.0	6.8	6.6	6.3	6.0
91.4	33	7.4	7.3	7.2	6.9	6.7	6.4	6.2	5.9
93.2	34	7.3	7.2	7.1	6.8	6.6	6.3	6.1	5.8
95.0	35	7.2	7.1	7.0	6.7	6.5	6.2	6.0	5.7
96.8	36	7.1	7.0	6.9	6.6	6.4	6.1	5.9	5.6
98.6	37	7.0	6.8	6.7	6.5	6.3	6.0	5.8	5.6
100.4	38	6.9	6.7	6.6	6.4	6.2	5.9	5.7	5.5
102.2	39	6.8	6.6	6.5	6.3	6.1	5.8	5.6	5.4
104.0	40	6.7	6.5	6.4	6.2	6.0	5.7	5.5	5.3
105.8	41	6.6	6.4	6.3	6.1	5.9	5.6	5.4	5.2
107.6	42	6.5	6.3	6.2	6.0	5.8	5.6	5.3	5.1
109.4	43	6.4	6.2	6.1	5.9	5.7	5.5	5.2	5.0
111.2	44	6.3	6.1	6.0	5.8	5.6	5.4	5.2	4.9
113.0	45	6.2	6.0	5.9	5.7	5.5	5.3	5.1	4.8
114.8	46	6.1	5.9	5.9	5.6	5.4	5.2	5.0	4.8
116.6	47	6.0	5.9	5.8	5.6	5.3	5.1	4.8	4.7
118.4	48	5.9	5.8	5.7	5.5	5.3	5.0	4.8	4.6
120.2	49	5.8	5.7	5.6	5.4	5.2	5.0	4.7	4.5
122.0	50	5.7	5.6	5.5	5.3	5.1	4.9	4.7	4.4

## CHEMICAL ANALYSIS INFORMATION, continued

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### Sample Collection, Preservation and Storage

Correct sampling and storage are critical for accurate testing. For greatest accuracy, thoroughly clean sampling devices and containers to prevent carryover from previous samples. Preserve the sample properly; each procedure has information about sample preservation.

- The least expensive containers are polypropylene or polyethylene.
- The best and most expensive containers are quartz or PTFE (polytetrafluoroethylene, Teflon).
- Avoid soft glass containers for metals in the microgram-per-liter range.
- Store samples for silver determination in light-absorbing containers, such as amber bottles.

Avoid contaminating the sample with metals from containers, deionized water or membrane filters. Thoroughly clean sample containers as described under Acid Washing Bottles.

Preservation slows the chemical and biological changes that continue after collection. These processes may change the amount of a chemical species available for analysis. Normally, analyze the samples as soon as possible after collection, especially when the analyte concentration is expected to be low. This also reduces the chance for error and minimizes labor.

Preservation methods include pH control, chemical addition, refrigeration and freezing. *Table 4* gives the recommended preservation for various substances. It also includes suggested types of containers and the maximum recommended holding times for properly preserved samples.

Preserve aluminum, cadmium, chromium, cobalt, copper, iron, lead, nickel, potassium, silver and zinc samples for at least 24 hours by adding one Nitric Acid Solution Pillow 1:1 (Cat. No. 2540-98) per liter of sample. Check the pH with pH indicator paper or a pH meter to assure the pH is 2 or less. Add additional pillows if necessary. Adjust the sample pH prior to analysis by adding an equal number of Sodium Carbonate Anhydrous Powder Pillows (Cat. No. 179-98). Or raise the pH to 4.5 with Sodium Hydroxide Standard Solution, 1 N or 5 N. Correct for the added volume of the preservatives; see *Correcting For Volume Additions*.

## CHEMICAL ANALYSIS INFORMATION, continued

**Table 4 Required Containers, Preservation Techniques and Holding Times<sup>1</sup>**

Parameter No./Name	Container <sup>2</sup>	Preservation <sup>3,4</sup>	Maximum Holding Time <sup>5</sup>
<b>Table 1A - Bacterial Tests:</b>			
1-4. Coliform, fecal and total	P,G	Cool, 4°C, 0.008%, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours
5. Fecal streptococci	P,G	Cool, 4°C, 0.008%, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	6 hours
<b>Table 1B - Inorganic Tests:</b>			
1. Acidity	P, G	Cool, 4°C	14 days
2. Alkalinity	P, G	Cool, 4°C	14 days
4. Ammonia	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
9. Biochemical oxygen demand (BOD)	P, G	Cool, 4°C	48 hours
11. Bromide	P, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours
15. Chemical oxygen demand	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
16. Chloride	P, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze immediately
21. Color	P, G	Cool, 4°C	48 hours
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH>12, 0.6 g ascorbic acid <sup>6</sup>	14 days <sup>7</sup>
25. Fluoride	P	None required	28 days
27. Hardness	P, G	HNO <sub>3</sub> to pH<2, H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43. Kjeldahl and organic nitrogen	P, G	Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
<b>Metals:<sup>8</sup></b>			
18. Chromium VI	P, G	Cool, 4°C	24 hours
35. Mercury	P, G	HNO <sub>3</sub> to pH<2	6 months
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. <sup>9</sup> Metals, except boron, chromium VI and mercury	P, G	do	6 months
38. Nitrate	P, G	Cool, 4°C	48 hours
39. Nitrate-nitrite	P, G	Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
40. Nitrite	P, G	Cool, 4°C	48 hours
41. Oil and grease	G	Cool, 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
42. Organic Carbon	P, G	Cool, 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> or H <sub>3</sub> PO <sub>4</sub> to pH<2	28 days
44. Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
46. Oxygen, dissolved probe	G Bottle and top	None required	Analyze immediately
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours

## CHEMICAL ANALYSIS INFORMATION, continued

**Table 4 Required Containers, Preservation Techniques and Holding Times<sup>1</sup> (continued)**

Parameter No./Name	Container <sup>2</sup>	Preservation <sup>3,4</sup>	Maximum Holding Time <sup>5</sup>
48. Phenols	G only	Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
49. Phosphorus, elemental	G	Cool, 4°C	48 hours
50. Phosphorus, total	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
53. Residue, total	P, G	Cool, 4°C	7 days
54. Residue, filterable	P, G	Cool, 4°C	7 days
55. Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
56. Residue, Settleable	P, G	Cool, 4°C	48 hours
57. Residue, volatile	P, G	Cool, 4°C	7 days
61. Silica	P, PFTE or quartz	Cool, 4°C	28 days
64. Specific conductance	P, G	Cool, 4°C	28 days
65. Sulfate	P, G	Cool, 4°C	28 days
66. Sulfide	P, G	Cool 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, G	none required	Analyze immediately
68. Surfactants	P, G	Cool, 4°C	48 hours
69. Temperature	P, G	None required	Analyze immediately
73. Turbidity	P, G	Cool, 4°C	48 hours

<sup>1</sup> This table was taken from Table II published in the Federal Register, July 1, 1995, 40 CFR, Part 136.3, pages 643-645. Organic tests are not included.

<sup>2</sup> Polyethylene (P) or glass (G).

<sup>3</sup> Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

<sup>4</sup> When any sample is to be shipped by common carrier or sent through United States Mails, it must comply with the Department of Transportation Hazardous Material Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

<sup>5</sup> Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less after sample collection.

<sup>6</sup> Should only be used in the presence of residual chlorine.

<sup>7</sup> Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

<sup>8</sup> Samples should be filtered immediately on-site before adding preservative for dissolved metals.

<sup>9</sup> Numbers refer to parameter numbers in 40 CFR Part 136.3, Table 1B.

## **CHEMICAL ANALYSIS INFORMATION, continued**

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### **Collecting Water Samples**

Obtain the best sample by careful collection. In general, collect samples near the center of the vessel or duct and below the surface. Use only clean containers (bottles, beakers). Rinse the container several times first with the water to be sampled.

Take samples as close as possible to the source of the supply. This lessens the influences of the distribution system on the sample. Let the water run long enough to flush the system. Fill sample containers slowly with a gentle stream to avoid turbulence and air bubbles. Collect water samples from wells after the pump has run long enough to deliver water representative of the ground water feeding the well.

It is hard to obtain a truly representative sample when collecting surface water samples. Obtain best results by testing several samples. Use samples taken at different times from several locations and depths. The results can be used to establish patterns for that particular body of water.

Generally, as little time as possible should elapse between collecting the sample and analyzing it.

Depending on the test, special precautions in handling the sample may be necessary. This prevents natural interferences such as organic growth or loss or gain of dissolved gases. Each procedure describes sample preservatives and storage techniques for samples that are held for testing.

## CHEMICAL ANALYSIS INFORMATION, continued

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### Acid Washing Bottles

If a procedure suggests acid-washing, use the following instructions:

- a) Clean the glassware or plasticware with laboratory detergent (phosphate-free detergent is recommended).
- b) Rinse well with tap water.
- c) Rinse with a 1:1 Hydrochloric Acid Solution or 1:1 Nitric Acid Solution.
- d) Rinse well with deionized water at least four times. Up to 12-15 rinses may be necessary if chromium is being determined.
- e) Air dry.

Use chromic acid or chromium-free substitutes to remove organic deposits from glass containers. Rinse containers thoroughly with water to remove traces of chromium.

Wash glassware for phosphate determinations with phosphate-free detergents and acid-wash with 1:1 HCl. Thoroughly rinse the glassware with deionized water. For ammonia and Kjeldahl nitrogen, rinse with ammonia-free water.

### Correcting for Volume Additions

If you use a large volume of preservative, correct for the volume of preservative added. This accounts for dilution due to the acid added to preserve the sample and the base used to adjust the pH to the range of the procedure. This correction is made as follows:

1. Determine the volume of initial sample, the volume of acid and base added, and the total or final volume of the sample.
2. Divide the total volume by the initial volume of sample.
3. Multiply the test result by this factor.

#### Example:

A one-liter sample was preserved with 2 mL of nitric acid. It was neutralized with 5 mL of 5 N sodium hydroxide. The result of the analysis procedure was 10.00 mg/L. What is the volume correction factor and correct result?

1. Total Volume = 1000 mL + 2 mL + 5 mL = 1007 mL
2.  $\frac{1007}{1000} = 1.007 = \text{volume correction factor}$
3.  $10.0 \text{ mg/L} \times 1.007 = 10.07 \text{ mg/L} = \text{correct result}$

## CHEMICAL ANALYSIS INFORMATION, continued

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Each 1:1 Nitric Acid Pillows contain 2.5 mL of acid; correct for this volume. The addition of a Sodium Carbonate Power Pillow (neutralizes the 1:1 Nitric Acid Solution Pillow) does not need to be corrected for.

### Boiling Aids

Boiling is necessary in some procedures. Using a boiling aid such as boiling chips (Cat. No. 14835-31) helps reduce bumping. Bumping is caused by the sudden, almost explosive conversion of water to steam as it is heated. Avoid bumping; it may cause injury or sample loss.

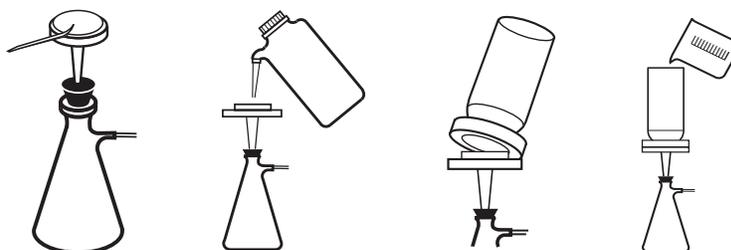
Make sure the boiling aids will not contaminate the sample. Do not use boiling aids (except glass beads) more than once. Loosely covering the sample during boiling will prevent splashing, reduce the chances of contamination and minimize sample loss.

### Sample Filtration

Filtration separates particles from the aqueous sample. Filtration uses a medium, usually filter paper, to retain particles but pass solution. This is especially helpful when sample turbidity interferes with analysis. Two general methods of filtration are gravity and vacuum. Gravity filtration uses gravity to pull the sample through the filter paper. Vacuum filtration uses suction and gravity to move the sample through the filter. An aspirator or vacuum pump creates the suction. Vacuum filtration is faster than gravity filtration. Vacuum filter (see *Figure 1*) as follows:

1. Using tweezers, place a filter paper into the filter holder.
2. Place the filter holder assembly in the filtering flask. Wet the filter with deionized water to ensure adhesion to the holder. Empty the flask before filtering the sample.
3. Position the funnel housing on the filter holder assembly.
4. While applying a vacuum to the filtering flask, transfer the sample to the filtering apparatus.
5. Slowly release the vacuum from the filtering flask and transfer the solution from the filter flask to another container.

Figure 1 Vacuum Filtration



## CHEMICAL ANALYSIS INFORMATION, continued

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### REQUIRED APPARATUS FOR VACUUM FILTRATION

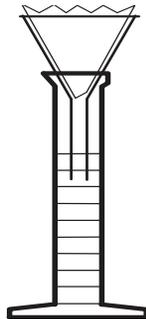
Description	Unit	Cat. No.
Filter Discs, glass 47 mm, 1.5 $\mu$ m .....	100/pkg.....	2530-00
Filter Holder, membrane .....	each.....	13529-00
Flask, filter, 500 mL.....	each.....	546-49
Pump, vacuum, hand operated.....	each.....	14283-00
OR		
Pump, vacuum, portable, 115 V.....	each.....	14697-00
Pump, vacuum, portable, 230 V .....	each.....	14697-02

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Several procedures in this manual use gravity filtration. The only labware required is filter paper, a conical funnel and a receiving vessel. This labware is included under Optional Apparatus at the end of a procedure. Gravity filtration is better for retaining fine particles. For faster filtering, add solution until the filter paper cone is three-fourths filled. Never fill the cone completely. Gravity filter (see *Figure 2*) as follows:

1. Place a filter paper into the funnel.
2. Wet the filter with deionized water to ensure adhesion to the funnel. Allow all the deionized water to drain.
3. Place the funnel into an erlenmeyer flask or graduated cylinder.
4. Pour the sample into the funnel.

Figure 2 Gravity Filtration



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### REQUIRED APPARATUS FOR GRAVITY FILTRATION

Description	Unit	Cat No.
Cylinder, graduated, 100 mL .....	each.....	508-42
Funnel, poly, 65 mm .....	each.....	1083-67
Filter Paper, 12.5 cm.....	each.....	1894-57
Flask, erlenmeyer, 125 mL .....	each.....	505-43

## CHEMICAL ANALYSIS INFORMATION, continued

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Testing for metals requires acid and heat to pretreat the sample. Since these conditions destroy filter paper, vacuum filtration with glass fiber filter discs is recommended. Also, glass filter discs, unlike paper, do not retain colored species.

### Temperature Considerations

For best results, perform most tests in this manual with sample temperatures between 20 °C (68 °F) and 25 °C (77 °F). If a test requires closer temperature control, notes in the procedure will indicate this.

### Sample Dilution Techniques

Ten and 25 mL are the volumes used for most colorimetric tests. However, in some tests, the color developed in the sample may be too intense to be measured. Unexpected colors may develop in other tests. In both cases, dilute the sample to determine if interfering substances are present.

To dilute the sample easily, pipet the chosen sample portion into a clean graduated cylinder (or volumetric flask for more accurate work). Fill the cylinder (or flask) to the desired volume with deionized water. Mix well. Use the diluted sample when running the test.

To help with dilutions, *Table 5* shows the amount of sample used, the amount of deionized water used to bring the volume up to 25 mL and the multiplication factor.

The concentration of the sample is equal to the diluted sample reading multiplied by the multiplication factor.

More accurate dilutions can be done with a pipet and a 100-mL volumetric flask (see *Table 6* for more information). Pipet the sample and dilute to volume with deionized water. Swirl to mix.

**Table 5 Sample Dilution Volumes**

Sample Volume (mL)	mL Deionized Water Used to Bring the Volume to 25 mL	Multiplication Factor
25.0	0.0	1
12.5	12.5	2
10.0 <sup>1</sup>	15.0	2.5
5.0 <sup>1</sup>	20.0	5
2.5 <sup>1</sup>	22.5	10
1.0 <sup>1</sup>	24.0	25
0.250 <sup>1</sup>	24.75	100

<sup>1</sup> For sample sizes of 10 mL or less, use a pipet to measure the sample into the graduated cylinder or volumetric flask.

## CHEMICAL ANALYSIS INFORMATION, continued

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Table 6 Multiplication Factors for Diluting to 100 mL

Sample Volume (mL)	Multiplication Factor
1	100
2	50
5	20
10	10
25	4
50	2

### Sample Dilution and Interfering Substances

Sample dilution may influence the level at which a substance may interfere. The effect of the interferences decreases as the dilution increases. In other words, higher levels of an interfering substance can be present in the original sample if it is diluted before analysis.

#### An Example:

Copper does not interfere at or below 100 mg/L for a 25.00 mL sample in a procedure. If the sample volume is diluted with an equal volume of water, what is the level at which copper will not interfere?

$$\frac{\text{Total volume}}{\text{Sample volume}} = \text{Dilution factor}$$

$$\frac{25}{12.5} = 2$$

$$\text{Interference Level} \times \text{Dilution Factor} = \text{Interference level in sample}$$

$$100 \times 2 = 200$$

The level at which copper will not interfere in the undiluted sample is at or below 200 mg/L.

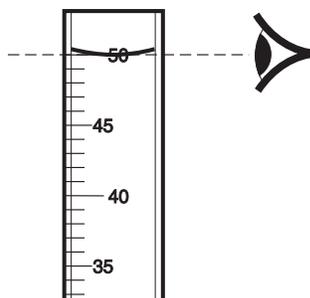
### Using Pipets and Graduated Cylinders

When small sample quantities are used, the accuracy of measurements is important. *Figure 3* illustrates the proper way of reading the sample level or the meniscus formed when the liquid wets the cylinder or pipet walls.

## CHEMICAL ANALYSIS INFORMATION, continued

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Figure 3 Reading the Meniscus



Rinse the pipet or cylinder two or three times with the sample to be tested before filling. Use a pipet filler or pipet bulb to draw the sample into the pipet. Never pipet chemical reagent solutions or samples by mouth. When filling a pipet, keep the tip of the pipet below the surface of the sample as the sample is drawn into the pipet.

Serological pipets have marks that indicate the volume of liquid delivered by the pipet. The marks may extend to the tip of the pipet or may be only on the straight portion of the tube. If the marks are only on the straight part of the tube, fill serological pipets to the zero mark and discharge the sample by draining the sample until the meniscus is level with the desired mark. If the serological pipet has marks extended to the tip of the pipet, fill the pipet to the desired volume and drain all the sample from the pipet. Then blow the sample out of the pipet tip for accurate measurements.

Volumetric (transfer) pipets have a bulb in the middle and a single ring above the bulb to indicate the volume of liquid when it is filled to the mark. To discharge a volumetric pipet, hold the pipet vertical until only a small amount of liquid remains (about  $\frac{3}{4}$  inch), then hold the pipet at a slight angle against the container wall to drain. Do not attempt to discharge the solution remaining in the tip of the pipet after draining. Volumetric pipets are designed to retain a small amount of sample in the pipet tip.

If sample drops stay on the walls of the pipet, the pipet is dirty and is not delivering the correct amount of sample. Wash the pipet thoroughly with a laboratory detergent or cleaning solution and rinse several times with deionized water.

## CHEMICAL ANALYSIS INFORMATION, continued

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### Using the TenSette Pipet

For best results use a new tip each time you pipet. After several uses, the pipet tip may retain some liquid, causing inaccurate delivery. Each pipet is supplied with 50 tips; order Hach replacement tips for best results.

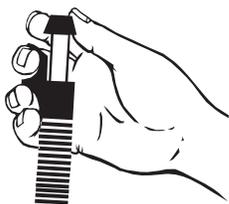
Always use careful, even hand movements for best reproducibility. If the pipet does not operate smoothly, disassemble and coat the piston and retainer with high-quality stopcock grease. Also coat the metering turret lightly with grease. Refer to the TenSette Pipet manual.

For best pipetting accuracy, the solution and the room temperature should be between 20-25 °C.

Never lay the pipet down with the liquid in the tip. Solution could leak into the pipet and cause corrosion.

### Operating the TenSette Pipet

1. Attach a clean tip by holding the pipet body in one hand and gently pressing the large end of the pipet tip onto the tapered end of the pipet. Be sure a good seal is obtained.
2. Turn the turret cap to align the desired volume with the mark on the pipet body.
3. Using a smooth motion, press down on the turret cap until it reaches the stop. Immerse the tip about 5 mm ( $\frac{1}{4}$  inch) below the solution surface to avoid drawing air into the pipet. Do not insert the tip any deeper or the delivery volume may be affected.
4. While maintaining a constant pressure, allow the turret to return slowly to the extended position. A rapid return may affect the delivery volume.
5. With the turret up, take the tip out of the solution and move it to the receiving vessel. Do not press on the turret cap while moving the pipet.



## CHEMICAL ANALYSIS INFORMATION, continued

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6. Use the thumb and forefinger to twist the turret cap to the next higher volume position to ensure quantitative transfer of the sample. The "F" position provides full blowout.



7. With the tip in contact with the side of the receiving vessel, slowly and smoothly press down on the turret cap until it reaches the stop and the solution is completely discharged.

### Mixing Water Samples

The following two methods may be helpful in tests that require mixing sample with chemicals (usually indicated by "invert to mix" instructions).

1. When mixing sample in a round sample cell or mixing cylinder, invert the cell or cylinder; see *Figure 4*. Hold the cell in a vertical position with the cap on top. Invert the cell so the cap is on the bottom. Return the cell to the original position. Do the same with the mixing cylinder.
2. Swirling is recommended when mixing samples in a graduated cylinder or a titration flask. Grip the cylinder (or flask) firmly with the tips of three fingers; see *Figure 5*. Hold the cylinder at a 45-degree angle and twist the wrist. This should move the cylinder in an approximately 12-inch circle, creating enough rotation to complete the mixing in a few turns.

These mixing procedures are the most gentle. Both methods are simple but take a bit of practice to obtain the best results.

## CHEMICAL ANALYSIS INFORMATION, continued

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Figure 4 Inverting a Sample Cell

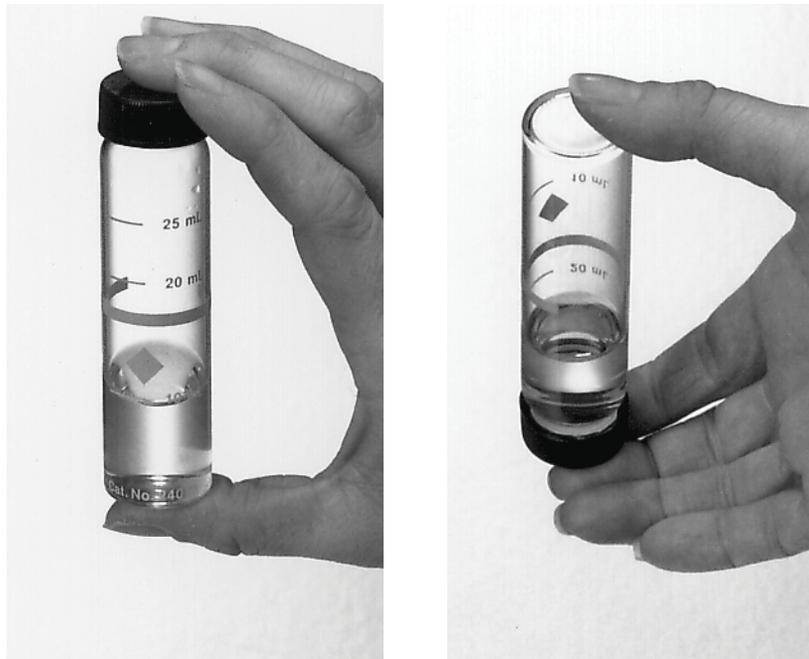


Figure 5 Swirling a Graduated Cylinder



## **CHEMICAL ANALYSIS INFORMATION, continued**

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### **Using Sample Cells**

#### **Orientation of Sample Cells**

Two round sample cells are shipped with the DR/820, DR/850 and DR/890. They are marked with 10-, 20- and 25-mL fill lines which may be used to measure the sample volume unless the procedure instructs you to use other glassware to measure the sample volume.

To minimize variability of measurements using a particular cell, always place the cell into the cell holder with the same orientation. The cells are placed in the instrument with the fill marks facing the user.

In addition to proper orientation, the sides of the cells should be free of smudges, fingerprints, etc. to ensure accurate readings. Wipe the sides of the cells with a moist cloth followed by a dry soft cloth to clean the surface before taking measurements.

#### **Care of Hach Sample Cells**

Store sample cells in their boxes when not in use to protect them from scratching and breaking. It is good laboratory practice to empty and clean sample cells after analyses are complete--avoid leaving colored solutions in the cells for extended periods of time. Finish the cleaning procedure with a few rinses of deionized water and allow to dry. Individual procedures often recommend specific cleaning methods.

#### **Cleaning Sample Cells**

Most laboratory detergents can be used at recommended concentrations. Neutral detergents such as Neutracon are safer if regular cleaning is required, as in the case of protein residues.

If using a detergent, you can speed cleaning by increasing the temperature or using an ultrasonic bath.

Rinsing is more efficient when using deionized water.

#### **Using the COD/TNT Adapter**

Use care when seating a vial into the COD/ TNT adapter (for COD vials and Test 'N Tubes). Place the vial into the adapter and press straight down on the top of the vial until it seats solidly. Do not move the vial from side to side; this can cause errors.

#### **Volume Measurement Accuracy**

The sample cells supplied with the instrument have fill marks to indicate 10, 20 or 25 mL. The fill marks are intended to measure the volume to be analyzed. Do not use these fill marks to perform sample dilutions.

If a sample must be diluted, use a pipet, graduated mixing cylinder and/or a volumetric flask for accurate measurement. When diluting, accuracy is important because a slight mistake in measuring a small sample will cause

## CHEMICAL ANALYSIS INFORMATION, continued

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a substantial error in the result. For instance, a 0.1-mL mistake in the dilution of a 1.0-mL final volume produces a 10% error in the test result.

Volumes for standard additions can be measured using the 25-mL mark, but it is not recommended for the 10-mL mark due to a potentially excessive relative error. An error of 0.5 mL in 25 mL is only 2%, while 0.5 mL error in 10 mL is 5%.

### For 10 mL standard additions, follow this procedure:

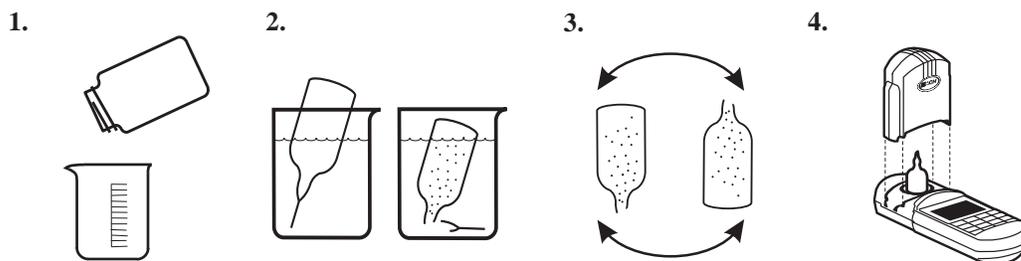
1. Transfer 10.0 mL of sample into a clean, dry sample cell (the unspiked sample).
2. Add the standard (spike) to a 25 mL portion of sample in a 25-mL mixing cylinder. Stopper and mix thoroughly.
3. Transfer 10 mL to another sample cell (use fill mark) for analysis.

### Using AccuVac Ampuls

AccuVac ampuls contain pre-measured powder or liquid in optical-quality glass ampuls.

1. Collect the sample in a beaker or other open container.
2. Place the ampul tip well below the sample surface and break the tip off (see *Figure 6*) against the beaker wall. The break must be far enough below the surface to prevent air from being drawn in as the level of the sample lowers (the AccuVac Breaker may be used instead of breaking the ampul against the beaker side).
3. Invert the ampul several times to dissolve the reagent. Do not place your finger over the broken end; the liquid will stay in the ampul when inverted. Wipe the ampul with a towel to remove fingerprints, etc.
4. Insert the ampul into the instrument and read the results directly.

Figure 6 Using AccuVac Ampuls



## CHEMICAL ANALYSIS INFORMATION, continued

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### Using Reagent Powder Pillows

Hach uses dry powdered reagents when possible. This minimizes leakage and deterioration problems. Some powders are packaged in individual, pre-measured, polyethylene "powder pillows" or foil pillows called PermaChem® pillows. Each pillow contains enough reagent for one test. Open the poly powder pillows with nail clippers or scissors; see *Figure 7*.

**Figure 7** Opening Powder Pillows



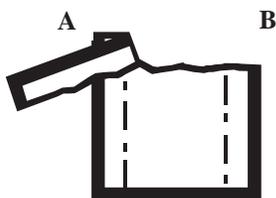
### Using PermaChem Pillows

1. Tap the pillow on a hard surface to collect the powdered reagent in the bottom.
2. Tear (or cut) across the top of the pillow, from B to A, holding the pillow away from your face.
3. Using two hands, push both sides toward each other to form a spout.
4. Pour the pillow contents into the sample cell and continue the procedure according to the instructions. Tap the pillow to remove any powder from the corners.

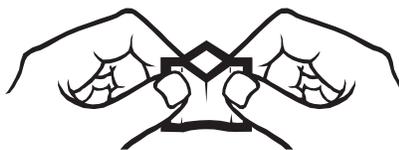
## CHEMICAL ANALYSIS INFORMATION, continued

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### 1. Tear



### 2. Push



### 3. Pour



### Reagent and Standard Stability

Hach always strives to make stable formulations and package them to provide maximum protection. Most chemicals and prepared reagents do not deteriorate after manufacture. However, the way they are stored and the packaging can affect how long the reagents are stable. Light, bacterial action, and absorption of moisture and gases from the atmosphere can affect shelf life. Some chemicals may react with the storage container or they may react with other chemicals.

Chemicals supplied with the colorimeter have an indefinite shelf life when stored under average room conditions, unless the packaging says something different. Product labels state any special storage conditions required. Otherwise, store reagents in a cool, dry, dark place for maximum life. It is always good practice to date chemicals when you receive them. Use older supplies first. If in doubt about the reagent shelf life, run a standard to check its effectiveness.

### Interferences

Substances in the sample may interfere with a measurement. Hach mentions common interferences in the test procedures. The reagent formulations eliminate many interferences. You can remove others with sample pretreatments described in the procedure.

If you get an unusual answer, a color that you don't expect, or you notice an unusual odor or turbidity, the result may be wrong. Repeat the test on a sample diluted with deionized water; see *Sample Dilution Techniques*. Compare the result (corrected for the dilution) with the result of the original test. If these two are not close, the original result may be wrong and you should make an additional dilution to check the second test (first dilution). Repeat this process until you get the same corrected result twice in a row.

More information about interferences and methods to overcome them is contained in *Standard Additions* of this manual and the *General Introduction* section of APHA Standard Methods. Hach urges the analyst to obtain this book and refer to it when problems are encountered.

## CHEMICAL ANALYSIS INFORMATION, continued

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One of the greatest aids is knowing what is in the sample. You don't need to know exactly what is in each sample, but be aware of substances that are likely to interfere in the analysis method you use. When using a method, it may be helpful to determine if those interferences are present.

### pH Interference

Many of the procedures in this manual only work within a certain pH range. Hach reagents contain buffers to adjust the pH of the typical sample to the correct pH range. However, the reagent buffer may not be strong enough for some samples. This occurs most often with highly buffered samples or samples with extreme sample pH.

The *Sampling and Storage* section of each procedure usually gives the proper pH range for the sample.

Adjust the sample to the proper pH range before testing. If this information is not given, follow these steps:

1. Measure the pH of your analyzed sample with a pH meter. For measuring  $\text{Ag}^+$ ,  $\text{K}^+$  or  $\text{Cl}^-$ , use pH paper.
2. Prepare a sample using deionized water. Add all reagents called for in the procedure. Timer sequences, etc., may be ignored. Mix well.
3. Measure the pH of the reagent blank with a pH meter.
4. Compare the pH values of your analyzed sample with the reagent blank.
5. If there is little difference in the values of your analyzed sample and the reagent blank, then pH interference is not the problem. Follow the *Accuracy Check* given in the procedure to help identify the problem.
6. If there is a large difference between the value of your analyzed sample and the reagent blank, adjust the sample pH to the value of the reagent blank. Adjust the sample pH to this same pH for all future samples from the same source before analysis. Use the appropriate acid, usually nitric acid, to lower the pH (do not use nitric acid for nitrate or nitrogen testing). Use the appropriate base, usually sodium hydroxide, to raise the pH. Adjust the final result for any dilution caused by adding acid or base; see *Correcting for Volume Additions*.
7. Analyze the sample as before.
8. Some purchased standards may be very acidic and will not work directly with Hach procedures. Adjust the pH of these standards as described above. Adjust the final concentration of the standard for the dilution. The Hach standard solutions suggested in the procedures are formulated so that no pH adjustment is necessary.

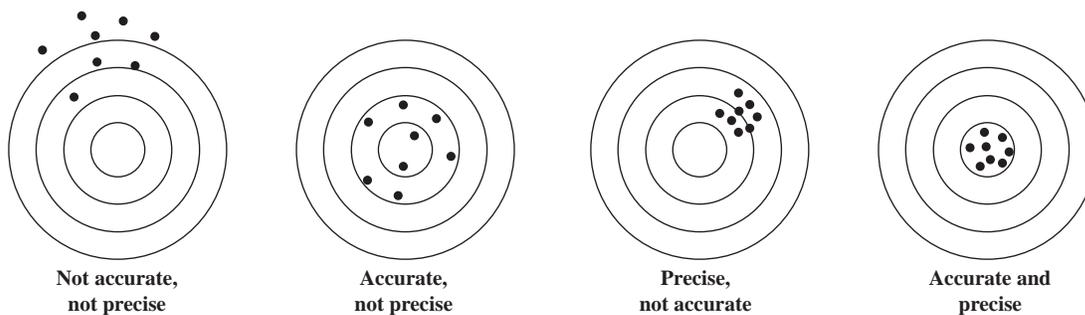
## CHEMICAL ANALYSIS INFORMATION, continued

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### Accuracy and Precision

Accuracy is the nearness of a test result to the true value. Precision is how closely repeated measurements agree with each other. Although good precision suggests good accuracy, precise results can be inaccurate (see *Figure 8*). The following paragraphs describe how to improve accuracy and precision of analyses by using Standard Additions.

**Figure 8** Precision and Accuracy Illustrated



### Standard Additions

Standard Additions is a common technique for checking test results. Other names are “spiking” and “known additions.” The standard additions technique can test for interferences, bad reagents, faulty instruments, and incorrect procedures.

Perform Standard Additions by following the Standard Additions Method section in the procedure under *Accuracy Check*. Follow the detailed instructions given.

If you get about 100% recovery for each addition, everything is working right and your results are correct.

If you don't get about 100% recovery for each addition, a problem exists. You can tell if you have an interference. Repeat the Standard Additions using deionized water as your sample. If you get about 100% recovery for each addition, you have an interference. If you didn't get good recoveries with the deionized water, the following checklist may help to find the problem quickly:

1. Check to see that you are following the procedure exactly:
  - a) Are you using the proper reagents in the proper order? Are you using 10-mL reagents with a 10-mL sample or 25-mL reagents with a 25-mL sample?
  - b) Are you waiting the necessary time for color to develop?

## CHEMICAL ANALYSIS INFORMATION, continued

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- c) Are you using the correct glassware?
- d) Is the glassware clean?
- e) Does the test need a specific sample temperature?
- f) Is the sample's pH in the correct range?

Hach's written procedure should help you to answer these questions.

2. Check your reagents. Repeat the Standard Additions using new, fresh reagents. If your results are good, the original reagents were bad.
3. If nothing else is wrong, the standard is almost certainly bad. Repeat the Standard Additions with a new standard.

If the check list does not determine the problem, use the decision tree (*Figure 9*) and explanation of each branch, below, to identify the problem.

### Branch A

Suppose a single standard addition to the sample did not give the correct concentration increase. A possible cause could be interferences. Other causes include defective reagents, incorrect technique, a defective instrument/apparatus or defective standard used for the standard addition.

If interferences are known or assumed to be absent, proceed to Branch B. If interferences are known to be present, proceed to Branch C.

### Branch B

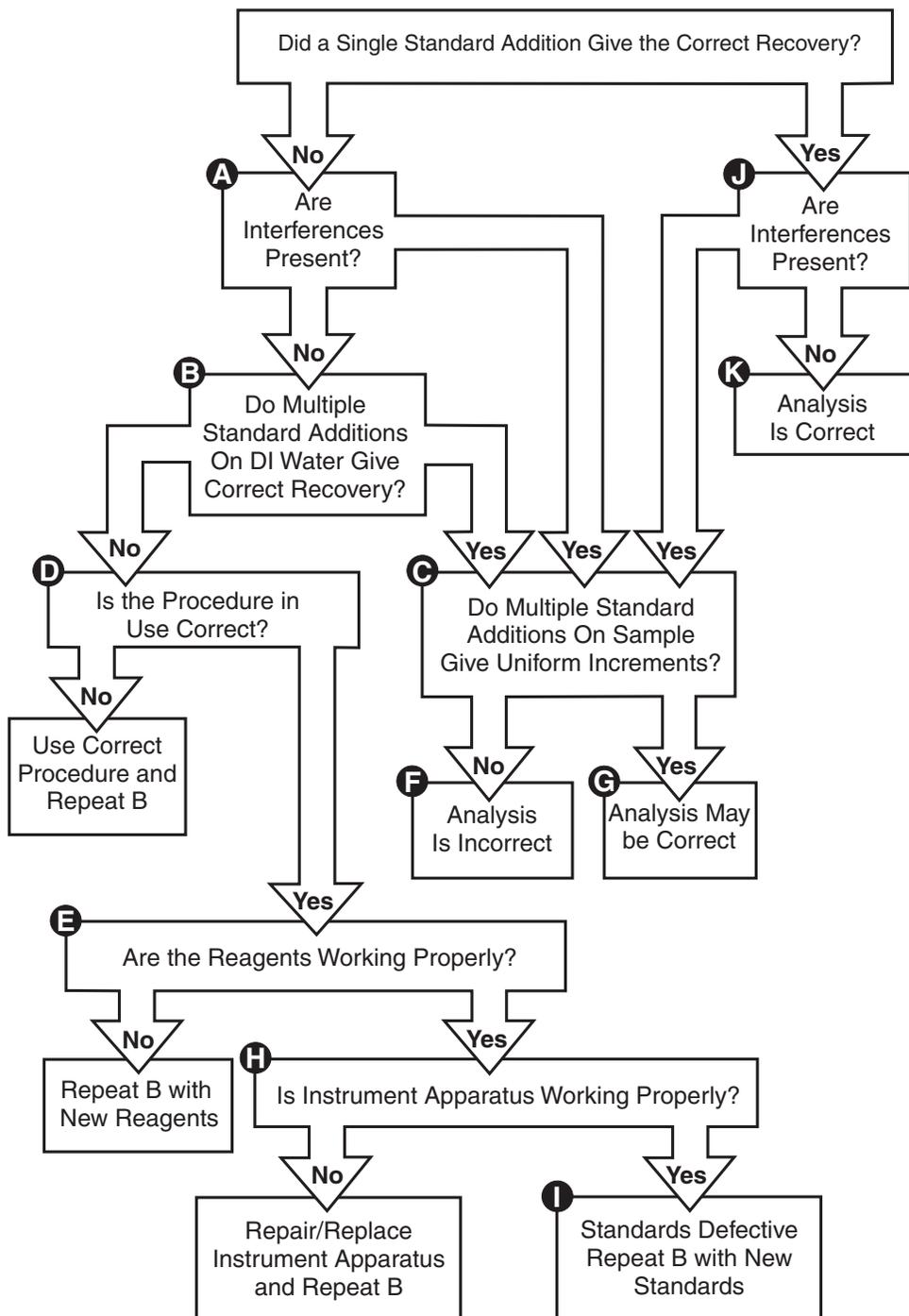
Perform multiple standard additions on a sample of deionized water as in the following example using iron as the analyte of interest:

1. Pour 25 mL of deionized water into a 25-mL sample cell.
2. Add 0.1 mL of a 50-mg/L iron standard solution to a second 25 mL sample of deionized water.
3. Add 0.2 mL of the same standard to a third 25 mL sample of deionized water.
4. Add 0.3 mL of the same standard to a fourth 25 mL sample of deionized water. Analyze all these samples for iron.
5. Tabulate the data as shown below:

mL of Standard Added	mg/L of Standard Added	mg/L of Iron Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

## CHEMICAL ANALYSIS INFORMATION, continued

Figure 9 Standard Additions Decision Tree



## CHEMICAL ANALYSIS INFORMATION, continued

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### The data show several points:

- The chemicals, instrument, procedure/technique and standards are working correctly because the iron added to the water sample was completely recovered in the same uniform steps that match the standard addition increments.
- Because iron added to the deionized water was recovered, but iron added to an actual sample was not recovered (Branch A), the sample contains an interference which prevents the test reagents from working properly.
- An iron analysis previously done on the actual sample using this method gave an inaccurate result.

If the results of multiple standard additions give the correct increment for each addition, proceed to Branch C.

If the results of multiple standard additions do not give the correct increment for each addition, go to Branch D.

### Branch C

If interfering substances are present, the analysis may be incorrect. However, with multiple standard additions, it may be possible to arrive at an approximate result if the increases are uniform.

Suppose the sample result for iron was 1.0 mg/L. Because interferences may be present, a standard addition of 0.1 mL of a 50 mg/L iron standard to a 25 mL sample is made. The expected increase in the iron concentration is 0.2 mg/L, but the actual increase is 0.1 mg/L. Then 0.2 and 0.3 mL of the same standard are added to two more 25 mL samples and analyzed for iron.

If there is a uniform increase in concentration between each addition (i.e., 0.1 mg/L difference between each addition), use Branch G. If the increase in concentration is not uniform (i.e., 0.1, 0.08, 0.05), go to Branch F.

### Branch D

Carefully check the instructions for the test. Make sure to use the correct reagents in the correct order. Be sure the glassware in use is what is required. Be sure time for color development and the sample temperature are as specified. If the procedure technique was incorrect, repeat Branch B. If the procedure was correctly followed, proceed to Branch E.

### Branch E

Check the reagent performance. This may be done by obtaining a fresh lot of reagent or by using a known standard solution to run the test. Make sure the color development time given in the procedure is equal to the

## CHEMICAL ANALYSIS INFORMATION, continued

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time required for the reagent in question. If the reagent(s) is defective, repeat Branch B with new reagents. If the reagents are good, proceed with Branch H.

### Branch F

Examples of non-uniform increments between standard additions are shown below.

#### Example A

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	1.0
0.1	0.2	1.10
0.2	0.4	1.18
0.3	0.6	1.23

#### Example B

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0
0.2	0.4	0.2
0.3	0.6	0.4

These examples show the effect of interferences on the standard addition. Data plotted on the graph in *Figure 10* for samples A and B show that the four data points do not lie on a straight line.

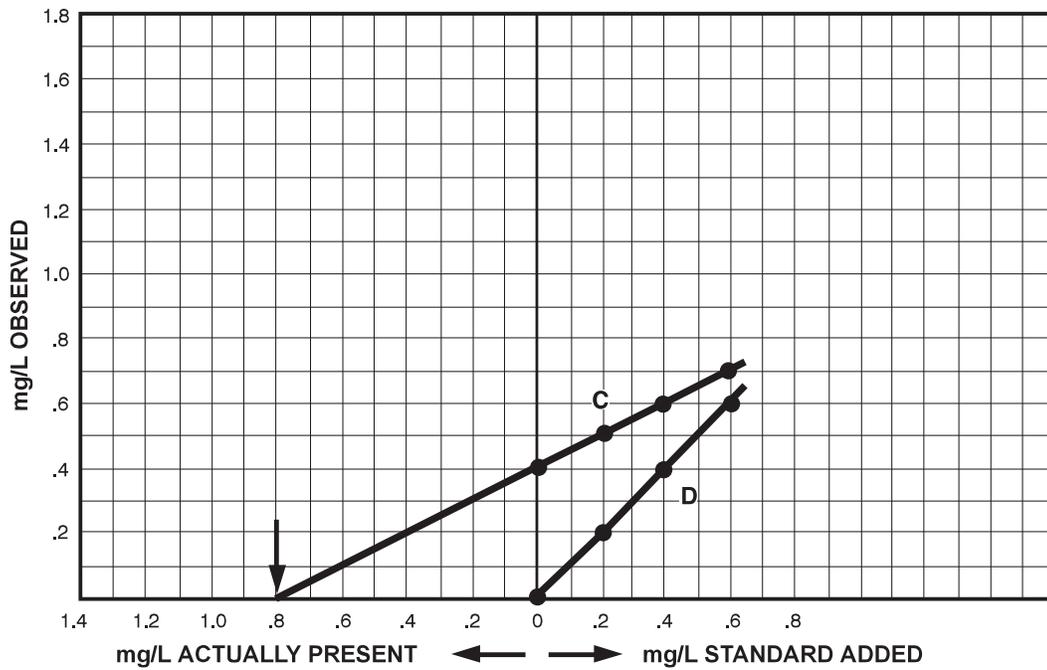
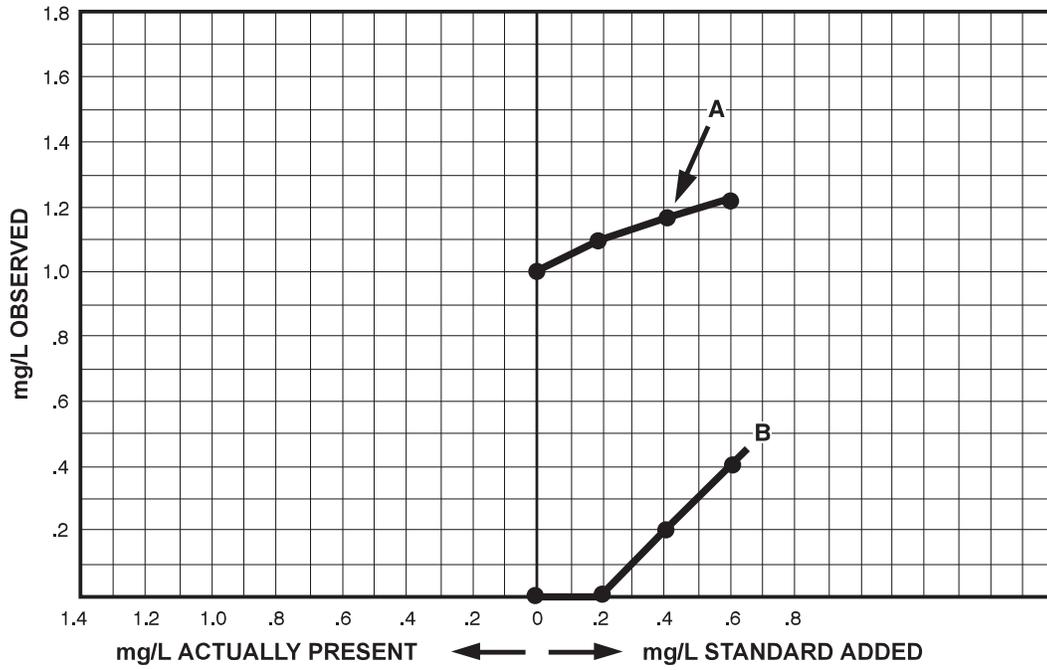
The plot for sample A illustrates an interference that becomes progressively worse as the concentration of the standard increases. This type of interference is uncommon and may be caused by an error or malfunction of the procedure, reagents or instrument. It is recommended Branch B be performed to verify the supposed interference.

The plot for sample B shows a common chemical interference which becomes less or even zero as the concentration of standard increases. The graph shows the first addition was consumed by the interference and the remaining additions gave the correct increment of 0.2 mg/L.

The apparent interference in Example B could be the result of an error made in the standard addition. Repeat the analysis to see if an error was made during standard addition. If not, the method is not appropriate for the sample matrix. When these two types of interferences occur, try to analyze the sample with a method which uses a different type of chemistry.

# CHEMICAL ANALYSIS INFORMATION, continued

Figure 10 Multiple Standard Additions Graph



## CHEMICAL ANALYSIS INFORMATION, continued

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### Branch G

Examples of uniform increments between standard additions are given below.

#### Example C

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0.4
0.1	0.2	0.5
0.2	0.4	0.6
0.3	0.6	0.7

The plot for sample C illustrates a common interference with a uniform effect on the standard and the substances in the sample. The four data points form a straight line which may be extended back through the horizontal axis. The point where the line meets the axis can be used to determine the concentration of the substance you are measuring.

In this example, the first analysis gave 0.4 mg/L. After extrapolating the line to the horizontal axis, the graph shows the result should be much closer to the correct result: 0.8 mg/L.

Apparent interferences may also be caused by a defect in the instrument or standards. Before assuming the interference is chemical, check Branch B.

#### Example D

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

The plot for sample D illustrates a problem for the analyst. The increments are uniform and the recovery of the standard was complete. The result of the first analysis was 0 mg/L and the line extrapolates back through 0 mg/L. If interferences are known to be present, the interferences may be present in an amount equal to the substance in question, preventing the analyst from finding the substance. This would be an uncommon situation.

## CHEMICAL ANALYSIS INFORMATION, continued

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### Branch H

Check operation of the instrument and/or apparatus used to perform the test. Check glassware used in the procedure and make sure it is extremely clean. Dirty pipets and graduated cylinders can cause contamination and will not deliver the correct volume. Check delivery of pipets by using deionized water and a balance; 0.2 mL = 0.2 grams.

If a defect is found in the instrument and/or apparatus, repeat Branch B after repair or replacement. If the instrument and apparatus are working, proceed with Branch I.

### Branch I

After determining the procedure, reagents, instrument and/or apparatus are correct and working properly, you may conclude the only possible cause for standard additions not functioning correctly in deionized water is the standard used for performing standard additions. Obtain a new standard and repeat Branch B.

### Branch J

If the standard additions gives the correct result, the analyst must then determine if an interfering substance(s) is present. If interfering substances are present, proceed to Branch C. If they are not present, the analysis is correct.

If you still cannot identify the problem, extra help is available. Please call our Technical Support Group at 800-227-4224 (U.S.A.) or 970-669-3050. A representative will be happy to help you.

## Method Performance

### Estimated Detection Limit

Ranges for chemical measurements have limits. The lower limit is important because it determines whether a measurement is different from zero. Many experts disagree about the definition of this detection limit, and determining it can be difficult. The Code of Federal Regulations (40 CFR, Part 136, Appendix B) provides a procedure to determine the "Method Detection Limit" or MDL. The MDL is the lowest concentration that is different from zero with a 99% level of confidence. A measurement below this MDL may be useful, but there is a greater chance that it is actually zero.

The MDL is not fixed; it varies for each reagent lot, instrument, analyst, sample type, etc. Therefore, a published MDL may be a useful guide, but is only accurate for a specific set of circumstances. Each analyst should determine a more accurate MDL for each specific sample matrix using the same equipment, reagents and standards that will routinely be used for measurements.

## CHEMICAL ANALYSIS INFORMATION, continued

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Hach provides a value called the Estimated Detection Limit (EDL) for all programs. It is the calculated lowest average concentration in a deionized water matrix that is different from zero with a 99% level of confidence. Specifically, it is the upper 99% confidence limit for zero concentration based on the calibration data used to prepare the pre-programmed calibration curve. **Do not use the EDL as the MDL.** The conditions for MDL determination must be exactly the same as the conditions used for analysis. The EDL may be useful to the analyst as a starting point in determining a MDL or as a way to compare methods. Measurements below the EDL may also be valuable because they can show a trend, indicate the presence of analyte and/or provide statistical data. However, these values have a large uncertainty.

### Method Detection Limit (MDL)

This method is in accordance with the USEPA definition in 40 CFR, Part 136, Appendix B (see most current edition).

The USEPA defines the method detection limit (MDL) as the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero. Since the MDL will vary from analyst to analyst, it is important that analysts determine the MDL based on their unique operating conditions.

The procedure for determining MDL is based on replicate analyses at a concentration 1 to 5 times the estimated detection limit. The MDL value is calculated from the standard deviation of the replicate study results multiplied by the appropriate Student's *t* value for a 99% confidence interval. For this definition, the MDL does not account for variation in sample composition and can only be achieved under ideal conditions.

1. Estimate the detection limit. Use the Hach estimated detection limit (EDL) value stated in the *Method Performance* section of the analysis procedure.
2. Prepare a laboratory standard of the analyte in deionized water which is free of the analyte that is 1 to 5 times the estimated detection limit.
3. Analyze at least seven portions of the laboratory standard and record each result.
4. Calculate the average and standard deviation (*s*) of the results.

## CHEMICAL ANALYSIS INFORMATION, continued

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5. Compute the MDL using the appropriate Student's  $t$  value (see table below) and the standard deviation value:

$$\text{MDL} = \text{Student's } t \times s$$

Number of Test Portions	Student's $t$ Value
7	3.143
8	2.998
9	2.896
10	2.821

**For example:**

The EDL for measuring iron using the FerroZine method is 0.003 mg/L. An analyst accurately prepared 1 liter of a 0.010 mg/L (about 3x the EDL) laboratory standard by diluting a 10-mg/L iron standard in iron-free deionized water.

Eight portions of the standard were tested according to the FerroZine method with the following results:

Sample #	Result (mg/L)
1	0.009
2	0.010
3	0.009
4	0.010
5	0.008
6	0.011
7	0.010
8	0.009

Using a calculator program, the average concentration = 0.010 mg/L and the standard deviation ( $s$ ) = 0.0009 mg/L

Based on the USEPA's definition, calculate the MDL as follows:

$$\text{MDL for FerroZine method} = 2.998 (\text{Student's } t) \times 0.0009 (s)$$

$$\text{MDL} = 0.003 \text{ mg/L (agrees with initial estimate)}$$

## CHEMICAL ANALYSIS INFORMATION, continued

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*Note:* Occasionally, the calculated MDL may be very different than Hach's estimate of the detection limit. To test how reasonable the calculated MDL is, repeat the procedure using a standard near the calculated MDL. The average result calculated for the second MDL derivation should agree with the initial calculated MDL. Refer to 40 CFR, Part 136, Appendix B (7-1-94), pages 635-637 for detailed procedures to verify the MDL determination.

*Note:* Run a laboratory blank, containing deionized water without analyte, through the test procedure to confirm that the blank measurement is less than the calculated MDL. If the blank measurement is near the calculated MDL, repeat the MDL procedure using a separate blank for analysis for each standard solution portion analyzed. Subtract the average blank measurement from each standard and use the corrected standard values to calculate the average and standard deviation used in the MDL.

### Precision

Every measurement has some degree of uncertainty. Just as a ruler with markings of 0.1 mm leaves some doubt as to the exact length of a measurement, chemical measurements also have some degree of uncertainty. The quality of the entire chemical method determines the precision.

Uncertainty in chemical measurements may be due to systematic errors and/or random errors. A systematic error is a mistake that is always the same for every measurement made. For example, a blank can add to each measurement for a specific compound, giving consistently high results (a positive bias). Random errors are different for every test and add either positive or negative bias. Random errors may be caused by variation in analytical technique and cause response variation. Hach chemists work hard to eliminate systematic errors in Hach procedures using Hach reagents, but response variation occurs in all chemical measurements.

### Estimating Precision

The method performance section in each procedure provides an estimate of the procedure's precision. The procedures use a "replicate analysis" estimate, based on real data.

In replicate analysis, a Hach chemist prepares a specific concentration of the analyte in a deionized water matrix. The standard is then analyzed seven individual times with the two reagent lots used in the calibration (14 total samples). A standard deviation of the two sets of seven values is calculated. The larger value is reported in the method. The reported value provides an estimate of the "scatter" of results at a particular point in the calibration curve.

It is important to stress that the estimates are based on a deionized water matrix. Precision on real samples with varying matrices can be quite different than these estimates.

## CHEMICAL ANALYSIS INFORMATION, continued

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### Reagent Blank Correction

The Reagent Blank Correction subtracts the color absorbed when running the test with deionized water instead of sample. The blank value is subtracted from every result to correct for any background color due to reagents.

When using the Reagent Blank Correction feature, the blank correction should be entered before the Standard Adjust feature is used.

To enter a programmed correction for the reagent blank:

1. Run the test using deionized water with each new lot of reagents.
2. Press **READ** to obtain the blank value.
3. Press **SETUP**, scroll to **BLANK** and press **ENTER**. The display will show **BLANK?**.
4. Enter the blank value just read from the instrument.
5. Press **ENTER** to accept the value as the blank to be subtracted from each reading.
6. The display will show 0.00 mg/L (resolution and units vary) and the sample cell icon will be displayed, indicating that the reagent blank feature is enabled and the blank value will be subtracted from each reading. Repeat the reagent blank adjust for each new lot of reagents.

*Note:* After entering a reagent blank adjust, the display may flash "limit" when zeroing if the sample used for zeroing has a lower absorbance value than the reagent blank.

To disable the Reagent Blank adjust feature, press **SETUP**, scroll to **BLANK** and press **ENTER** twice. The concentration readings will be displayed without subtracting the blank. The sample cell icon will no longer appear in the display.

Do not use the Reagent Blank Adjust feature if the procedure uses a reagent blank for zeroing.

### Standard Adjust (Adjusting the Standard Curve)

The colorimeter has Hach Programs permanently installed in memory. A program usually includes a pre-programmed calibration curve. Each curve is the result of an extensive calibration performed under ideal conditions and is normally adequate for most testing. Deviations from the curve can occur from using compromised testing reagents, defective sample cells, incorrect test procedure, incorrect technique, or other correctable causes. Interfering substances or other causes may be beyond the analyst's control.

## CHEMICAL ANALYSIS INFORMATION, continued

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In some situations, using the pre-programmed curve may not be convenient:

- a) Running tests where frequent calibration curve checks are required.
- b) Testing samples which give a consistent test interference.

Consider the following before adjusting the calibration curve:

1. Will future test results be improved by adjusting the curve?
2. Are interfering substances consistent in all the samples that you will test?

Any precision and test range information provided with the procedure may not apply to an adjusted curve calibration.

You can adjust many of the calibration curves by following the steps found in the test procedures. Working carefully is important. After the adjustment, it is wise to run standard solutions of several concentrations to make sure the adjusted curve is satisfactory. Perform standard additions on typical samples to help determine if the adjusted curve is acceptable.

Think of the standard adjust measurement as a two-step process. First, the instrument measures the sample using the pre-programmed calibration. Second, it multiplies this measurement by an adjustment factor. The factor is the same for all concentrations. The instrument will remember the factor indefinitely and will display the standard adjustment icon when it is used.

Adjust the calibration curve using the reading obtained with a Hach Standard Solution or carefully prepared standard made from a concentrated Hach Standard Solution. It is important to adjust the curve in the correct concentration range. For most purposes, Hach recommends adjusting the curve using a standard concentration that is 70 to 85% of the maximum concentration range of the test.

For example, the Hach pre-programmed method for fluoride has a range of 0-2.0 mg/L F. To adjust the calibration curve, use a standard with a concentration between 1.4-1.6 mg/L. Hach provides a 1.60 mg/L Fluoride Standard Solution (80% of the full range). This is a convenient standard to use for adjusting the calibration curve.

If the range of all your samples is known to be below a concentration that is less than 50% of the full range (50% of 2.0 is 1.0 mg/L), then adjust the standard curve with a standard that is within that range. For example, if all the samples contain 0.6-0.9 mg/L F, you may use a 1.00 mg/L fluoride standard to adjust the curve. You may use the 1.00 mg/L standard because it is closer to the sample range you are working with.

## CHEMICAL ANALYSIS INFORMATION, continued

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If you are using a Reagent Blank Correction, the blank correction should be entered before the standard curve is adjusted.

To adjust the standard curve:

1. Prepare the standard.
2. Use the standard as the sample in the procedure.
3. When the reading for the standard is obtained, press **SETUP**.
4. Use the arrow keys to scroll to the “**STD**” setup option.
5. Press **ENTER** to activate the standard adjust option.
6. Edit the standard concentration to match that of the standard used.
7. Press **ENTER**. A small plot of a line through a point will be displayed, indicating that the curve has been adjusted with the standard.

*Note: If the attempted correction is outside the allowable adjustment limit, the instrument will beep and flash  $\emptyset$  and the operation will not be allowed.*

### Preparing a User-Entered Calibration Curve

1. Prepare five or more standards of known concentration that cover the expected range of the test. Run tests as described in the procedure on each prepared standard. Pour the customary volume of each known solution into a separate clean sample cell of the type specified for your instrument.
2. Standardize (zero) the instrument using an untreated water sample or a reagent blank, whichever the procedure instructs you to use.
3. Measure and record the absorbance or %T of the known solutions. To use %T vs. concentration see *%T Versus Concentration Calibration*. To use absorbance vs. concentration, see *Absorbance Versus Concentration Calibration*. Or create a user-entered program by storing a custom calibration in the non-volatile memory of the instrument. Refer to the section on entering user-entered programs in the instrument manual.

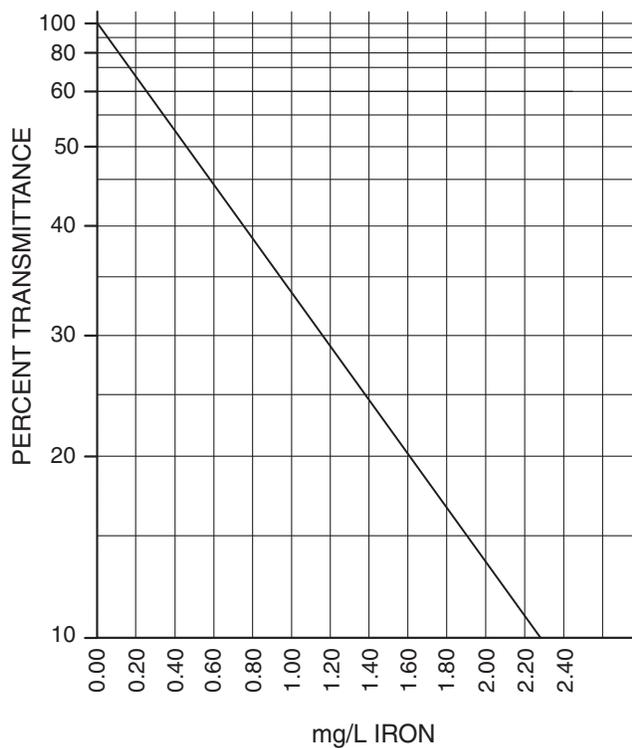
### %T Versus Concentration Calibration

If measuring %T, use semilogarithmic graph paper and plot %T (vertical scale) versus concentration (horizontal scale). In *Figure 11*, iron standard solutions of 0.1, 0.2, 0.4, 0.8, 1.2, 1.6, and 2.0 mg/L were measured on a spectrophotometer at 500 nm using half-inch test tubes. Results were plotted and the calibration table values were extrapolated from the curve (*Table 7*).

## CHEMICAL ANALYSIS INFORMATION, continued

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Figure 11 Logarithmic Calibration Curve



To convert %T readings to concentration, prepare a table such as *Table 7* and select the appropriate line from the “%T Tens” column and the appropriate column from the %T Units columns. The %T Ten value is the first number of the %T reading and the %T Units value is the second number of the %T reading. For example, if the instrument reading was 46%, the 40 line in the %T Tens column and the 6 column in the %T Units would be selected. The cell where these two intersect (0.78 mg/L) is the iron concentration of the sample.

## CHEMICAL ANALYSIS INFORMATION, continued

Table 7 Calibration Table

%T Tens	%T Units									
	0	1	2	3	4	5	6	7	8	9
0										
10	2.30	2.21	2.12	2.04	1.97	1.90	1.83	1.77	1.72	1.66
20	1.61	1.56	1.51	1.47	1.43	1.39	1.35	1.31	1.27	1.24
30	1.20	1.17	1.14	1.11	1.08	1.04	1.02	.99	.97	.94
40	.92	.89	.87	.84	.82	.80	.78	.76	.73	.71
50	.69	.67	.65	.64	.62	.60	.58	.56	.55	.53
60	.51	.49	.48	.46	.45	.43	.42	.40	.39	.37
70	.36	.34	.33	.32	.30	.29	.28	.26	.25	.24
80	.22	.21	.20	.19	.17	.16	.15	.14	.13	.12
90	.11	.09	.08	.07	.06	.05	.04	.03	.02	.01

### Absorbance Versus Concentration Calibration

To read concentration values directly from the instrument, create a user-entered program. See the instrument manual for more information.

If absorbance values are measured, plot the results on linear graph paper. Plot the absorbance value on the vertical axis and the concentration on the horizontal axis.

Plot increasing absorbance values from bottom to top. Plot increasing concentration values from left to right. Values of 0.000 absorbance units and 0 concentration will begin at the bottom left corner of the graph. A calibration table can be extrapolated from the curve or the concentration values can be read directly from the graph for determining an equation for the line using the slope and the y-intercept.

### USEPA Approved and Accepted Definitions

The United States Environmental Protection Agency (USEPA) establishes limits for maximum contamination levels of certain constituents in water. It also requires that specific methodology be used to analyze for these constituents. These methods originate from several sources. The USEPA has developed some of these methods. In other cases, the USEPA has evaluated and approved methods developed by manufacturers, professional groups and public agencies such as:

- American Public Health Association

## **CHEMICAL ANALYSIS INFORMATION, continued**

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- American Water Works Association
- Water Environmental Federation
- American Society for Testing and Materials
- United States Geological Survey
- Associates of Official Analytical Chemists

All USEPA approved methods are cited in the *Federal Register* and compiled in the Code of Federal Regulations. USEPA approved methods may be used for reporting results to the USEPA and other regulatory agencies.

### **USEPA Accepted**

Hach has developed several procedures that are equivalent to USEPA approved methods. Even though minor modifications exist, the USEPA has reviewed and accepted certain procedures for reporting purposes. These methods are not published in the *Federal Register*, but are referenced to the equivalent USEPA method in the procedure.

## SECTION 2 SAMPLE PRETREATMENT

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### Digestion

Several procedures require sample digestion. Digestion uses chemicals and heat to break down a substance into components that can be analyzed. This section has three different digestion procedures.

The Hach Digesdahl<sup>®</sup> system is a process that yields a digest suitable for the determination of metals, total phosphorus and total kjeldahl nitrogen (TKN). It is rapid, convenient and the method of choice for digesting most samples analyzed by Hach methods.

For USEPA reporting purposes, USEPA-approved digestions are required. USEPA presents two digestions (mild and vigorous) for metals analysis. These are much more inconvenient and time consuming compared to the Hach Digesdahl system. Other digestion procedures are required for phosphorus and TKN.

#### **EPA Mild Digestion with Hot Plate for Metals Analysis Only**

1. Acidify the entire sample at the time of collection with concentrated nitric acid by adding 5 mL of acid per liter (or quart) of sample.
2. Transfer 100 mL of well-mixed sample to a beaker or flask. Add 5 mL of distilled 1:1 hydrochloric acid (HCl).
3. Heat using a steam bath or hot plate until the volume has been reduced to 15-20 mL. Make certain the sample does not boil.
4. After this treatment, the sample may be filtered to remove any insoluble material.
5. Adjust the digested sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution. Mix thoroughly and check the pH after each addition.
6. Quantitatively transfer the sample with deionized water to a 100-mL volumetric flask and dilute to volume with deionized water. Continue with the procedure. This mild digestion may not suffice for all sample types. A reagent blank also should be carried through the digestion and measurement procedures.

## SAMPLE PRETREATMENT, continued

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### EPA Vigorous Digestion with Hot Plate for Metals Analysis Only

A vigorous digestion can be followed to ensure all organo-metallic bonds are broken.

1. Acidify the entire sample with redistilled 1:1 Nitric Acid Solution to a pH of less than two. Do not filter the sample before digestion.
2. Transfer an appropriate sample volume (see *Table 8*) into a beaker and add 3 mL of concentrated redistilled nitric acid.
3. Place the beaker on a hot plate and evaporate to near dryness, making certain the sample does not boil.
4. Cool the beaker and add another 3 mL of the concentrated redistilled nitric acid.
5. Cover the beaker with a watch glass and return it to the hot plate. Increase the temperature of the hot plate so that a gentle reflux occurs. Add additional acid, if necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change color or appearance with continued refluxing).
6. Again, evaporate to near dryness (do not bake) and cool the beaker. If any residue or precipitate results from the evaporation, add redistilled 1:1 hydrochloric acid (5 mL per 100 mL of final volume). See *Table 8*.
7. Warm the beaker. Add 5 mL of 5.0 N sodium hydroxide and quantitatively transfer the sample with deionized water to a volumetric flask. See *Table 8* below for the suggested final volume.
8. Adjust the sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution; mix thoroughly and check the pH after each addition. Dilute to volume with deionized water. Multiply the result by the correction factor in *Table 8*. A reagent blank also should be carried through the digestion and measurement procedures.

**Table 8 Vigorous Digestion Volumes**

Expected Metal Concentration	Suggested Sample Vol. for Digestion	Suggested Volume of 1:1 HCl	Suggested Final Volume After Digestion	Correction Factor
1 mg/L	50 mL	10 mL	200 mL	4
10 mg/L	5 mL	10 mL	200 mL	40
100 mg/L	1 mL	25 mL	500 mL	500

## SAMPLE PRETREATMENT, continued

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### General Digesdahl Digestion (Not USEPA accepted)

Many samples may be digested using the Digesdahl Digestion Apparatus (Cat. No. 23130). It is designed to digest many types of samples such as oils, wastewater, sludges, feeds, grains, plating baths, food, and soils. In this procedure the sample is oxidized by a mixture of sulfuric acid and hydrogen peroxide. Digestion of a dry sample requires less than ten minutes, while liquid samples require about 1 minute/mL. The digestion is done in a special flat-bottomed 100-mL volumetric flask. Aliquots (sample portions) are taken for analysis using colorimetric methods.

Procedures for digestion and using the Digesdahl Digestion Apparatus are based on the type and form of the sample, and are found in the Digesdahl Digestion Apparatus Instruction Manual, which is included with each Digesdahl Digestion Apparatus.

### Distillation

Distillation is an effective way of separating chemical components for analysis. The Hach Distillation Apparatus (see *Figure 12*) is adapted easily for many test needs and is suitable for water and wastewater samples. Sample distillations are easy and safe to perform.

#### Applications for the General Purpose Distillation Apparatus include:

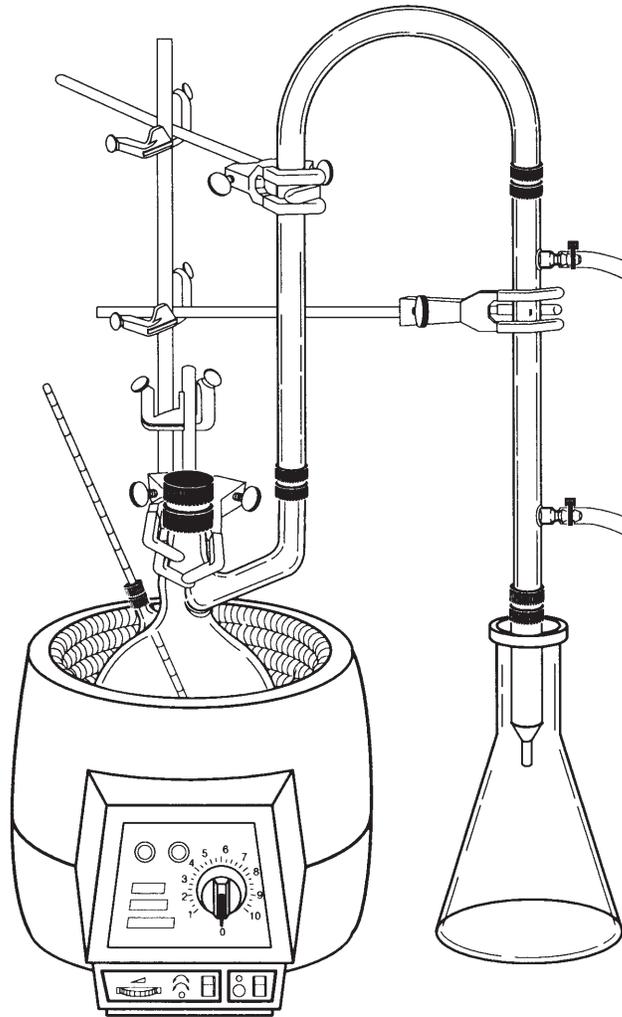
- fluoride
- albuminoid nitrogen
- ammonia nitrogen
- phenols
- selenium
- volatile acids

Arsenic and cyanide require special glassware sets in addition to the General Purpose Set (the Arsenic Distillation Apparatus and the Cyanide Distillation Apparatus). All connecting glassware is manufactured with threaded connectors for ease and safety. The General Purpose Heater provides efficient heating and the Support Apparatus anchors the glassware.

## SAMPLE PRETREATMENT, continued

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Figure 12 General Purpose Distillation Apparatus with Heater and Support Apparatus



## **SECTION 3 WASTE MANAGEMENT AND SAFETY**

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### **Waste Management**

This section provides guidelines for laboratory waste management. It should assist you in complying with USEPA regulations governing waste management. It summarizes basic requirements, but does not contain all USEPA regulations. It does not relieve people from complying with all regulations contained in the Code of Federal Regulations. Regulations change regularly and additional state and local laws may apply to your waste. Each waste generator is responsible for knowing and obeying the laws that apply to them.

### **Waste Minimization**

Waste minimization is the foundation of good waste management. Minimizing waste greatly reduces the disposal problems and expense. If possible, try to generate less waste rather than recycle or re-use it. For laboratories, ways to reduce waste include:

- Use the smallest sample size possible.
- Choose methods that use non-hazardous or “less” hazardous reagents when possible.
- Buy chemicals in small quantities which will be used before they expire. This eliminates disposal of outdated materials.
- Clean glassware and laboratory apparatus with non-hazardous soaps when possible, rather than solvents or acids which may be hazardous.

### **Regulatory Overview**

Federal waste disposal regulations were issued in accordance with the Resource Conservation and Recovery Act (RCRA). They are given in Title 40 Code of Federal Regulations (CFR) part 260. The Act controls all forms of solid waste disposal and encourages recycling and alternative energy sources. The major emphasis is controlling hazardous waste disposal. The regulations create a system to identify wastes and track waste generation, transport, and ultimate disposal. Each facility involved in managing hazardous waste must be registered with the USEPA. This includes the generator, transporters, and treatment, storage, and disposal facilities (TSDF).

Under federal regulations, there are three categories of generators with increasingly more strict regulation for larger quantity generators. The categories are based on the amount of hazardous waste generated in any given month.

## WASTE MANAGEMENT AND SAFETY, continued

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### The categories are as follows:

- Conditionally Exempt Small Quantity Generator - less than 100 kg (220 lb.) per month
- Small Quantity Generator - between 100 kg (220 lb.) and 1,000 kg (2,200 lb.) per month
- Large Quantity Generator - greater than 1,000 kg (2,200 lb.) per month

*Note: If a laboratory generates acutely hazardous waste (as defined on 40 CFR 261) or accumulates more than a certain amount of waste, the facility may be moved into a larger generator status. Check with your environmental compliance manager or state and local officials to determine which category your facility is in.*

### Hazardous Waste Definition

For regulatory purposes, a “hazardous waste” is a material which is subject to special laws by the USEPA under 40 CFR 261. In addition, many states or local authorities regulate additional materials as hazardous waste. Be aware that many very toxic compounds are not regulated by this definition of hazardous waste. However, improper management or disposal of these compounds may lead to legal problems under other laws such as CERCLA (Superfund) or common law torts.

The 40 CFR 261 defines a hazardous waste as a solid waste which is not excluded from regulation and meets any of the following criteria:

- It is a discarded commercial chemical product, off-specification species, container residue, or spill residue of materials specifically listed in 40 CFR 261.33;
- It is a waste from a specific source listed in 40 CFR 261.32;
- It is a waste from a non-specific source listed in 40 CFR 261.31; or
- It displays any of the following characteristics of hazardous waste defined in 40 CFR 261.20-24:
  - ignitability
  - corrosivity
  - reactivity
  - toxicity

There are many exceptions to these regulations, and each generator should review the regulations and determine if they are excluded from the regulations.

## WASTE MANAGEMENT AND SAFETY, continued

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### Characteristic Hazardous Waste Codes

Hazardous wastes are categorized by specific codes assigned in 40 CFR 261.20-261.33. These codes will help you identify hazardous waste. The generator is responsible for making the actual waste code determination.

Selected characteristic waste codes for chemicals which may be generated using Hach methods for water analysis are given in the following table. A complete list of waste codes is found in 40 CFR 261.24.

USEPA Code	Characteristic	CAS No.	Regulatory Level (mg/L)
D001	Ignitability	na	na
D002	Corrosivity	na	na
D003	Reactivity	na	na
D004	Arsenic	6440-38-2	5.0
D005	Barium	6440-39-3	100.0
D018	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-9	1.0
D022	Chloroform	67-66-3	6.0
D007	Chromium	7440-47-3	5.0
D008	Lead	7439-92-1	5.0
D009	Mercury	7439-97-6	0.2
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0

### How to Determine if Waste is Hazardous

Federal laws do not require you to test a material to decide if it is a hazardous waste. You may apply product knowledge to decide if a material is hazardous. Often, information on a material safety data sheet (MSDS) is enough to decide. If the product is specifically listed in the regulation, it is a hazardous waste.

You also need to decide if it has any characteristics of a hazardous waste. Physical information on the MSDS may help you decide. If the flash point is below 60 °F (15 °C) or is classified by DOT as an oxidizer, the material may be ignitable. If the pH of the material is  $\leq 2$  or  $\geq 12.5$ , the material may be corrosive. If the material is unstable, reacts violently with water, or may generate toxic gases, vapors, or fumes when mixed with water, it may be reactive.

## **WASTE MANAGEMENT AND SAFETY, continued**

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Use the chemical composition data to decide if a material is toxic. This decision is based on the concentration of certain contaminants (heavy metals and a number of organic compounds). If the waste is a liquid, compare the concentration of the contaminants in the liquid to the concentrations listed in 40 CFR 261.24. If the waste is a solid, analyze the sample by the Toxicity Characteristic Leachability Procedure (TCLP) and compare the results to the concentration listed in the 40 CFR 261.24. Levels above the threshold amount listed in the table are hazardous.

See “Sections of the MSDS” on page 63, describing the MSDS for help in finding information for making hazardous waste determinations.

### **Examples of Hazardous Waste**

A number of chemicals used in and final solutions created from Hach procedures are hazardous wastes when they are disposed. In addition, substances in the sample matrix may be a hazardous waste. Sometimes, reagents which would be hazardous are neutralized or changed during the analytical procedure. In that case, the final solutions are not regulated. Finally, many reagents and final solutions may be non-regulated. The generator must either use their knowledge of the materials used or conduct analytical tests to determine if the final material is a hazardous waste.

Examples of tests using Hach reagents that generate hazardous waste include those containing mercury or mercury compounds such as COD tests or Nessler’s reagent. Conversely, a test using Hach reagents such as ManVer 2 Hardness Indicator Powder Pillows and EDTA Titration Cartridges do not produce a hazardous waste unless the sample contains a hazardous substance.

### **Hazardous Waste Disposal**

Hazardous waste must be managed and disposed of according to federal, state, and local regulations. The waste generator is responsible for making hazardous waste determinations. Analysts should check with the facility’s environmental compliance people for specific instructions.

Hazardous wastes should be handled by treatment, storage, and disposal facilities (TSDF) that have USEPA permits. In some cases, the generator may treat the hazardous waste. In most cases, a permit from the USEPA is required to treat hazardous waste. Laboratories are not exempt from these regulations. If your facility is a “Conditionally Exempt Small Quantity Generator,” special rules may apply. Check 40 CFR 261 to determine if have to comply with all the laws.

The most common allowed treatment is elementary neutralization. This refers to neutralizing wastes that are hazardous only because they are corrosive or are listed only for that reason. Neutralize acidic solutions by adding a base such as sodium hydroxide; neutralize basic solutions by

## WASTE MANAGEMENT AND SAFETY, continued

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adding an acid such as hydrochloric acid. Slowly add the neutralizing agent while stirring. Monitor the pH. When it is at or near 7, the material is neutralized and may be flushed down the drain. Many wastes generated from Hach procedures may be treated in this manner.

Other chemical or physical treatments such as cyanide destruction or evaporation may require a permit. Check with your environmental department or local regulators to determine which rules apply to your work facility.

Laboratory chemicals may be mixed and disposed of with other hazardous wastes generated at your facility. They may also be accumulated in accordance with 40 CFR 262.34 satellite accumulation rules. After collection they may be disposed of in a "labpack." A number of environmental and hazardous waste companies offer labpacking services. They will inventory, sort, pack, and arrange proper disposal for hazardous waste. Find companies offering these services in the Yellow Pages under "Waste Disposal - Hazardous" or contact state and local regulators for assistance.

### Management of Specific Wastes

Hach has several documents to assist customers in managing waste generated from our products. You can obtain the following documents by calling 1-800-227-4224 or 970-669-3050 and requesting the literature codes given:

Literature Code	Title
1321	Waste Reduction: A Primer
9323	Mercury Waste Disposal Firms
9325	COD Waste Management
9326	COD Heavy Metal Total Concentrations

### Special Considerations for Cyanide-Containing Materials

Several procedures in this manual use reagents that contain cyanide compounds. These materials are regulated as reactive (D003) waste by the Federal RCRA. Waste disposal instructions provided with each procedure tell you how to collect these materials for proper disposal. It is imperative that these materials be handled safely to prevent the release of hydrogen cyanide gas (an extremely toxic material with the smell of bitter almonds). Most cyanide compounds are stable and can be safely stored for disposal in highly alkaline solutions (pH >11) such as 2 N sodium hydroxide. Never mix these wastes with other laboratory wastes that may contain lower pH materials such as acids or even water.

## WASTE MANAGEMENT AND SAFETY, *continued*

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If a cyanide-containing compound is spilled, you must be careful not to be exposed to hydrogen cyanide gas. Take the following steps to destroy the cyanide compounds in an emergency:

- a) Use a fume hood, supplied air or self-contained breathing apparatus.
- b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and either calcium hypochlorite or sodium hypochlorite (household bleach).
- c) Add an excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
- d) Neutralize the solution and flush it down the drain with a large amount of water. If the solution contains other regulated materials such as chloroform or heavy metals, it may still need to be collected for hazardous waste disposal. Never flush hazardous wastes down the drain.

### Resources

Many sources of information on proper waste management are available. The USEPA has a hotline number for questions about the Resource Conservation and Recovery Act (RCRA). The RCRA Hotline number is 1-800-424-9346. You may also get a copy of the appropriate regulations. Federal hazardous waste regulations are found in 40 CFR 260-99. Obtain this book from the U.S. Government Printing Office or a number of other vendors. Other documents which may be helpful to the laboratory hazardous waste manager include:

1. Task Force on Laboratory Waste Management. *Laboratory Waste Management, A Guidebook*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1994.
2. Task Force on Laboratory Waste Management. *Waste Management Manual for Laboratory Personnel*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1990.
3. Task Force on Laboratory Waste Management. *Less is Better*; 2nd ed.; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1993.
4. Committee on Chemical Safety. *Safety in Academic Chemistry Laboratories*, 5th ed.; American Chemical Society: Washington, DC, 1990.
5. Armour, Margaret-Ann. *Hazardous Laboratory Chemicals Disposal Guide*; CRC Press: Boca Raton, FL, 1991.

## WASTE MANAGEMENT AND SAFETY, continued

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6. *Environmental Health and Safety Manager's Handbook*; Government Institutes, Inc.: Rockville, MD, 1988.
7. Lunn, G; Sansone, E.B. *Destruction of Hazardous Chemicals in the Laboratory*; John Wiley and Sons: New York, 1990.
8. National Research Council. *Prudent Practices for Disposal of Chemicals from Laboratories*; National Academy Press: Washington, DC, 1983.
9. National Research Council. *Prudent Practices for Handling Hazardous Chemicals in Laboratories*; National Academy Press: Washington, DC, 1981.
10. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *The RCRA Orientation Manual*; U.S. Government Printing Office: Washington, DC, 1991.
11. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *Understanding the Small Quantity Generator Hazardous Waste Rules: A Handbook for Small Business*; U.S. Government Printing Office: Washington, DC, 1986.

### Material Safety Data Sheets

Material safety data sheets (MSDS) describe the hazards of chemical products. This section describes the information provided on a Hach MSDS and how to locate important information for safety and waste disposal. The information provided on the MSDS applies to the product as sold by Hach. The properties of any mixtures obtained by using this product will be different.

### How to Obtain an MSDS

Hach ships an MSDS to each customer with the first order of any chemical product. A new MSDS may be sent when the information on the data sheet is updated. Please review all new MSDS's for new information. If you need another copy of an MSDS, simply call 1-800-227-4227.

### Sections of the MSDS

Each MSDS has ten sections. The sections and the information found in them are described below.

### Header Information

The Hach catalog number, MSDS date, change number, company address and telephone number, and emergency telephone numbers are listed at the top of the MSDS.

## WASTE MANAGEMENT AND SAFETY, continued

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### 1 Product Identification

This section contains:

- Each product name
- Chemical Abstract Services (CAS) number
- Chemical name
- Chemical formula, if appropriate
- Chemical family to which the material belongs

### 2 Ingredients

This section lists each component in the product. It contains the following information for each component:

- PCT: Percent by weight of this component
- CAS NO.: Chemical Abstract Services (CAS) registry number for this component
- SARA: Superfund Amendments and Reauthorization Act, better known as the “Community Right to Know Law” tells you if the component is listed in SARA 313. If the component is listed and you use more than the amount listed, you must report this to the USEPA every year.
- TLV: Threshold Limit Value. The maximum airborne concentration for an 8 hour exposure that is recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).
- PEL: Permissible Exposure Limit. The maximum airborne concentration for an 8 hour exposure that is regulated by the Occupational Safety and Health Administration (OSHA).
- HAZARD: Physical and health hazards of the component are explained.

### 3 Physical Data

The physical properties of the product are given in this section. They include the physical state, color, odor, solubility, boiling point, melting point, specific gravity, pH, vapor density, evaporation rate, corrosivity, stability, and storage precautions.

## WASTE MANAGEMENT AND SAFETY, continued

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### 4 Fire, Explosion Hazard And Reactivity Data

This section contains the flash point and flammable limits of the material. It also includes how to fight fires if the material catches on fire. Key terms in this section include:

- Flashpoint: The temperature at which a liquid will give off enough flammable vapor to ignite.
- Flammability and ignitability are usually defined by the flash point.
- Lower Flammable Limit (LFL or LEL): The lowest concentration that will produce a fire or flash when an ignition source is present.
- Upper Flammable Limit (UFL or UEL): The vapor concentration in air above which the concentration is too rich to burn.
- NFPA Codes: The National Fire Protection Association (NFPA) has a system to rate the degree of hazards presented by a chemical. These codes are usually placed in a colored diamond. The codes range from 0 for minimal hazard to 4 for extreme hazard. They are grouped into the following hazards: health (blue), flammability (red), reactivity (yellow), and special hazards (white).

### 5 Health Hazard Data

This section describes different ways the chemical can enter your body (ingestion, inhalation, skin contact). It also gives acute (immediate) and chronic (long-term) health effects. If the material causes cancer or genetic damage, it is identified in this section.

### 6 Precautionary Measures

This section contains special precautions for the material. These may include special storage instructions, handling instructions, conditions to avoid, and protective equipment required to use this material safely.

### 7 First Aid

First aid instructions for exposures to the chemical are given in this section. Be sure to read this section before inducing vomiting in a victim. Some chemicals are better treated by not inducing vomiting. Seek prompt medical attention for all chemical exposures.

### 8 Spill And Disposal Procedures

This section tells about safe work practices for cleaning up and disposing of spilled material. Please refer to the Waste Management section of this manual. Final determination of proper and legal disposal options is the responsibility of the waste generator. Be sure you know the federal, state, and local laws that apply to your facility.

## WASTE MANAGEMENT AND SAFETY, continued

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### 9 Transportation Data

Domestic and International shipping information is provided in this section. It gives shipping name, hazard class, and ID number of the product.

### 10 References

This section lists the reference materials used to write the MSDS.

Following the Reference section, the product is listed as having SARA 313 chemicals or California Proposition 65 List Chemicals, if applicable. Also found here is any special information about the product.

### Safety

Safety is the responsibility of each person performing analytical procedures. Because many of the procedures in this methods manual use potentially hazardous chemicals and equipment, it is important to prevent accidents by practicing good laboratory techniques. The following guidelines apply to water analysis. These guidelines do not cover every aspect of safety, but they are important for preventing injuries.

### Material Safety Data Sheet

A material safety data sheet (MSDS) comes with the first shipment of all products. The MSDS provides environmental and safety information about the products. Always read the MSDS before using a new product.

### Reading Labels Carefully

Read each reagent label carefully. Pay particular attention to the precautions given. Never remove or block the label on a reagent container while it contains reagent. Do not put a different reagent into a labeled container without changing the label. When preparing a reagent or standard solution, label the container clearly. If a label is hard to read, re-label promptly according to your facility's hazard communication program.

Warning labels also appear on some of the apparatus used with the test procedures. The protective shields with the COD Reactor and the Digesdahl Digestion Apparatus point out potential hazards. Be sure these shields are in place during use and observe the precautions on the label.

### Protective Equipment

Use the right protective equipment for the chemicals and procedures. The MSDS contains this information. Protective equipment may include:

- Eye protection such as safety glasses or goggles to protect from flying objects or chemical splashes.
- Gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials, or broken glass. Use tongs or finger cots when transferring hot apparatus.

## WASTE MANAGEMENT AND SAFETY, continued

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- Laboratory coats or splash aprons to protect skin and clothing from splashes.
- Footwear to protect feet from spills. Open toed shoes should not be worn in chemistry settings.
- Respirators may be needed to protect you from breathing toxic vapors if adequate ventilation, such as fume hoods, are not available.
- Use fume hoods as directed by the procedure or as recommended in the MSDS.
- For many procedures, adequate ventilation is enough. Be sure there is enough fresh air and air exhaust to protect against unnecessary exposure to chemicals.

### First Aid Equipment and Supplies

Most first aid instructions for chemical splashes in eyes or on skin call for thorough flushing with water. Laboratories should have eyewash and shower stations. For field work, carry a portable eyewash unit. Laboratories should also have appropriate fire extinguishers and fume hoods.

### General Safety Rules

Follow these rules to make work with toxic and hazardous chemicals safer:

1. **Never** pipet by mouth. Always use a mechanical pipet or pipet bulb to avoid ingesting chemicals.
2. Follow test procedures carefully and observe all precautionary measures. Read the entire procedure carefully before beginning.
3. Wipe up all spills promptly. Get proper training and have the right response equipment to clean up spills. See your safety director for more information.
4. **Do not** smoke, eat, or drink in an area where toxic or irritating chemicals are used.
5. Use reagents and equipment only as directed in the test procedure.
6. **Do not** use damaged labware and broken equipment.
7. Minimize all chemical exposures. **Do not** breathe vapors or let chemicals touch your skin. Wash your hands after using chemicals.
8. Keep work areas **neat and clean**.

## **WASTE MANAGEMENT AND SAFETY, continued**

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**9. Do not** block exits or emergency equipment.

### **OSHA Chemical Hygiene Plan**

The Occupational Safety and Health Administration (OSHA) enforces laws about the control exposure to hazardous chemicals in laboratories. These regulations are in Title 29 CFR 1910.1450. They apply to all employers who use hazardous chemicals. They require employers to develop and use a written Chemical Hygiene Plan and appoint a qualified person as the Chemical Hygiene Officer.

## **SECTION 4 PROCEDURES**

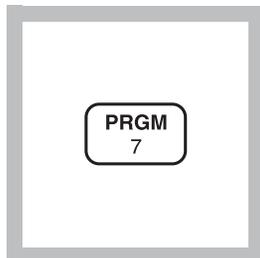
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**ALUMINUM (0 to 0.80 mg/L)**

For water and wastewater

**Aluminon Method\***

**1.** Enter the stored program number for aluminum (Al).

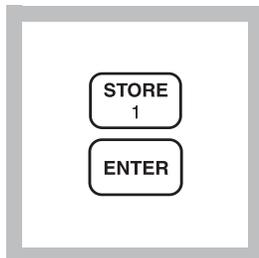
Press: **PRGM**

The display will show:

**PRGM ?**

*Note:* Adjust the pH of stored samples before analysis.

*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

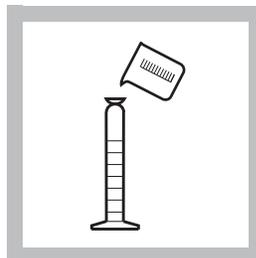


**2.** Press: **1 ENTER**

The display will show **mg/L, Al** and the **ZERO** icon.

*Note:* Total aluminum determination requires a digestion prior to analysis (see Section 2).

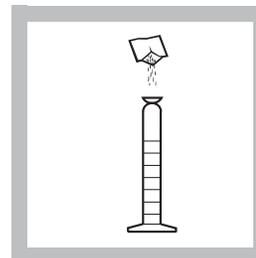
*Note:* For alternate form ( $Al_2O_3$ ), press **CONC**.



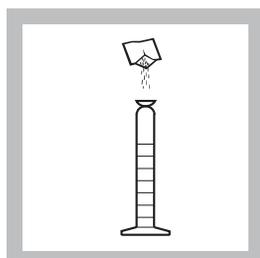
**3.** Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.

*Note:* Rinse cylinder with 1:1 Hydrochloric Acid and deionized water before use to avoid errors due to contaminants absorbed on the glass.

*Note:* Sample temperature must be 20-25 °C (68-77 °F) for accurate results.



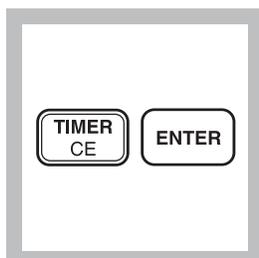
**4.** Add the contents of one Ascorbic Acid Powder Pillow. Stopper. Invert several times to dissolve powder.



**5.** Add the contents of one AluVer<sup>®</sup> 3 Aluminum Reagent Powder Pillow. Stopper.

*Note:* A red-orange color develops if aluminum is present.

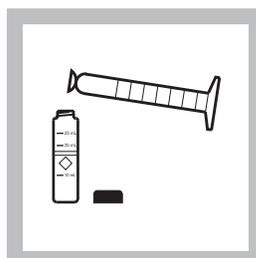
*Note:* Inconsistent results will occur if any powder is undissolved.



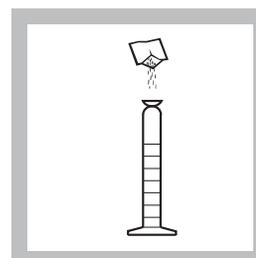
**6.** Press:

**TIMER ENTER**

A three-minute reaction period will begin. Invert the cylinder repeatedly for the three minutes.



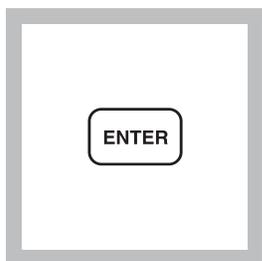
**7.** Pour 25 mL of mixture into a 25-mL sample cell (the prepared sample).



**8.** Add the contents of one Bleaching 3 Reagent Powder Pillow to the remaining 25 mL in the mixing graduated cylinder (the blank). Stopper the cylinder.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

## ALUMINUM, continued

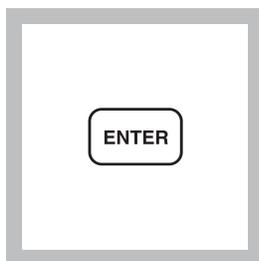


**9.** The display will show: **00:30 Timer 2**  
Press: **ENTER**  
A thirty-second reaction period will begin. Vigorously shake the cylinder for the 30-second period.

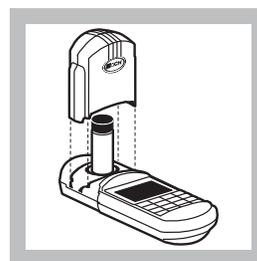
*Note: This solution should turn a light to medium orange upon bleaching. It will not become colorless.*



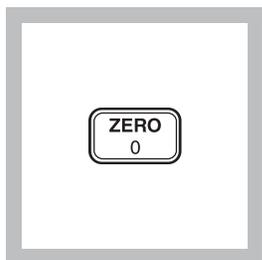
**10.** Pour the 25 mL of mixture in the cylinder into a second 25-mL sample cell (the blank).



**11.** The display will show: **15:00 TIMER 3**  
Press: **ENTER**  
A 15-minute reaction period will begin.



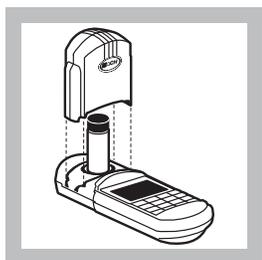
**12.** Within three minutes after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



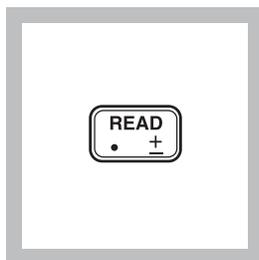
**13.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.000 mg/L Al**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**14.** Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**15.** Press: **READ**  
The cursor will move to the right, then the result in mg/L aluminum will be displayed.

*Note: Clean the graduated cylinder and sample cells with soap and brush immediately following the test.*

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

## ALUMINUM, continued

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### Sampling and Storage

Collect samples in a clean glass or plastic container. Preserve the sample by adjusting the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 3.5–4.5 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off an Aluminum Voluette Ampule Standard Solution, 50 mg/L as Al.
- b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to three 50-mL samples. Swirl gently to mix. Also prepare a sample without any standard added (the unspiked sample).
- c) Analyze each sample as described above. The aluminum concentration should increase 0.1 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

#### Standard Solution Method

Prepare a 0.40-mg/L aluminum standard solution by pipetting 1.00 mL of Aluminum Standard Solution, 100 mg/L as Al<sup>3+</sup>, into a 250-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution immediately before use. Perform the aluminum procedure as described above. The mg/L Al reading should be 0.40 mg/L Al.

Or, using the TenSette Pipet, add 0.8 mL of solution from an Aluminum Voluette Ampule Standard Solution (50 mg/L as Al) into a 100-mL volumetric flask. Dilute to volume with deionized water. Prepare this standard immediately before testing and use as the sample.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.40 mg/L Al and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.013$  mg/L Al.

## ALUMINUM, continued

### Estimated Detection Limit

The estimated detection limit for program #1 is 0.013 mg/L Al. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Acidity interferes at greater than 300 mg/L as CaCO <sub>3</sub> . Treat samples with greater than 300 mg/L acidity as CaCO <sub>3</sub> as follows: <ol style="list-style-type: none"> <li>1. Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3.</li> <li>2. Add one drop of 5.0 N Sodium Hydroxide Standard Solution. Stopper the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow.</li> <li>3. Add one drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow back to colorless. Continue with the test.</li> </ol>
Alkalinity	1000 mg/L as CaCO <sub>3</sub> . Eliminate interferences from higher alkalinity concentrations using the following pretreatment: <ol style="list-style-type: none"> <li>1. Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3. A yellow color indicates excessive alkalinity.</li> <li>2. Add one drop of 5.25 N Sulfuric Acid Standard Solution. Stopper the cylinder. Invert to mix. If the yellow color persists, repeat until the sample becomes colorless. Continue with the test.</li> </ol>
Calcium	Does not interfere.
Fluoride	Interferes at all levels. See graph below.
Iron	Greater than 20 mg/L.
Phosphate	Greater than 50 mg/L.
Polyphosphate	Polyphosphate interferes at all levels by causing negative errors and must not be present. Before running the test, polyphosphate must be converted to orthophosphate by acid hydrolysis as described under the phosphorus procedures.

Fluoride interferes at all levels by complexing with aluminum. The actual aluminum concentration can be determined using the Fluoride Interference Graph when the fluoride concentration is known. To use the fluoride interference graph:

## ALUMINUM, continued

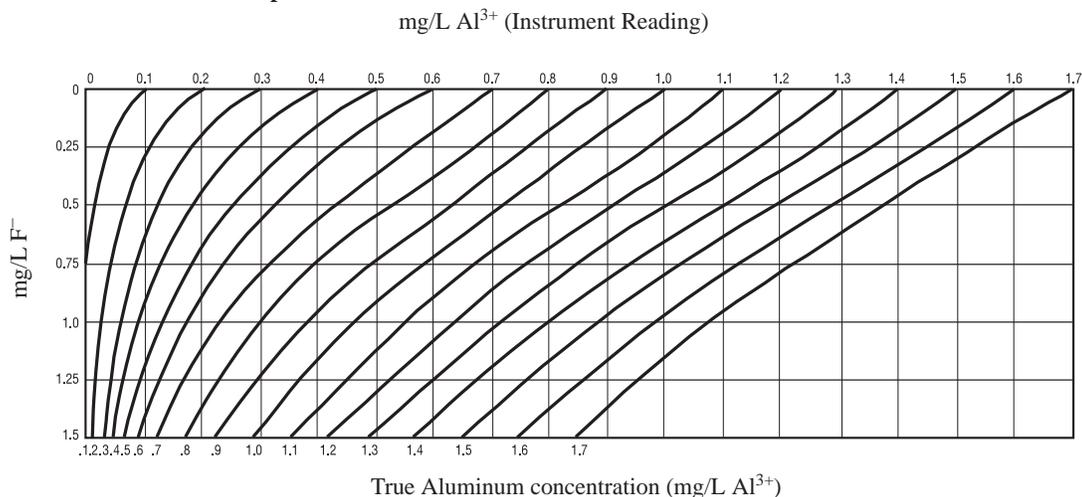
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1. Select the vertical grid line along the top of the graph that represents the aluminum reading obtained in Step 15.
2. Locate the point of the vertical line (instrument reading) where it intersects with the horizontal grid line that indicates how much fluoride is present in the sample.
3. Extrapolate the true aluminum concentration by following the curved lines on either side of the intersect point down to the true aluminum concentration.

For example, if the aluminum test result was 0.7 mg/L Al<sup>3+</sup> and the fluoride present in the sample was 1.0 mg/L F<sup>-</sup>, the point where the 0.7 grid line intersects with the 1.0 mg/L F<sup>-</sup> grid line falls between the 1.2 and 1.3 mg/L Al curves. In this case, the true aluminum content would be 1.27 mg/L.

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### Fluoride Interference Graph



### Summary of Method

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminum Reagent, packaged in powder form shows exceptional stability and is applicable for fresh water samples.

## ALUMINUM, continued

### REQUIRED REAGENTS

	Cat. No.
Aluminum Reagent Set (100 Tests) .....	22420-00
Includes: (1) 14290-99, (1) 14577-99, (1) 14294-49	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
AluVer 3 Aluminum Reagent Powder Pillow .....	1 pillow .....	100/pkg.....	14290-99	
Ascorbic Acid Powder Pillow.....	1 pillow .....	100/pkg.....	14577-99	
Bleaching 3 Reagent Powder Pillow .....	1 pillow .....	100/pkg.....	14294-49	

### REQUIRED APPARATUS

Cylinders, graduated mixing, 50 mL .....	1 .....	each.....	1896-41
Sample Cell, 10-20-25 mL, w/ cap.....	2 .....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

Aluminum Standard Solution, 100 mg/L.....	100 mL.....	14174-42
Aluminum Standard Solution, Voluette ampule, 50 mg/L as Al, 10 mL.....	16/pkg.....	14792-10
Hydrochloric Acid Solution, 6N (1:1) .....	500 mL.....	884-49
m-Nitrophenol Indicator Solution, 10 g/L .....	100 mL.....	2476-32
Nitric Acid, ACS.....	500 mL.....	152-49
Nitric Acid Solution, 1:1 .....	500 mL.....	2540-49
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB.....	2450-32
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL SCDB.....	2450-26
Sulfuric Acid Standard Solution, 5.25 N .....	100 mL MDB.....	2449-32
Water, deionized.....	4 L.....	272-56

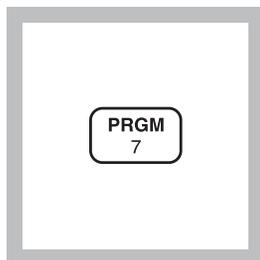
### OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....	21968-00
Brush.....	each.....	690-00
Flask, volumetric, Class A, 100 mL .....	each.....	14574-42
Flask, volumetric, Class A, 250 mL .....	each.....	14574-46
Fluoride Combination Electrode.....	each.....	51928-00
Fluoride ISA Powder Pillows .....	25/pkg.....	2589-99
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg.....	391-33
pH/ISE Meter, <i>sension</i> <sup>TM</sup> 2, portable.....	each.....	51725-00
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
Pipet, Volumetric, Class A, 1.00 mL .....	each.....	14515-35
Thermometer, -20 to 110 °C, non-mercury .....	each.....	26357-02

#### **For Technical Assistance, Price and Ordering**

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**BENZOTRIAZOLE (0 to 16.0 mg/L) or TOLYLTRIAZOLE (0 to 16.0 mg/L)****UV Photolysis Method\*****For cooling or boiler water**

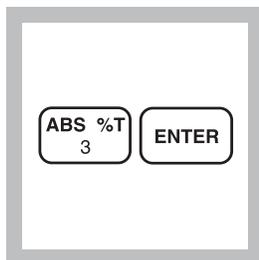
**1.** Enter the stored program number for benzotriazole (Benzo) or tolyltriazole (Toly).

Press: **PRGM**

The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



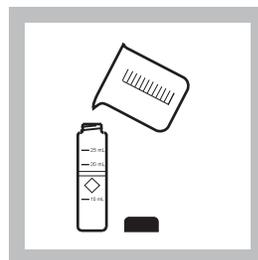
**2.** Press: **3 ENTER** for either triazole test.

The display will show **mg/L, BENZO**, and the **ZERO** icon

or

the display will show **mg/L, TOLY**, and the **ZERO** icon.

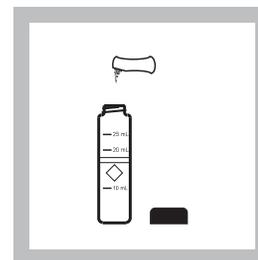
Press the **CONC** key to choose the desired triazole.



**3.** Fill a sample cell with 25 mL of sample.

*Note: Sample temperature should be between 20-25 °C (68-77 °F).*

*Note: If sample contains nitrite or borax (sodium borate), adjust the pH to between 4 and 6 with 1 N sulfuric acid.*

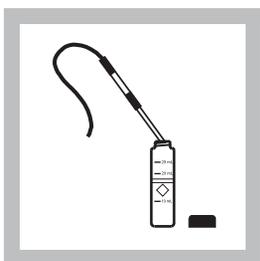


**4.** Add the contents of one Triazole Reagent Powder Pillow. Swirl to dissolve completely.

*Note: If the sample contains more than 500 mg/L hardness (as CaCO<sub>3</sub>), add 10 drops of Rochelle Salt Solution.*

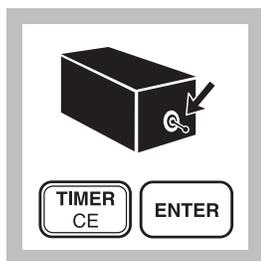
\* Adapted from Harp, D., Proceedings 45th International Water Conference, 299 (October 22-24, 1984)

## BENZOTRIAZOLE OR TOLYLTRIAZOLE, continued



**5.** Insert the ultraviolet lamp into the sample cell.

*Note: UV safety goggles should be worn while the lamp is on.*

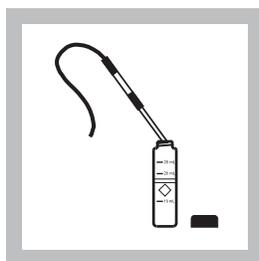


**6.** Turn the UV lamp ON and press:

**TIMER ENTER**

A five-minute reaction period will begin.

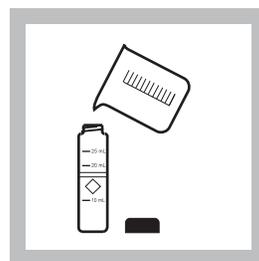
*Note: A yellow color will form if triazole is present.*



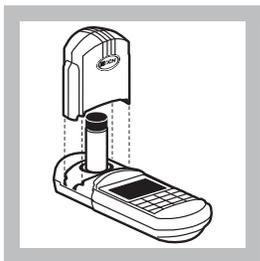
**7.** When the timer beeps, turn the lamp off and remove it from the cell (the prepared sample). Swirl the cell to mix thoroughly.

*Note: Low results will occur if photolysis (lamp ON) takes place for more or less than five minutes.*

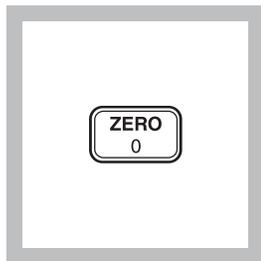
*Note: Avoid handling the quartz surface of the lamp. Rinse the lamp and wipe with a soft, clean tissue between tests.*



**8.** Fill another sample cell with 25 mL of sample (the blank).



**9.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



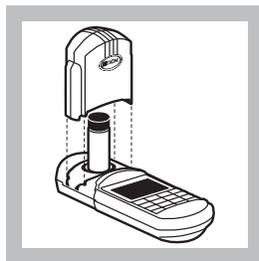
**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.0 mg/L Benzo**

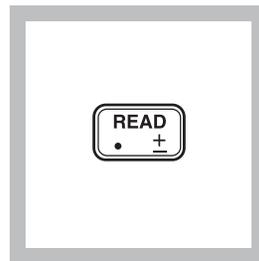
or

**0.0 mg/L Toly**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L benzotriazole or tolyltriazole will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

## BENZOTRIAZOLE OR TOLYLTRIAZOLE, continued

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### Sampling And Storage

The most reliable results are obtained when samples are analyzed as soon as possible after collection.

### Accuracy Check

#### Standard Additions Method

- a) Use the TenSette pipet to add 0.1, 0.2 and 0.3 mL of 500-mg/L Benzotriazole Standard Solution to three 25-mL samples. Perform the test according to the above procedure.

*Note:* The test will not distinguish between benzotriazole and tolyltriazole.

- b) Each addition of 0.1 mL of standard solution should increase the benzotriazole reading by 2 mg/L over the reading of an unspiked sample.
- c) If these increases are not obtained see *Standard Additions* in *Section 1* for more information.

### UV Lamp Check

To verify the ultraviolet lamp (normal life equals 5000 hours) is working properly, perform the following test:

- a) Prepare a 5.0 mg/L benzotriazole standard solution by pipetting 10.0 mL of Benzotriazole Standard Solution, 500 mg/L benzotriazole, into a 1000-mL volumetric flask. Dilute to volume.
- b) Analyze according to the above procedure. If the result is significantly below 5.0 mg/L, replace the lamp.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 9.0 mg/L triazole and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.21$  mg/L benzotriazole and  $\pm 0.20$  mg/L tolyltriazole.

#### Estimated Detection Limit

The estimated detection limit for program 3 is 0.7 mg/L benzotriazole or tolyltriazole. For more information on the estimated detection limit, see *Section 1*.

## BENZOTRIAZOLE OR TOLYLTRIAZOLE, continued

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### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Acrylates (as methyl acrylate)	50 mg/L
Alum	400 mg/L
Borate (as sodium tetraborate)	4000 mg/L
Chlorine (as Cl <sub>2</sub> )	20 mg/L
Chromium (as chromate)	12 mg/L
Copper	10 mg/L
Hardness	500 mg/L as CaCO <sub>3</sub>
Iron	20 mg/L
Lignosulfonates	40 mg/L
Magnesium	300 mg/L as CaCO <sub>3</sub>
Molybdenum (as molybdate)	200 mg/L
Nitrite	4000 mg/L
Phosphonates (AMP or HEDP)	100 mg/L
Sulfate	200 mg/L
Zinc	80 mg/L

Strong oxidizing or reducing agents present in the sample will interfere directly with the test.

### Summary of Method

Benzotriazole or tolyltriazole, used in many applications as corrosion inhibitors for copper and copper alloys, are determined by a proprietary catalytic ultraviolet (UV) photolysis procedure requiring less than 10 minutes to perform.

## BENZOTRIAZOLE OR TOLYLTRIAZOLE continued

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### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Triazole Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	21412-99	

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06
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#### Select one based on available voltage:

Lamp, UV, with power supply, 115 V, 60 Hz, with goggles.....	1 .....	each .....	20828-00
Lamp, UV, with power supply, 230 V, 50 Hz, with goggles.....	1 .....	each .....	20828-02

### OPTIONAL REAGENTS

Benzotriazole Standard Solution, 500 mg/L .....	100 mL .....	21413-42
Rochelle Salt Solution.....	29 mL* DB .....	1725-33
Sulfuric Acid Standard Solution, 1.00 N.....	100 mL MDB .....	1270-32
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Flask, volumetric, Class A, 1000 mL.....	each .....	14574-53
Lamp, UV (lamp only).....	each .....	26710-00
pH Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>sension</i> <sup>TM</sup> <i>I</i> , portable with electrode.....	each .....	51700-10
Pipet Filler, safety bulb .....	each .....	14651-00
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, volumetric, 10.0 mL, Class A .....	each .....	14515-38
Safety Goggles, UV.....	each .....	21134-00
Stopwatch .....	each .....	14645-00
Thermometer, -20 to 110 °C, non-mercury .....	each .....	26357-02
Timer, interval, 1 second to 99 hours .....	each .....	23480-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

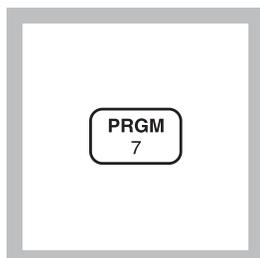
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\* Contact Hach for larger sizes.



**BROMINE (0 to 4.50 mg/L)**

For water, wastewater, and seawater

**DPD Method\*** (Powder Pillows or AccuVac Ampuls)**Using Powder Pillows**

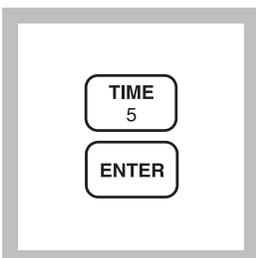
**1.** Enter the stored program number for bromine (Br<sub>2</sub>)-powder pillows.

Press: **PRGM**

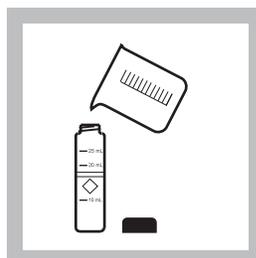
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

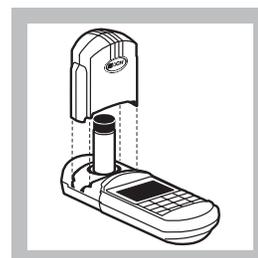


**2.** Press: **5 ENTER**  
The display will show **mg/L, Br2** and the **ZERO** icon.



**3.** Fill a sample cell with 10 mL of sample (the blank).

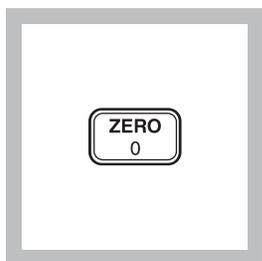
*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

## BROMINE, continued

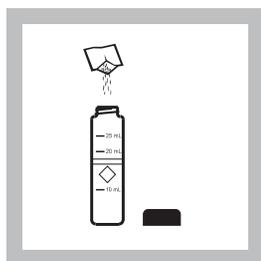


### 5. Press: ZERO

The cursor will move to the right, then the display will show:

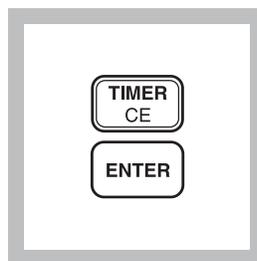
**0.00 mg/L Br<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



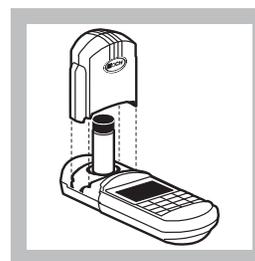
6. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

*Note: It is not necessary that all the powder dissolves. A pink color will develop if bromine is present.*

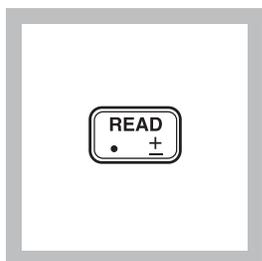


### 7. Press: TIMER ENTER

A three-minute reaction period will begin.



8. When the timer beeps, place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



### 9. Press: READ

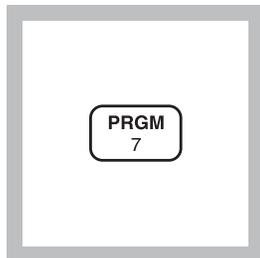
The cursor will move to the right, then the result in mg/L bromine will be displayed.

*Note: If samples temporarily turn yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute fresh samples and repeat the test. A slight loss of bromine may occur during dilution. Multiply results by the dilution factor; see Section 1.*

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

## BROMINE, continued

### Using AccuVac Ampuls



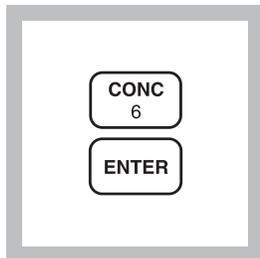
1. Enter the stored program number for bromine (Br<sub>2</sub>) AccuVac Ampuls.

Press: **PRGM**

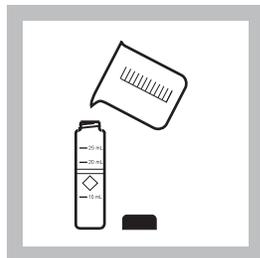
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

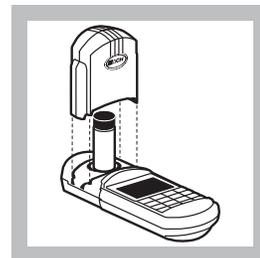


2. Press: **6 ENTER**  
The display will show **mg/L, Br<sub>2</sub>** and the **ZERO** icon.

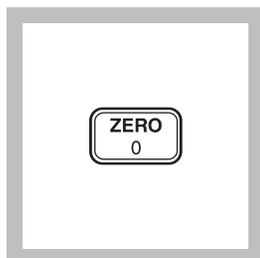


3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



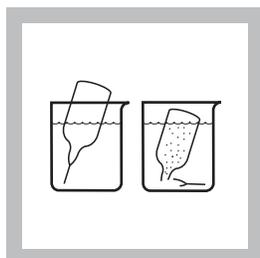
4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

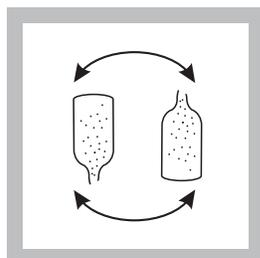
**0.00 mg/L Br<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



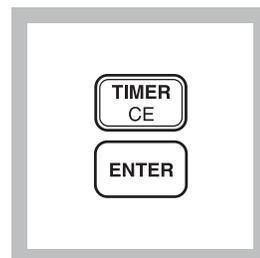
6. Fill one DPD Total Chlorine Reagent AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

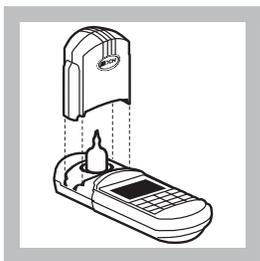
*Note: A pink color will form if bromine is present.*



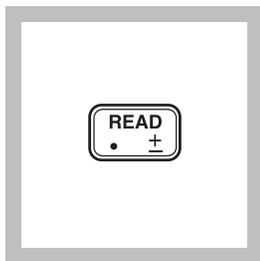
8. Press: **TIMER ENTER**  
A three-minute reaction period will begin.

## BROMINE, continued

---



**9.** After the timer beeps, place the AccuVac ampuL into the cell holder. Tightly cover the ampule with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

*Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute a fresh sample and repeat the test. A slight loss of bromine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

---

## Sampling and Storage

Analyze samples for bromine **immediately** after collection.

**Avoid plastic containers** since these may have a large bromine demand. **Pretreat glass** sample containers to remove any bromine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for bromine is introduced when a representative sample is not obtained. If sampling from a tap, let the sample flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark.

## BROMINE, continued

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Perform the bromine analysis immediately after collection.

### Accuracy Check

#### Standard Additions Method (using powder pillows)

- a) Snap the top off a LR Chlorine PourRite<sup>®</sup> Ampule Standard Solution.
- b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Re-zero the instrument using the original sample (the blank).
- d) Place the spiked sample in the cell holder and press **READ**. Record the result.
- e) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.1 (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{10.1 (\text{sample} + \text{standard volume})}$$

- f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Br<sub>2</sub> added (step e).
- g) If this increase does not occur, see *Standard Additions in Section 1* for more information.

#### Standard Additions Method (using AccuVac Ampuls)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.
- c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.
- d) Fill a DPD Total Chlorine AccuVac completely from each beaker.
- e) Analyze the spiked and unspiked sample as described in the procedure.
- f) Calculate the equivalent concentration of mg/L bromine added to the sample:

## BROMINE, continued

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$$\text{mg/L Bromine added} = \frac{0.2 (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{25.2 (\text{sample} + \text{standard volume})}$$

- g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Br<sub>2</sub> added (step f).
- h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 2.34 mg/L bromine and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L bromine.

In a single laboratory using a standard solution of 2.31 mg/L bromine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation ± 0.02 mg/L bromine.

#### Estimated Detection Limit

The estimated detection limit for program 5 is 0.04 mg/L Br<sub>2</sub> and 0.03 mg/L Br<sub>2</sub> for program 6. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## BROMINE, continued

### Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Chlorine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, Oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium , Oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6-7.</li> <li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>3. Mix and wait 1 minute.</li> <li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>5. Analyze 10 mL of the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct bromine concentration.</li> </ol>
Monochloramine	Interferes at all levels
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences, Section 1</i> .

### Summary of Method

Bromine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a magenta color which is proportional to the total bromine concentration.

## BROMINE, continued

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### Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interference will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

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### REQUIRED REAGENTS (USING POWDER PILLOWS)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
DPD Total Chlorine Reagent Powder Pillows .....	1 pillow .....	100/pkg.....	21056-69	

### REQUIRED REAGENTS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls .....	1 ampule .....	25/pkg.....	25030-25
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### REQUIRED APPARATUS (USING POWDER PILLOWS)

Sample Cells, 10-20-25-mL, w/ cap .....	6/pkg.....	24019-06
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### REQUIRED APPARATUS (USING ACCUVAC AMPULS)

Beaker, 50 mL.....	1 .....	each.....	500-41
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### OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL .....	20/pkg.....	26300-20
DPD Total Chlorine Reagent, SwifTest .....	250 Tests.....	28024-00
Potassium Iodide Solution, 30 g/L.....	100 mL* MDB.....	343-32
Sodium Arsenite, 5 g/L.....	100 mL* MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1.000 N .....	100 mL* MDB.....	1045-32
Sulfuric Acid Standard Solution, 1 N .....	100 mL* MDB.....	1270-32
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each.....	24052-00
PourRite Ampule Breaker.....	each.....	24846-00
Cylinder, graduated, 25 mL .....	each.....	508-40
pH Meter, <i>sensio</i> <sup>TM</sup> <b>I</b> , portable .....	each.....	51700-00
pH Indicator Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28

### ***For Technical Assistance, Price and Ordering***

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

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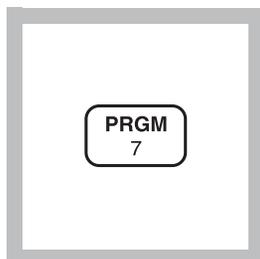
\* Contact Hach for larger sizes

# CHLORAMINE, MONO, Low Range (0–4.50 mg/L Cl<sub>2</sub>)

Method 10171

Indophenol Method\*

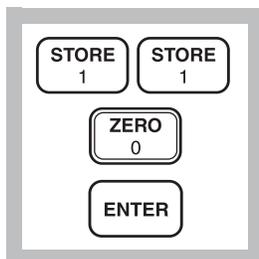
For chlorinated drinking water and chlorinated wastewater



1. Enter the user program number for monochloramine.

Press: **PRGM**

The display will show:  
**PRGM?**



2. Press:

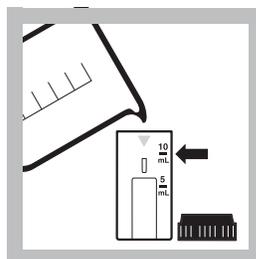
**110 ENTER**

The display will show

**mg/L Cl<sub>2</sub>**

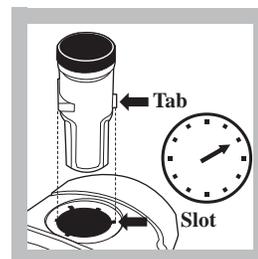
then: **ZERO**

*Note:* For alternate forms, press the **CONC** key.



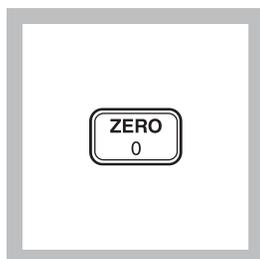
3. Fill the 10-mL/1-cm cell to the 10-mL line with sample.

*Note:* For the most accurate results, determine a reagent blank for each new lot of reagent by running the test using deionized water instead of sample.



4. Place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

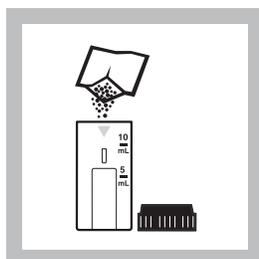
*Note:* Place the cell into the cell holder as illustrated. The cell's tab should be at the 2 o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.



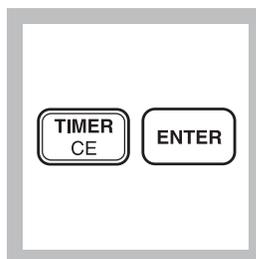
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L Cl<sub>2</sub>**



6. Remove the cell from the cell holder and add the contents of one pillow of Monochlor-F to the sample. Cap and shake the cell about 20 seconds to dissolve.

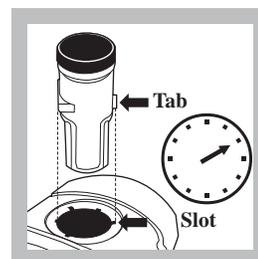


7. Press:

**TIMER ENTER**

A 5-minute reaction period will begin.

*Note:* The color development time depends on the sample temperature. Refer to Table 3 for the actual time required.



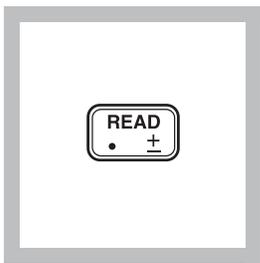
8. After the timer beeps, place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

*Note:* Place the cell into the cell holder as illustrated. The cell's tab should be at the 2-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

\* Patent pending

## CHLORAMINE, MONO, Low Range, continued

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### 9. Press: **READ**

The cursor will move to the right, then the result in mg/L monochloramine (as Cl<sub>2</sub> or chosen units) will be displayed.

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### Sampling and Storage

Analyze samples for monochloramine immediately after collection. If sampling with the sample cell, rinse the sample cell several times with the sample, then carefully fill to the 10-mL mark. If sampling from a tap, let the water flow for at least 5 minutes. Let the container overflow with the sample several times, then cap the container so there is no headspace (air) above the sample.

### Accuracy Check

1. Prepare the following monochloramine standard fresh before use.
2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.
3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH<sub>3</sub>-N into the flask.
4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.
5. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

## CHLORAMINE, MONO, Low Range, continued

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6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.
7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:  
$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$
8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.
9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.
10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl<sub>2</sub>) monochloramine standard.

Use this standard within 1 hour of preparation.

### Method Performance

#### Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl<sub>2</sub> and representative lots of reagent, a single operator obtained a standard deviation of ±0.12 mg/L Cl<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl<sub>2</sub>. For more information on the estimated detection limit, see *Section 1* of the *Procedure Manual*.

### Interferences

The following have been tested for interference and found *not* to interfere up to the indicated levels:

**Table 9 Non-interfering Substances**

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br <sup>-</sup>

## CHLORAMINE, MONO, Low Range, continued

Table 9 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Bromine	15 mg/L Br <sub>2</sub>
Calcium	1000 mg/L CaCO <sub>3</sub>
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO <sub>2</sub>
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN <sup>-</sup>
Free chlorine	10 mg/L Cl <sub>2</sub>
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L as N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO <sub>4</sub> <sup>3-</sup>
Silica	100 mg/L SiO <sub>2</sub>
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO <sub>3</sub> <sup>2-</sup>
Tyrosine	1 mg/L N
Urea	10 mg/L N
Zinc	5 mg/L

Table 10 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Magnesium	+	Above 400 mg/L CaCO <sub>3</sub>	Add 5 drops Rochelle Salt Solution prior to testing.
Manganese (+7)	-	Above 3 mg/L	
Ozone	-	Above 1 mg/L	Usually doesn't coexist with monochloramine.
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine.
Thiocyanate	-	Above 0.5 mg/L	

## CHLORAMINE, MONO, Low Range, continued

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### Summary of Method

In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample.

Sample Temperature		Minutes
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	4
20	73	3
23	75	2.5
25	77	2
>25	>77	2

### Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time, you may use the arrow keys to scroll back to review or change a number already entered.

## CHLORAMINE, MONO, Low Range, continued

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Line Number	Entry	Line Number	Entry
1	110	29	108
2	42	30	78
3	74	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	57
9	0	37	199
10	0	38	104
11	0	39	62
12	64	40	74
13	176	41	61
14	120	42	45
15	106	43	1
16	0	44	204
17	0	45	0
18	0	46	5
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	3
27	50	55	0
28	67	56	255

## CHLORAMINE, MONO, Low Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Monochlor F Reagent Pillows.....	1.....	50/pkg.....	28022-46

### REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm.....	1.....	2/pkg.....	48643-02
Clippers, shears .....	1.....	each.....	23694-00

### OPTIONAL REAGENTS

Rochelle Salt Solution .....	29-mL DB .....	1725-33
Organic-Free Water .....	500-mL.....	26415-49
Buffer Powder Pillows, pH 8.3 .....	25/pkg.....	898-68
Nitrogen, Ammonia Standard Solution, 100 mg/L as NH <sub>3</sub> -N .....	500-mL.....	24065-49
Chlorine Solution Voluette Ampule, 50–75 mg/L .....	16/pkg.....	14268-10

### OPTIONAL APPARATUS

Beaker, 100-mL.....	each.....	500-42H
Flask, Volumetric, Class A, 100-mL .....	each.....	14574-42
Pipet, Mohr, Glass, 10-mL .....	each.....	20934-38
Pipet, Volumetric, Class A, 2.00 mL.....	each.....	14515-36
Pipet, Volumetric, Class A, 50.00 mL.....	each.....	14515-41
Stir Bar, Octagonal .....	each.....	20953-52
Stirrer, Magnetic, 110 V, 4" x 4" .....	each.....	28812-00

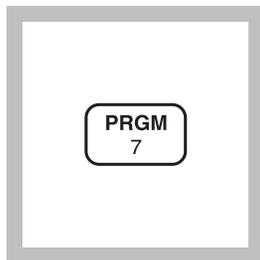


# CHLORAMINE, MONO, High Range (0–10.0 mg/L Cl<sub>2</sub>)

Method 10172

## Indophenol Method\*

For chlorinated drinking water and chlorinated wastewater

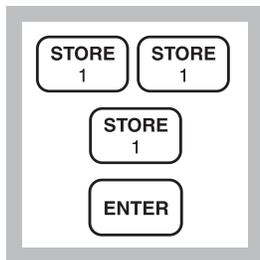


**1.** Enter the user program number for Chloramine, HR.

Press: **PRGM**

The display will show: **PRGM?**

*Note: For most accurate results, perform a Reagent Blank Correction (Section 1 of the DR/800 Instrument Manual).*



**2.** Press: **111 ENTER**

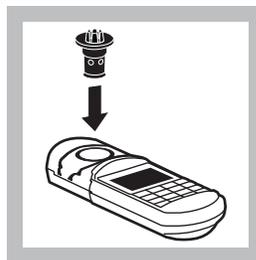
The display will show:

**mg/L Cl<sub>2</sub>**

and then

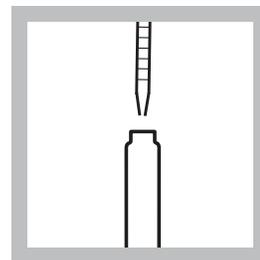
**Zero**

*Note: For alternate forms, press the **CONC** key.*

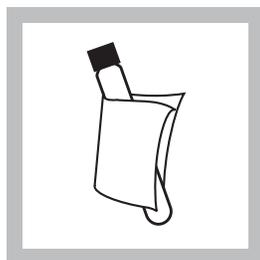


**3.** Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops in place. Push down to fully insert it.

*Note: For better performance, a diffuser band covers the light path holes on the adapter. Do not remove the band.*

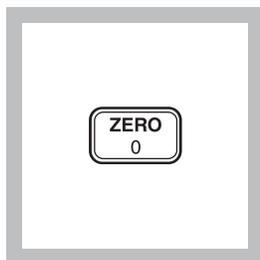


**4.** Remove the cap from one HR Monochloramine Diluent vial. Use a glass pipet to add 2.0 mL of sample to the vial. Re-cap and invert several times to mix.



**5.** Wipe the outside of the vial clean.

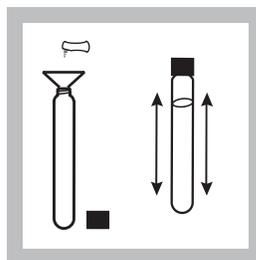
Place the vial into the adapter. Cover the sample vial tightly with the instrument cap.



**6.** Press: **ZERO**

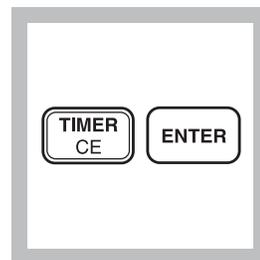
The cursor will move to the right and the display will show:

**0.0 mg/L Cl<sub>2</sub>**



**7.** Remove the vial from the cell holder, uncap, and add the contents of one Monochlor-F pillow to the sample. Cap and shake the vial about 20 seconds to dissolve.

*Note: Use the microfunnel as an aid in adding reagent powder to the vial.*



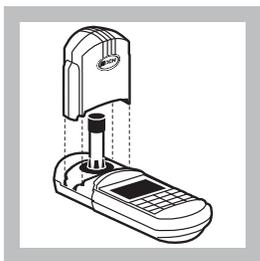
**8.** Press: **TIMER ENTER**

A five-minute reaction period will begin.

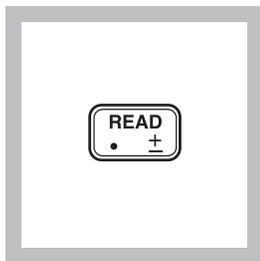
\* U.S. Patent 6,315,950

## CHLORAMINE, MONO, High Range, continued

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**9.** After the timer beeps, wipe the prepared vial and place it into the instrument. Cover the sample vial tightly with the instrument cap.



**10.** Press: **READ**.  
The cursor will move to the right, then the results in mg/L monochloramine (as Cl<sub>2</sub>) will be displayed.

### Sampling and Storage

Analyze samples for monochloramine immediately after collection. Rinse the sample container several times with the sample water allowing it to overflow each time. If sampling from a tap, let the water flow for at least 5 minutes. Cap the container so that there is no head space (air) above the sample.

### Accuracy Check

Prepare the following monochloramine standard fresh before use:

1. Using a clean 100-mL Class A volumetric flask, add the contents of one Buffer Powder Pillow, pH 8.3, to approximately 50 mL of organic-free water. Swirl to dissolve the powder.
2. Use a Class A volumetric pipet to transfer 2.00 mL of Nitrogen Ammonia Standard Solution, 100-mg/L as NH<sub>3</sub>-N, into a flask.
3. Dilute to volume with organic-free water. Cap and mix thoroughly. This is the 2.00-mg/L buffered ammonia standard.
4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a magnetic stir bar and place the beaker on a stir plate.
5. Note the free chlorine concentration for the Chlorine Solution Ampules, 50–70 mg/L. Use ampules from a recent lot.

## CHLORAMINE, MONO, High Range, continued

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6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Turn the stir plate on to medium speed.
8. Open an ampule. Use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard while it is mixing.
9. Allow the monochloramine solution to mix for 1 minute after all the Chlorine Solution is added.
10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water. Cap and mix thoroughly. This is a nominal 4.5-mg/L (as Cl<sub>2</sub>) monochloramine standard.

Use this solution within 1 hour of preparation.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 3.5 mg/L monochloramine as chlorine and two representative lots of reagent, a single operator obtained a standard deviation of  $\pm 0.2$  mg/L Cl<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit (EDL) for Method 10172 is 0.2 mg/L Cl<sub>2</sub>. For more information on the EDL, see *Section 1* of the DR/800 Procedure Manual.

### Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 11 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br <sup>-</sup>
Bromine	15 mg/L Br <sub>2</sub>
Calcium	1000 mg/L as CaCO <sub>3</sub>

## CHLORAMINE, MONO, High Range, continued

Table 11 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO <sub>2</sub>
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN <sup>-</sup>
Free Chlorine	10 mg/L Cl <sub>2</sub>
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	1000 mg/L as CaCO <sub>3</sub>
Manganese (VII)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO <sub>4</sub>
Silica	100 mg/L SiO <sub>2</sub>
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO <sub>3</sub> <sup>2-</sup>
Tyrosine	1 mg/L as N
Urea	10 mg/L as N
Zinc	5 mg/L

Table 12 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Ozone	-	Above 1 mg/L	Usually doesn't coexist with monochloramine
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine
Thiocyanate	-	Above 0.5 mg/L	

## CHLORAMINE, MONO, High Range, continued

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### Summary of Method

The sample is first diluted in a Test 'N Tube™. In the presence of a cyanoferrate catalyst, monochloramine (NH<sub>2</sub>Cl) in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate compound couples with excess substituted phenol to form a green indophenol. Color intensity is proportional to the amount of monochloramine present in the sample.

### Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

### Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890 instrument.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Key in "8138", then press **ENTER**.
6. Key the number in the "Enter" column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Table 13

Line number on display	Enter	Line number on display	Enter
1	111	29	108
2	42	30	78
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	58
9	0	37	61
10	0	38	112

## CHLORAMINE, MONO, High Range, continued

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Table 13 (Continued)

11	0	39	62
12	65	40	74
13	116	41	61
14	49	42	112
15	248	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	153
27	50	55	0
28	67	56	255

## CHLORAMINE, MONO, High Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
HR Monochloramine Test 'N Tubes, 50 tests .....		28051-45	
Includes:			
HR Monochloramine Diluent Vials .....	50 .....		*
Funnel, micro .....	1 .....	each .....	25843-35
Monochlor F Reagent Pillows .....	1 .....	50/pkg .....	28022-46

### REQUIRED APPARATUS

COD/TNT Vial Adapter, DR/800 .....	1 .....	each .....	48464-00
Pipet, Mohr, glass, 2.00-mL .....	1 .....	each .....	20936-36
Test Tube Rack .....	1 .....	each .....	18641-00

### OPTIONAL REAGENTS

Organic-free Water .....	500-mL .....		26415-49
Buffer Powder Pillows, pH 8.3 .....	25/pkg .....		898-68
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH <sub>3</sub> -N .....	500-mL .....		24065-49
Chlorine Solution Voluette <sup>®</sup> Ampule, 50-75 mg/L, 10-mL .....	16/pkg .....		14268-10

### OPTIONAL APPARATUS

Beaker, 100-mL .....		each .....	500-42H
Clippers (medium powder pillows) .....		each .....	968-00
Clippers (shears) .....		each .....	23694-00
Flask, Volumetric, Class A, 100-mL .....		each .....	14574-42
Pipet, Mohr, Glass, 10-mL .....		each .....	20934-38
Pipet, Volumetric, Class A, 2.00-mL .....		each .....	14515-36
Pipet, Volumetric, Class A, 50.00-mL .....		each .....	14515-41
Stir Bar, Octagonal .....		each .....	20953-52
Stirrer, Magnetic, 110 V, 4" x 4" .....		each .....	23436-00

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\* Not sold separately.



# CHLORINE DIOXIDE (0 to 5.00 mg/L)

Method 10126

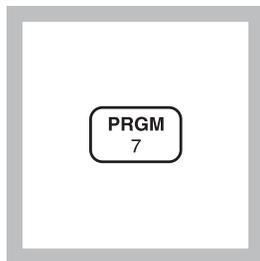
DPD Method\*

For water

USEPA accepted for reporting for drinking water analysis

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

## Using Powder Pillows

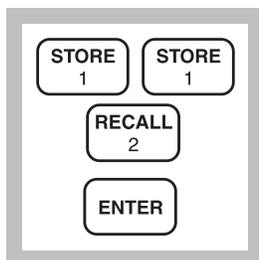


**1.** Enter the stored program number for chlorine dioxide (ClO<sub>2</sub>) powder pillows.

Press: **PRGM**

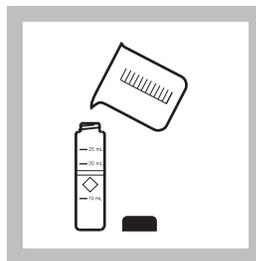
The display will show:

**PRGM ?**



**2.** Press: **112 ENTER**

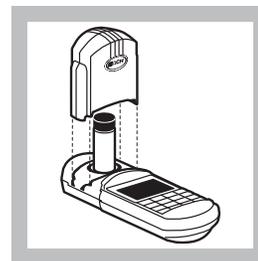
The display will show **mg/L, ClO<sub>2</sub>**, and the **ZERO** icon.



**3.** Fill a sample cell with 10 mL of sample (the blank).

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

*Note: Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*

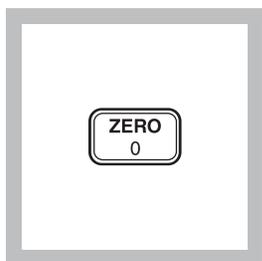


**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See Reagent Blank Correction in Section 1 of the DR/800 Procedure Manual.*

\* Procedure is equivalent to *Standard Method 4500, ClO<sub>2</sub>P*

## CHLORINE DIOXIDE, continued

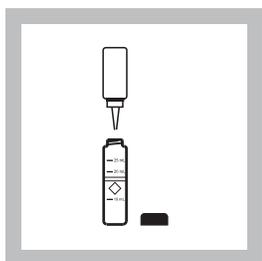


**5. Press: ZERO**

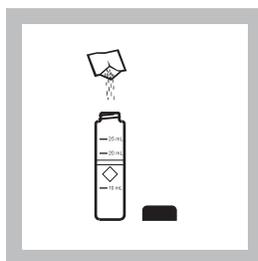
The cursor will move to the right, then the display will show:

**0.00 mg/L ClO<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1 of the DR/800 Procedures Manual.*



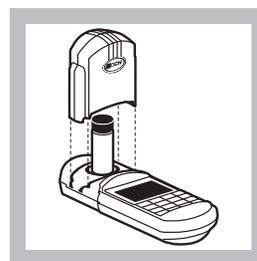
**6. Add four drops of Glycine Reagent to the sample cell. Swirl to mix.**



**7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl to mix.**

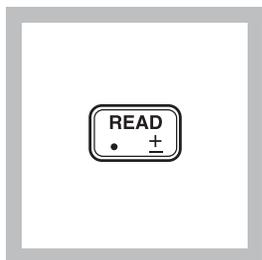
*Note: A pink color will develop if free chlorine dioxide is present.*

*Note: Perform step 9 within one minute of reagent addition.*



**8. Allow 30 seconds for undissolved powder to settle. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**

*Note: Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



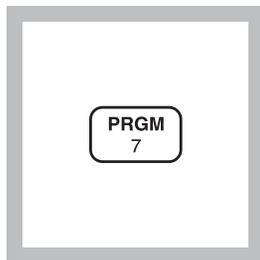
**9. Press: READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

*Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

## CHLORINE DIOXIDE, continued

### Using AccuVac® Ampuls

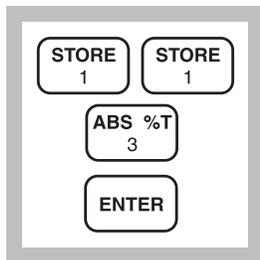


**1.** Enter the stored program number for chlorine dioxide (ClO<sub>2</sub>) AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**



**2.** Press: **113 ENTER**

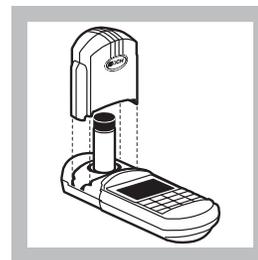
The display will show **mg/L, ClO<sub>2</sub>** and the **ZERO** icon.



**3.** Fill a sample cell with at least 10 mL of sample (the blank). Fill a 50-mL beaker with 40 mL of sample. Using the correct sample volume is important.

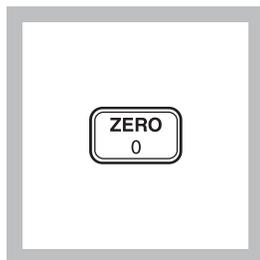
*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

*Note: Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See Reagent Blank Correction in Section 1 of the DR/800 Procedure Manual.*

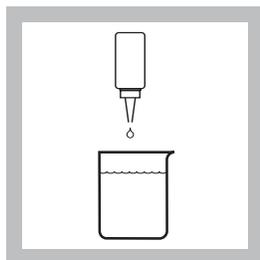


**5.** Press: **ZERO**

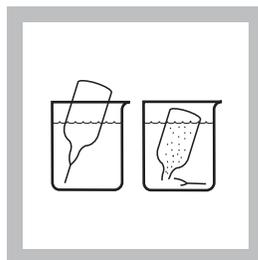
The cursor will move to the right, then the display will show:

**0.00 mg/L ClO<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1 of the DR/800 Procedures Manual.*



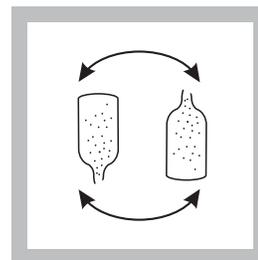
**6.** Add 16 drops of Glycine Reagent to the sample in the beaker. Swirl to mix.



**7.** Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampul fills completely.*

*Note: Perform step 10 within one minute of reagent addition.*

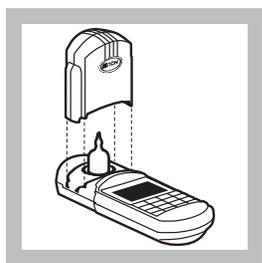


**8.** Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

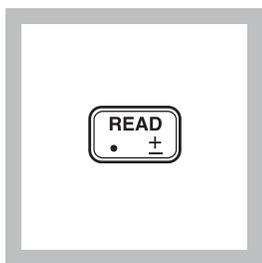
*Note: A pink color will form if chlorine dioxide is present.*

## CHLORINE DIOXIDE, continued

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**9.** Allow 30 seconds for undissolved powder to settle. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

*Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

---

## Sampling and Storage

Analyze samples for chlorine dioxide **immediately** after collection. Chlorine dioxide is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

**Avoid plastic containers** since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

## CHLORINE DIOXIDE, continued

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### Accuracy Check

Because chlorine dioxide is difficult and hazardous to produce, check the DPD and glycine reagents by using chlorine standards. Proceed as follows:

1. Prepare a 1-mg/L free chlorine standard.

#### Method 1

- a. Obtain Free Chlorine Standards, (Cat. No. 14268-10).
- b. Determine the concentration of the standard from the certificate of analysis shipped with the standard (50-75 mg/L). Calculate the volume of standard needed as follows:

$$\text{mL standard needed} = 100 \div \text{standard concentration}$$

- c. Pipet the volume of standard needed into a 100-mL volumetric flask. Dilute to the line with chlorine demand-free deionized water. Invert to mix.

#### Method 2

- a. Dilute 1 drop of commercial 5% chlorine bleach in 1 liter of chlorine demand-free deionized water. Use this as the standard.
2. Verify the standard's concentration using the Hach Free Chlorine Method, #8021.
  3. Perform the chlorine dioxide test on the standard without adding glycine (*step 6*).
  4. The chlorine dioxide reading should be about 2.45 times greater than the chlorine result. If so, this verifies the DPD and the instrument are functioning properly.
  5. Repeat the chlorine dioxide test on the chlorine standard, including the glycine addition (*step 6*). The reading should be less than 0.10 mg/L. This verifies that the glycine is eliminating free chlorine interference.

## CHLORINE DIOXIDE, continued

### Method Performance

#### Precision

<u>Program</u>	<u>Standard</u>	<u>95% Confidence Limits</u>
112	0.24 mg/L	0.22–0.26 mg/L ClO <sub>2</sub>
112	4.79 mg/L	4.67–4.91 mg/L ClO <sub>2</sub>
113	0.26 mg/L	0.21–0.27 mg/L ClO <sub>2</sub>
113	4.83 mg/L	4.71–4.97 mg/L ClO <sub>2</sub>

For more information on determining precision data and method detection limits, see *Section 1* of the *DR/800 Procedures Manual*.

#### Estimated Detection Limit (EDL)

<u>Program</u>	<u>EDL</u>
112	0.04 mg/L ClO <sub>2</sub>
113	0.04 mg/L ClO <sub>2</sub>

For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/800 Procedures Manual*.

#### Interferences

A substance interferes if it changes the final reading by 0.1 mg/L ClO<sub>2</sub> or more.

<b>Interfering Substance</b>	<b>Interference Levels and Treatments</b>
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see <i>Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see <i>Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual</i> ).
Bromine, Br <sub>2</sub>	Interferes at all levels.
Chlorine, Cl <sub>2</sub>	May interfere at levels greater than 6 mg/L. Additional glycine may be able to compensate for this interference.
Chloramines, organic	May interfere.
Flocculating agents	High levels of most flocculating agents can be tolerated. This tolerance is decreased if chlorine is present. See the information about metals in this table. In the presence of 0.6 mg/L Cl <sub>2</sub> , Al(SO <sub>4</sub> ) <sub>3</sub> (< 500 mg/L) and FeCl <sub>2</sub> (<200 mg/L) may be tolerated.
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub> .

## CHLORINE DIOXIDE, continued

Interfering Substance	Interference Levels and Treatments
Iodine, I <sub>2</sub>	Interferes at all levels.
Manganese, oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, oxidized (Cr <sup>6+</sup> )	Oxidized manganese interferes at all levels. Oxidized chromium interferes at levels greater than 2 mg/L. To remove the interferences: <ol style="list-style-type: none"> <li>1. Adjust sample pH to 6–7.</li> <li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>3. Mix and wait one minute.</li> <li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>5. Analyze 10 mL of the treated sample as described in the procedure.</li> <li>6. Subtract the result of this test from the original analysis to obtain the correct chlorine dioxide concentration.</li> </ol>
Metals	Various metals may interfere by combining with the glycine needed to remove the chlorine interference. Metal interference is limited except when chlorine is present. In the presence of 0.6 mg/L Cl <sub>2</sub> , both copper (>10 mg/L) and nickel (>50 mg/L) interfere. Other metals may also interfere, depending on their ability to prevent glycine from reacting with any Cl <sub>2</sub> in the sample. It may be necessary to add more glycine to overcome this interference.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L ClO <sub>2</sub> increase in the reading.
Ozone	Interferes at levels greater than 1.5 mg/L.
Peroxides	May interfere.
Extreme sample pH	Adjust to pH 6–7. See <i>Section 1, pH Interferences, in the DR/800 Procedures Manual.</i>
Highly buffered samples	Adjust to pH 6–7. See <i>Section 1, pH Interferences, in the DR/800 Procedures Manual.</i>

## Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

## Summary of Method

Chlorine dioxide reacts with DPD (N,N-diethyl-p-phenylenediamine) Indicator Reagent (to the extent of one-fifth of its total available chlorine content corresponding to reduction of chlorine dioxide to chlorite) to form a pink color. The color intensity is proportional to the ClO<sub>2</sub> in the sample. Chlorine interference is eliminated by adding glycine, which converts free chlorine to chloroaminoacetic acid, but has no effect on chlorine dioxide at the test pH.

## CHLORINE DIOXIDE, continued

### REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required		Cat. No.
	per test	Unit	
Chlorine Dioxide DPD/Glycine Reagent Set (100 tests).....			27709-00
Includes one of each:			
DPD Free Chlorine Reagent Powder Pillows, 10 mL . 1 pillow ..	100/pkg		21055-69
Glycine Reagent .....	4 drops	29 mL	27621-33

### REQUIRED REAGENTS (Using AccuVac® Ampuls)

Chlorine Dioxide DPD/Glycine AccuVac® Ampul Reagent Set (25 tests).....			27710-00
Includes one of each:			
DPD Free Chlorine Reagent AccuVac® Ampuls .....	1	25/pkg	25020-25
Glycine Reagent .....	16 drops	29 mL	27621-33

### OPTIONAL REAGENTS

Chlorine Standard Solution, Voluette™ ampule, 50-75 mg/L, 10 mL .....	16/pkg		14268-10
DPD Free Chlorine Reagent, SwifTest™ .....	250 tests		28023-00
Potassium Iodide Solution, 30 g/L .....	100 mL*	MDB	343-32
Sodium Arsenite, 5 g/L .....	100 mL*	MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N .....	100 mL*	MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N .....	100 mL*	MDB	1270-32
Water, deionized.....	4L		272-56
Water, sterile, chlorine dioxide-free.....	500 mL		26415-49

### OPTIONAL APPARATUS

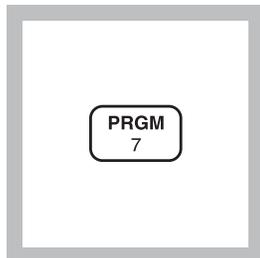
AccuVac® Snapper Kit .....	each		24052-00
Cylinder, graduated, 25 mL .....	each		508-40
pH Meter, <i>sens<sup>ion</sup></i> ™1, portable, with electrode .....	each		51700-10
pH Paper, 1 to 11 pH units.....	5 rolls/pkg		391-33
Pipet, TenSette®, 0.1 to 1.0 mL .....	each		19700-01
Pipet Tips, for 19700-01 TenSette® Pipet .....	50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette® Pipet .....	1000/pkg		21856-28
PourRite™ Ampule Breaker .....	each		24846-00

#### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

\* Marked Dropper Bottle - contact Hach for larger sizes.

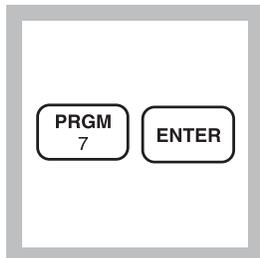
**CHLORINE DIOXIDE, Mid Range (0 to 50.0 mg/L) For water and wastewater****Direct Reading Method**

**1.** Enter the stored program number for mid-range chlorine dioxide ( $\text{ClO}_2$ ).

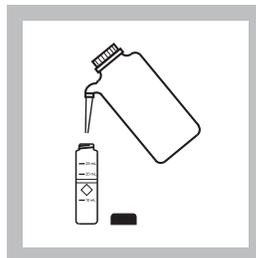
Press: **PRGM**

The display will show:

**PRGM ?**

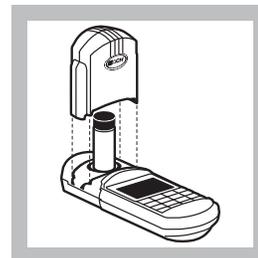


**2.** Press: **7 ENTER**  
The display will show **mg/L,  $\text{ClO}_2$**  and the **ZERO** icon.

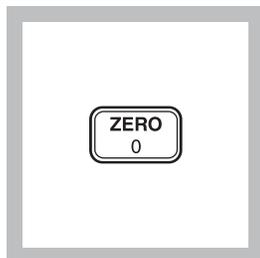


**3.** Fill a sample cell (the blank) with 10 mL of deionized water.

*Note: Analyze samples immediately after collection.*

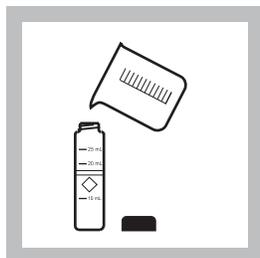


**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

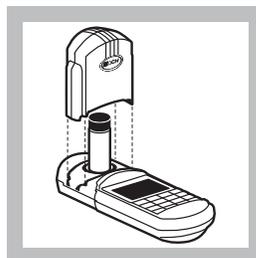


**5.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

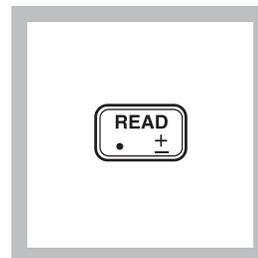
**0.0 mg/L  $\text{ClO}_2$**



**6.** Fill another sample cell with 10 mL of sample (the prepared sample).



**7.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**8.** Press: **READ**  
The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

*Note: If the display flashes "limit" it is due to high  $\text{ClO}_2$  levels. A slight loss of chlorine dioxide may occur during dilution. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*

## CHLORINE DIOXIDE, MR, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Chlorine dioxide is very volatile and unstable; analyze samples immediately upon collection.

### Accuracy Check

#### Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, **these standards are both explosive and volatile!** Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. Hach does not recommend independent standard preparation of chlorine dioxide standards. If independent standard preparation is required, please refer to the instructions in *Standard Methods for the Examination of Water and Wastewater*, 19th ed., under the headings “Stock chlorine dioxide solution” and “Standard chlorine dioxide solution” (pg. 4-54).

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 25.0 mg/L ClO<sub>2</sub>, a single operator obtained a standard deviation of ±0.3 mg/L ClO<sub>2</sub>. For more information on Hach’s precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program 7 is 7.3 mg/L ClO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

Chlorine dioxide, a yellow gas, can be measured directly in a water solution. This method uses a wavelength of 420 nm to increase the range of the test.

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## REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Sample Cell, 10-20-25 mL, w/ cap .....	2.....	6/pkg.....	24019-06	
Water, deionized.....	10 mL.....	4 L.....	272-56	

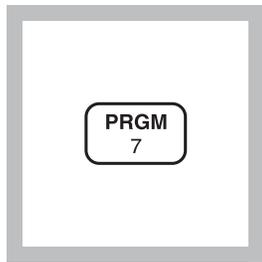
Outside the U.S.A.—Contact the Hach office or distributor serving you.

## CHLORINE, FREE, Ultra-high Range (0.0–10.0 mg/L Cl<sub>2</sub>) Method 10069

DPD Method

USEPA accepted for reporting drinking water analyses\*  
For testing higher levels of free chlorine (hypochlorous acid  
and hypochlorite) in drinking water, cooling water,  
and industrial process waters

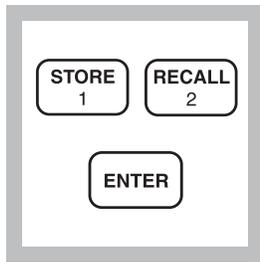
*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



1. Enter the user program number for Chlorine, UHR.

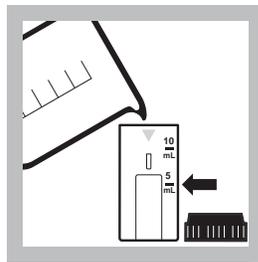
Press: **PRGM**  
The display will show:  
**PRGM?**

*Note: If the chlorine is typically less than 2.0 mg/L, use method 8021, program number 9.*

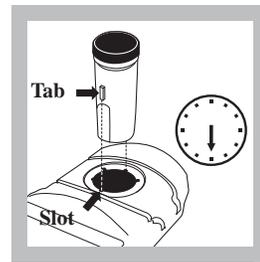


2. Press:  
**12 ENTER**  
The display will show

**mg/L Cl<sub>2</sub>**  
then: **ZERO**



3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.

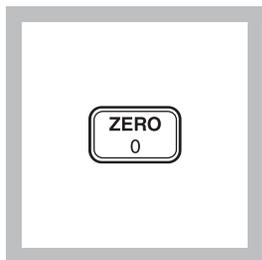


4. Place the cell into the instrument. Cover the sample cell tightly with the instrument cap.

*Note: Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

\* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.

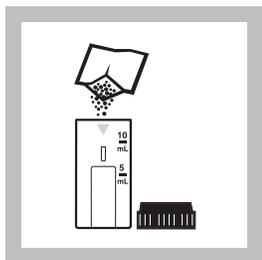
## CHLORINE, FREE, Ultra-high Range, continued



**5. Press: ZERO**

The cursor will move to the right, then the display will show:

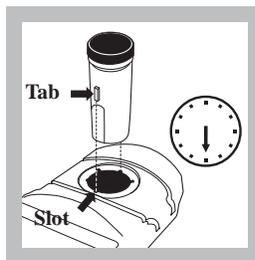
**0.0 mg/L Cl<sub>2</sub>**



**6.** Remove the sample cell from the cell holder and add the contents of one 25-mL DPD Free Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

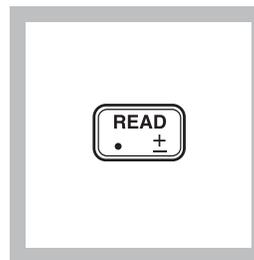
Proceed **immediately** to step 7.

*Note: A pink color will develop if chlorine is present.*



**7.** Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

*Note: Place the sample cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



**8.** Within one minute after reagent addition, press: **READ**.

The cursor will move to the right. The result in mg/L chlorine (as Cl<sub>2</sub>) will be displayed.

*Note: See "Interferences" on page 120 for samples with high monochloramine concentrations.*

### Sampling and Storage

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and reacts rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of free chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.

## CHLORINE, FREE, Ultra-high Range, continued

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- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

### Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.
2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl<sub>2</sub>. Using the TenSette<sup>®</sup> Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.
3. Analyze each standard addition sample as described in the procedure. Record each result.
4. Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of Cl}_2 \text{ standard ampule}}{\text{sample volume} + \text{volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl<sub>2</sub> added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

## CHLORINE, FREE, Ultra-high Range, continued

### Method Performance

#### Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl<sub>2</sub> and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit for Method 10069 is 0.1 mg/L Cl<sub>2</sub>. For more information on the estimated detection limit, see Section 1 of the DR/800 Procedure Manual.

### Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. <ol style="list-style-type: none"> <li>1. Neutralize to pH 6–7 with 1 N Sodium Hydroxide.</li> <li>2. Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.</li> <li>3. Correct for volume addition.</li> </ol>
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. <ol style="list-style-type: none"> <li>1. Neutralize to pH 6–7 with 1 N Sulfuric Acid.</li> <li>2. Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.</li> <li>3. Correct for volume addition.</li> </ol>
Bromine, Br <sub>2</sub>	Interferes at all levels
Chlorine Dioxide, ClO <sub>2</sub>	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I <sub>2</sub>	Interferes at all levels
Manganese, oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6–7.</li> <li>2. Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample.</li> <li>3. Mix and wait 1 minute.</li> <li>4. Add 2 drops of Sodium Arsenite (5 g/L) and mix.</li> <li>5. Analyze the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>

## CHLORINE, FREE, Ultra-high Range, continued

Interfering Substance	Interference Levels and Treatments																								
Monochloramine	<p>For conventional free chlorine disinfection (beyond the breakpoint), monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test varies with the sample temperature, the relative amount of monochloramine to free chlorine, and the time required to do the analysis. Approximate interference levels of monochloramine in the free chlorine test are listed below (as mg/L Cl<sub>2</sub>).</p> <table border="1"> <thead> <tr> <th rowspan="2">NH<sub>2</sub>Cl (as Cl<sub>2</sub>)</th> <th colspan="4">Sample Temperature °C (°F)</th> </tr> <tr> <th>5 (40)</th> <th>10 (50)</th> <th>20 (68)</th> <th>30(83)</th> </tr> </thead> <tbody> <tr> <td>1.2</td> <td>0.2</td> <td>0.2</td> <td>0.3</td> <td>0.3</td> </tr> <tr> <td>2.5</td> <td>0.4</td> <td>0.5</td> <td>0.6</td> <td>0.6</td> </tr> <tr> <td>3.5</td> <td>0.5</td> <td>0.6</td> <td>0.7</td> <td>0.8</td> </tr> </tbody> </table>	NH <sub>2</sub> Cl (as Cl <sub>2</sub> )	Sample Temperature °C (°F)				5 (40)	10 (50)	20 (68)	30(83)	1.2	0.2	0.2	0.3	0.3	2.5	0.4	0.5	0.6	0.6	3.5	0.5	0.6	0.7	0.8
NH <sub>2</sub> Cl (as Cl <sub>2</sub> )	Sample Temperature °C (°F)																								
	5 (40)	10 (50)	20 (68)	30(83)																					
1.2	0.2	0.2	0.3	0.3																					
2.5	0.4	0.5	0.6	0.6																					
3.5	0.5	0.6	0.7	0.8																					
Ozone	Interferes at all levels																								
Peroxides	May interfere																								
Extreme sample pH or highly buffered samples	Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide																								

### Summary of Method

The range of analysis using the DPD method for free chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Free Chlorine Reagent is added to a 5-mL sample portion.

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) reacts immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional in intensity to the chlorine concentration.

### Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.

## CHLORINE, FREE, Ultra-high Range, continued

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6. Key the number in the “Enter” column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

## CHLORINE, FREE, Ultra-high Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows, 25-mL.....	1.....	100/pkg.....	14070-99

### REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm.....	1.....	2/pkg.....	48643-02
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### OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L.....	20/pkg.....		14268-20
Potassium Iodide Solution, 30-g/L.....	100 mL	MDB.....	343-32
Sodium Arsenite Solution, 5-g/L.....	100 mL	MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1.00 N.....	100 mL	MDB.....	1045-32
Sulfuric Acid Standard Solution, 1.000 N.....	100 mL	MDB.....	1270-32
Water, deionized.....	4 L.....		272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....		24846-00
Cylinder, graduated, 10-mL, mixing.....	each.....		20886-38
pH Meter, sens <i>ion</i> ™1, portable, with electrode.....	each.....		51700-10
Pipet, TenSette®, 0.1 to 1.0 mL.....	each.....		19700-01
Pipet Tips, for 19700-01 TenSette Pipet.....	50/pkg.....		21856-96
Pipet Tips, for 19700-01 TenSette Pipet.....	1000/pkg.....		21856-28



## CHLORINE, TOTAL, Ultra-High Range (0.0–10.0 mg/L Cl<sub>2</sub>) Method 10070

DPD Method

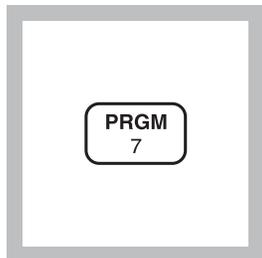
USEPA accepted for reporting water and wastewater analyses\*

For testing higher levels of total chlorine (free and combined)

in drinking water, cooling water,

industrial process waters, or treated wastewater

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



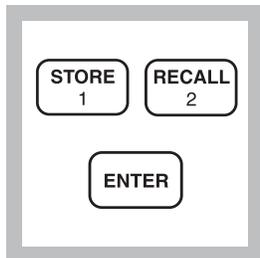
1. Enter the user program number for Chlorine, UHR.

Press: **PRGM**

The display will show:

**PRGM?**

*Note: If the chlorine is typically less than 2.0 mg/L, use method 8167, program number 9.*



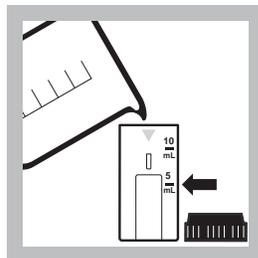
2. Press:

**12 ENTER**

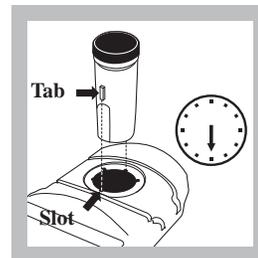
The display will show

**mg/L Cl<sub>2</sub>**

then: **ZERO**



3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.

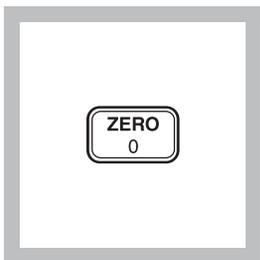


4. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

*Note: Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

\* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.

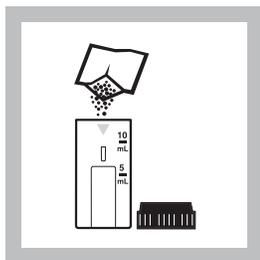
## CHLORINE, TOTAL, Ultra-High Range, continued



**5. Press: ZERO**

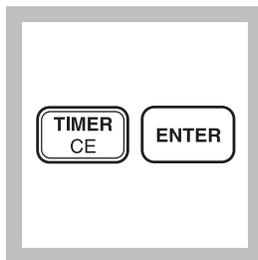
The cursor will move to the right, then the display will show:

**0.0 mg/L Cl<sub>2</sub>**



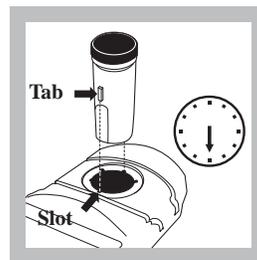
**6.** Remove the sample cell from the cell holder and add the contents of one 25- mL DPD Total Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

*Note: A pink color will develop if chlorine is present.*



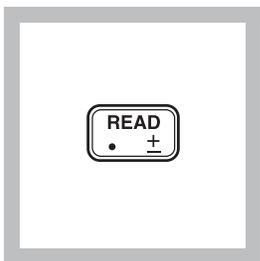
**7. Press: TIMER ENTER**

A 3-minute reaction period will begin.



**8.** Within 3 minutes after the timer beeps, place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

*Note: Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



**9. Press: READ**

The cursor will move to the right. The result in mg/L chlorine (as Cl<sub>2</sub>) will be displayed.

## CHLORINE, TOTAL, Ultra-High Range, continued

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### Sampling and Storage

Analyze samples for chlorine immediately after collection. Free and combined chlorine are strong oxidizing agents and react rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.
- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

### Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.
2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L  $\text{Cl}_2$ . Using the TenSette<sup>®</sup> Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.
3. Analyze each standard addition sample as described in the procedure. Record each result.

## CHLORINE, TOTAL, Ultra-High Range, continued

- Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of Cl}_2 \text{ standard ampule}}{\text{sample volume} + \text{volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl<sub>2</sub> added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

### Method Performance

#### Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl<sub>2</sub> and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit for Method 10070 is 0.05 mg/L Cl<sub>2</sub>. For more information on the estimated detection limit, see Section 1 of a DR/800 Procedure Manual.

### Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. <ol style="list-style-type: none"> <li>Neutralize to pH 6–7 with 1 N Sodium Hydroxide.</li> <li>Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.</li> <li>Correct for volume addition.</li> </ol>
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. <ol style="list-style-type: none"> <li>Neutralize to pH 6–7 with 1 N Sulfuric Acid.</li> <li>Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.</li> <li>Correct for volume addition.</li> </ol>
Bromine, Br <sub>2</sub>	Interferes at all levels
Chlorine Dioxide, ClO <sub>2</sub>	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I <sub>2</sub>	Interferes at all levels

## CHLORINE, TOTAL, Ultra-High Range, continued

Interfering Substance	Interference Levels and Treatments
Manganese, oxidized ( $Mn^{4+}$ , $Mn^{7+}$ ) or Chromium, oxidized ( $Cr^{6+}$ )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6–7.</li> <li>2. Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample.</li> <li>3. Mix and wait 1 minute.</li> <li>4. Add 2 drops of Sodium Arsenite (5 g/L) and mix.</li> <li>5. Analyze the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide

### Summary of Method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N,N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a pink color which is proportional in intensity to the total chlorine concentration.

### Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.
6. Key the number in the “Enter” column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

## CHLORINE, TOTAL, Ultra-High Range, continued

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Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

## CHLORINE, TOTAL, Ultra-High Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows, 25-mL .....	1.....	100/pkg.....	14064-99

### REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm.....	1.....	2/pkg.....	48643-02
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### OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L.....	20/pkg.....		14268-20
Potassium Iodide Solution, 30-g/L.....	100 mL	MDB.....	343-32
Sodium Arsenite Solution, 5-g/L .....	100 mL	MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1.00 N.....	100 mL	MDB.....	1045-32
Sulfuric Acid Standard Solution, 1.000 N.....	100 mL	MDB.....	1270-32
Water, deionized.....	4 L.....		272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit .....	each.....		24846-00
Cylinder, graduated, 10-mL, mixing .....	each.....		20886-38
pH Meter, sens <i>ion</i> ™1, portable, with electrode .....	each.....		51700-10
Pipet, TenSette®, 0.1 to 1.0 mL.....	each.....		19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....		21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....		21856-28

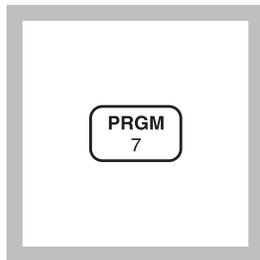


**CHLORINE, FREE (0 to 2.00 mg/L)**

For water, wastewater, and seawater

**DPD Method (Powder Pillows or AccuVac Ampuls) USEPA accepted for reporting wastewater and drinking water analyses\***

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

**Using Powder Pillows**

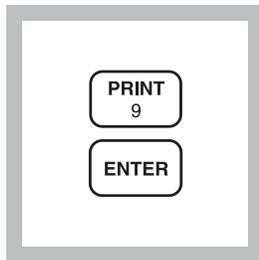
**1.** Enter the stored program number for free and total chlorine (Cl<sub>2</sub>) powder pillows.

Press: **PRGM**

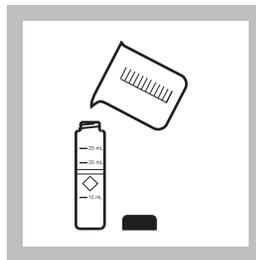
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



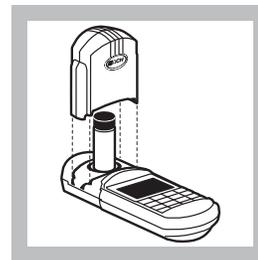
**2.** Press: **9 ENTER**  
The display will show **mg/L, Cl<sub>2</sub>** and the **ZERO** icon.



**3.** Fill a sample cell with 10 mL of sample (the blank).

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

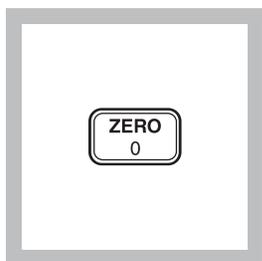
*Note: The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillows in step 7.*



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

\* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

## CHLORINE, FREE, continued



**5. Press: ZERO**

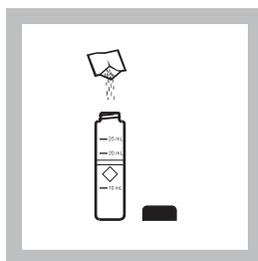
The cursor will move to the right, then the display will show:

**0.00 mg/L Cl<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

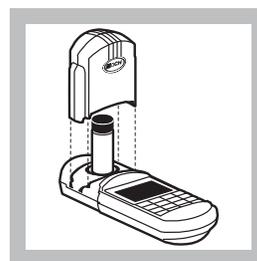


**6. Fill another cell with 10 mL of sample.**



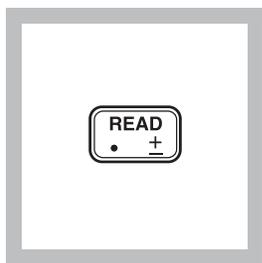
**7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.**

*Note: A pink color will develop if free chlorine is present.*



**8. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**

*Note: Perform Step 9 within one minute of reagent addition.*



**9. Press: READ**

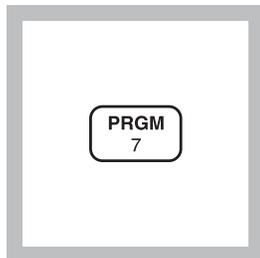
The cursor will move to the right, then the result in mg/L chlorine will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

*Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or, use the High Range Free Chlorine test, program #8.*

## CHLORINE, FREE continued

### Using AccuVac Ampuls



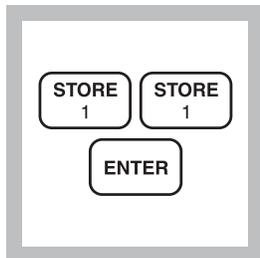
**1.** Enter the stored program number for free and total chlorine (Cl<sub>2</sub>)-AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

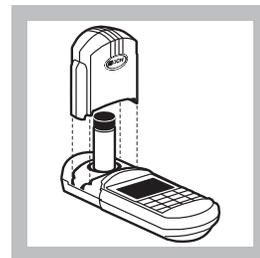


**2.** Press: **11 ENTER**  
The display will show **mg/L, Cl<sub>2</sub>** and the **ZERO** icon.

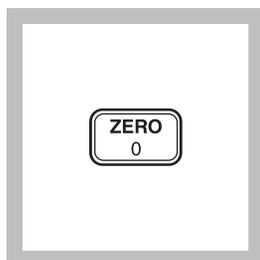


**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



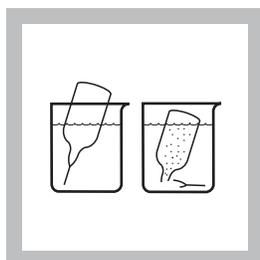
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**5.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

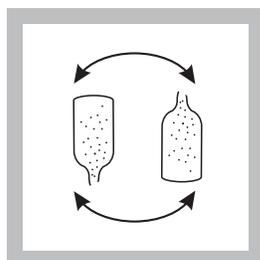
**0.00 mg/L Cl<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



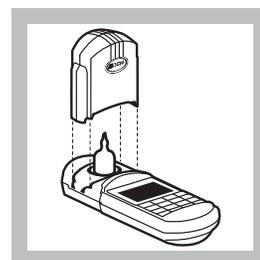
**6.** Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampule fills completely.*



**7.** Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

*Note: A pink color will form if chlorine is present.*

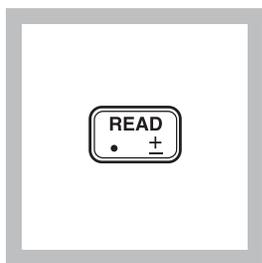


**8.** Immediately place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.

*Note: Perform step 9 within one minute of reagent addition.*

## CHLORINE, FREE continued

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### 9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

*Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

---

### Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

**Avoid plastic containers** since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

## CHLORINE, FREE continued

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A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

### Accuracy Check

#### Standard Additions Method (using powder pillows)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Re-zero the instrument using the original sample (the blank).
- d) Place the spiked sample in the cell holder and press **READ**. Record the results.
- e) Calculate the concentration of mg/L chlorine added to the sample:
$$\text{mg/L Chlorine added} = \frac{0.1 (\text{vol. standard added}) \times \text{Label value (mg/L Cl}_2\text{)}}{10.1 (\text{sample} + \text{standard volume})}$$
- f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step e).
- g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Additions Method (using AccuVac Ampuls)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.
- c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.
- d) Fill a DPD Free Chlorine AccuVac completely from each

## CHLORINE, FREE continued

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beaker.

- e) Analyze the spiked and unspiked sample as described in the procedure.
- f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L Chlorine added} = \frac{0.2(\text{vol. standard added}) \times \text{Label value (mg/L Cl}_2\text{)}}{25.2(\text{sample} + \text{standard volume})}$$

- g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step f).
- h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

#### Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl<sub>2</sub>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

#### Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

## CHLORINE, FREE continued

### Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, Oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, Oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6-7.</li> <li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>3. Mix and wait one minute.</li> <li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>5. Analyze 10 mL of the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences, Section 1</i> .

### Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

## CHLORINE, FREE continued

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
DPD Free Chlorine Powder Pillows, 10 mL.....	1	pillow	..	100/pkg21055-69
Sample Cell, 10, 20, 25 mL, w/ cap.....	2		.....	6/pkg24019-06

### REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

DPD Free Chlorine Reagent AccuVac Ampuls .....	1	ampul	.....	25/pkg25020-25
Beaker, 50 mL .....	1		.....	each 500-41H

### OPTIONAL REAGENTS

Description		Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL .....	20	/pkg.....	26300-20
DPD Free Chlorine Reagent, SwifTest .....	250	tests.....	28023-00
Potassium Iodide Solution, 30 g/L .....	100	mL* MDB.....	343-32
Sodium Arsenite, 5 g/L .....	100	mL* MDB .....	1047-32
Sodium Hydroxide Standard Solution, 1.000 N .....	100	mL* MDB.....	1045-32
Sulfuric Acid Standard Solution, 1.000 N .....	100	mL* MDB.....	1270-32
Water, deionized.....	4	L .....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each.....	24052-00
Cylinder, graduated, 25 mL .....	each.....	508-40
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode .....	each.....	51700-10
pH Paper, 1 to 11 pH units.....	5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg..	21856-96Pipet
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
PourRite Ampule Breaker.....	each.....	24846-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

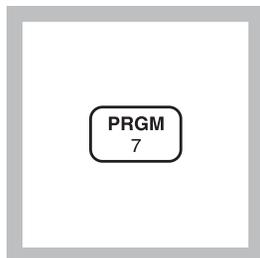
\* Marked Dropper Bottle - contact Hach for larger sizes.

**CHLORINE, TOTAL (0 to 2.00 mg/L)**

For water, wastewater and seawater

**DPD Method (Powder Pillows or AccuVac Ampuls)****USEPA accepted for reporting water and wastewater analyses\***

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

**Using Powder Pillows**

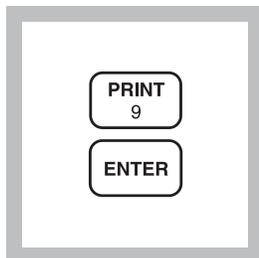
**1.** Enter the stored program number for total chlorine (Cl<sub>2</sub>) powder pillows.

Press: **PRGM**

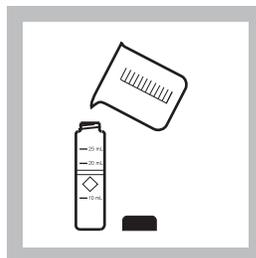
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

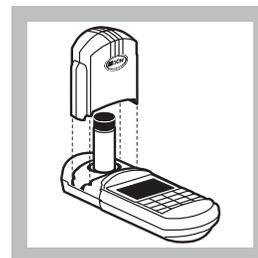


**2.** Press: **9 ENTER**  
The display will show **mg/L, Cl<sub>2</sub>** and the **ZERO** icon.



**3.** Fill a sample cell with 10 mL of sample (the blank).

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

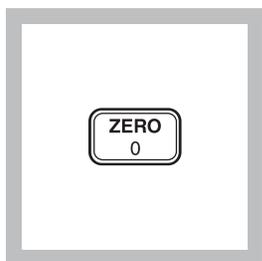


**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

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\* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

## CHLORINE, TOTAL, continued

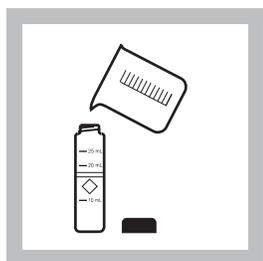


**5. Press: ZERO**

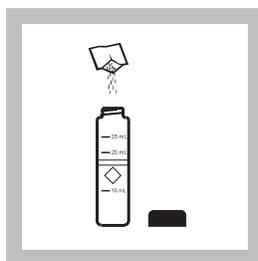
The cursor will move to the right, then the display will show:

**0.00 mg/L Cl<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

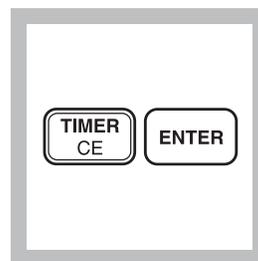


**6. Fill a second cell to the 10-mL mark with sample.**



**7. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap and swirl the sample cell vigorously to dissolve the powder.**

*Note: It is not necessary that all the powder dissolves.*

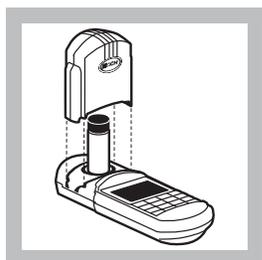


**8. Press:**

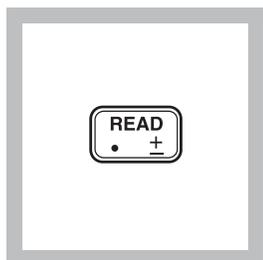
**TIMER ENTER**

A three-minute reaction period will begin. A pink color will develop if chlorine is present.

*Note: The SwifTest Dispenser for Total Chlorine can be used in place of the powder pillows in step 7.*



**9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**



**10. Press: READ**

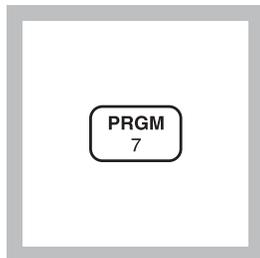
The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

*Note: If the sample temporarily turns yellow after sample addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or use the High Range Total Chlorine test, program #8.*

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

## CHLORINE, TOTAL, continued

### Using AccuVac Ampuls



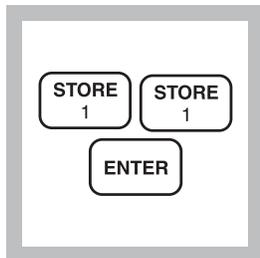
**1.** Enter the stored program number for total chlorine ( $\text{Cl}_2$ ) AccuVac Ampuls.

Press: **PRGM**

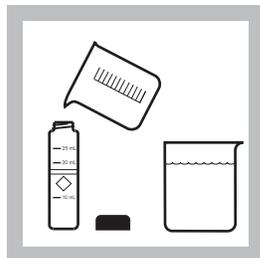
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

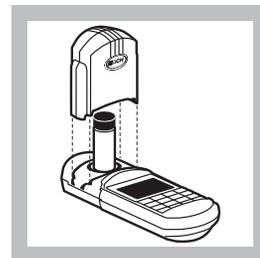


**2.** Press: **11 ENTER**  
The display will show **mg/L, Cl2** and the **ZERO** icon.

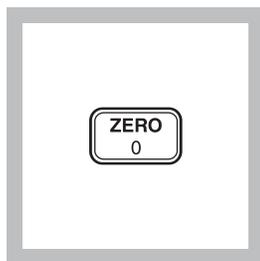


**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



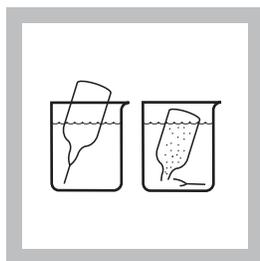
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**5.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

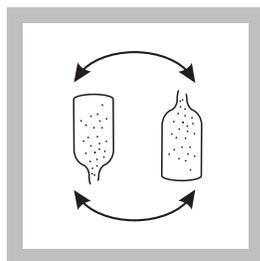
**0.00 mg/L Cl2**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



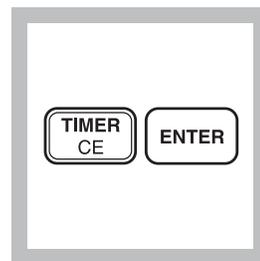
**6.** Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampule fills completely.*



**7.** Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

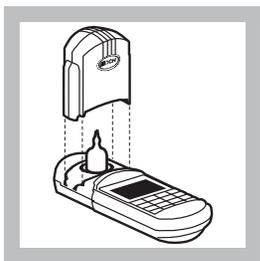
*Note: A pink color will form if chlorine is present.*



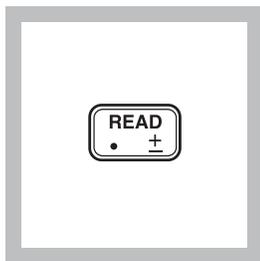
**8.** Press: **TIMER ENTER**  
A three-minute reaction period will begin.

## CHLORINE, TOTAL, continued

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**9.** When the timer beeps, place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

*Note: If the sample temporarily turns yellow after sample addition, or the display shows "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the appropriate dilution factor; see Section 1.*

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

**Avoid plastic containers** since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

## CHLORINE, TOTAL, continued

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Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine analysis immediately.

### Accuracy Check

#### Standard Additions Method (using powder pillows)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Re-zero the instrument using the original sample (the blank).
- d) Place the spiked sample into the cell holder and press **READ**. Record the results.
- e) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1 (\text{vol. standard added}) \times \text{Label value (mg/L Cl}_2\text{)}}{10.1 (\text{sample} + \text{standard volume})}$$

- f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step e).
- g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Additions Method (using AccuVac Ampuls)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- b) Use a graduated cylinder to measure 25 mL of sample into

## CHLORINE, TOTAL, continued

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each of two beakers.

- c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.
- d) Fill a DPD Total Chlorine AccuVac completely from each beaker.
- e) Analyze the spiked and unspiked sample as described in the procedure.
- f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.2 (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)}}{25.2 (\text{sample} + \text{standard volume})}$$

- g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step f).
- h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two lots of reagents with the instrument, a single operator obtained standard deviations of ±0.01 mg/L chlorine.

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

#### Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl<sub>2</sub>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## CHLORINE, TOTAL, continued

### Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> ).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, Oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, Oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6-7.</li> <li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>3. Mix and wait one minute.</li> <li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>5. Analyze 10 mL of the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences, Section 1</i> .

### Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine)

## CHLORINE, TOTAL, continued

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along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

### Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

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### REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Qty/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows.....	1 pillow .....	100/pkg.....	21056-69
Sample Cell, 10-20-25 mL, w/caps .....	2.....	6/pkg.....	24019-06

### REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls .....	1 ampul .....	25/pkg.....	25030-25
Beaker, 50 mL .....	1 .....	each.....	500-41H

### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L Cl <sub>2</sub> .....	20/pkg.....	26300-20
DPD Total Chlorine Reagent, SwifTest .....	250 tests.....	28024-00
Potassium Iodide Solution, 30 g/L.....	100 mL* MDB.....	343-32
Sodium Arsenite, 5 g/L.....	100 mL* MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1 N .....	100 mL* MDB.....	1045-32
Sulfuric Acid Standard Solution, 1 N .....	100 mL* MDB.....	1270-32
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each.....	24052-00
PourRite Ampule Breaker.....	each.....	24846-00
Cylinder, graduated, 25 mL .....	each.....	508-40
pH Indicator Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable .....	each.....	51700-00
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28

### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

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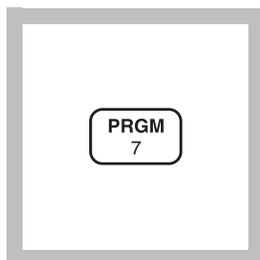
\* Marked Dropper Bottle - contact Hach for larger sizes.

**CHLORINE, FREE (0 to 5.00 mg/L)**

For water, wastewater, and seawater

**DPD Test 'N Tube™ Method\***

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



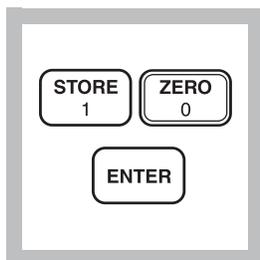
**1.** Enter the stored program number for Test 'N Tube free chlorine (Cl<sub>2</sub>).

Press: **PRGM**

The display will show:

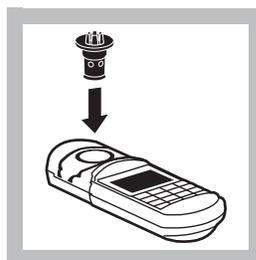
**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



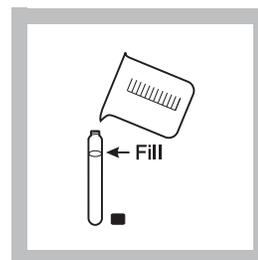
**2.** Press: **10 ENTER**

The display will show **mg/L, Cl<sub>2</sub>** and the **ZERO** icon.



**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down fully to insert it.

*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**4.** Fill an empty Test 'N Tube vial with sample (the blank).

*Note: Fill to the top of the Hach logo "oval" mark.*

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

## CHLORINE, FREE, continued



**5.** Wipe the outside of the blank vial with a towel.

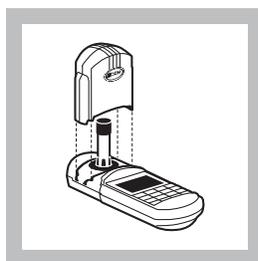
*Note: Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



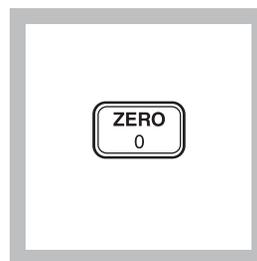
**6.** Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**7.** Cover the vial tightly with the instrument cap.

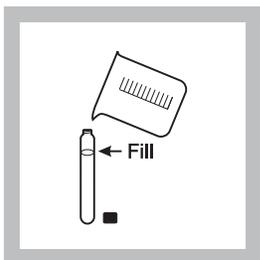


**8.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L Cl<sub>2</sub>**

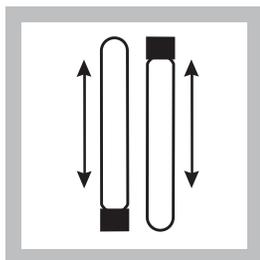
*Note: If Reagent Blank Correction is on, the display may show "limit". See Section 1.*



**9.** Remove the cap from a Free Chlorine DPD-TNT tube. Add 10 mL of sample.

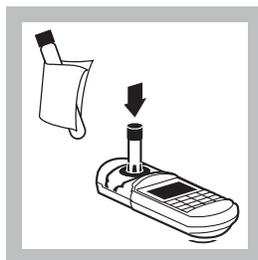
*Note: Fill to the top of the Hach logo "oval" mark.*

*Note: A pink color will develop if chlorine is present.*



**10.** Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

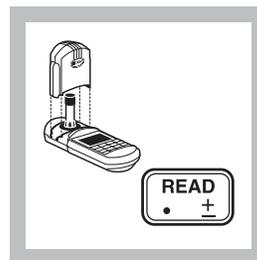
*Note: Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*



**11.** Within 30 seconds after mixing, wipe the prepared sample vial with a towel, then place it in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**12.** Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L free chlorine will be displayed.

## CHLORINE, FREE continued

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### Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

**Avoid plastic containers** since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

### Accuracy Check

#### Standard Additions Method

- a) Snap the top off a HR Chlorine PourRite™ Ampule Standard Solution.
- b) Use a TenSette® Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Analyze the spiked sample, beginning at Step 8 of the procedure.
- d) Calculate the concentration of mg/L chlorine added to the sample:  
$$\text{mg/L chlorine added} = \frac{0.1(\text{vol. standard added}) \times \text{Label value}(\text{mg/L Cl}_2)}{10.1(\text{sample} + \text{standard volume})}$$
- e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step d).
- f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

## CHLORINE, FREE continued

### Method Performance

#### Precision

In a single laboratory using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.14$  mg/L chlorine.

#### Estimated Detection Limit (EDL)

The estimated detection limit for program 10 is 0.03 mg/L Cl<sub>2</sub>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1, Correcting for Volume Additions</i> in the <i>DR/800 Series Procedures Manual</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Section 1 Correcting for Volume Additions</i> ).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"><li>1. Adjust sample pH to 6-7.</li><li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li><li>3. Mix and wait one minute.</li><li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li><li>5. Analyze 10 mL of the treated sample as described in the procedure.</li><li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li></ol>

## CHLORINE, FREE continued

Interfering Substance	Interference Level and Treatment																								
Monochloramine	<p>For conventional free chlorine disinfection (beyond the breakpoint), typical monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test depends on the sample temperature, relative amount of monochloramine to free chlorine, and the time required to do the analysis. Typical interference level of monochloramine in the free chlorine test are listed below (as mg/L Cl<sub>2</sub>).</p> <table border="1"> <thead> <tr> <th rowspan="2">NH<sub>2</sub>Cl as Cl<sub>2</sub></th> <th colspan="4">Sample Temp. °C (°F)</th> </tr> <tr> <th>5 (40)</th> <th>10 (50)</th> <th>20 (68)</th> <th>30 (83)</th> </tr> </thead> <tbody> <tr> <td>1.2 mg/L</td> <td>+0.15</td> <td>+0.19</td> <td>+0.30</td> <td>+0.29</td> </tr> <tr> <td>2.5 mg/L</td> <td>0.35</td> <td>0.38</td> <td>0.55</td> <td>0.61</td> </tr> <tr> <td>3.5 mg/L</td> <td>0.38</td> <td>0.56</td> <td>0.69</td> <td>0.73</td> </tr> </tbody> </table>	NH <sub>2</sub> Cl as Cl <sub>2</sub>	Sample Temp. °C (°F)				5 (40)	10 (50)	20 (68)	30 (83)	1.2 mg/L	+0.15	+0.19	+0.30	+0.29	2.5 mg/L	0.35	0.38	0.55	0.61	3.5 mg/L	0.38	0.56	0.69	0.73
NH <sub>2</sub> Cl as Cl <sub>2</sub>	Sample Temp. °C (°F)																								
	5 (40)	10 (50)	20 (68)	30 (83)																					
1.2 mg/L	+0.15	+0.19	+0.30	+0.29																					
2.5 mg/L	0.35	0.38	0.55	0.61																					
3.5 mg/L	0.38	0.56	0.69	0.73																					
Ozone	Interferes at all levels																								
Peroxides	May interfere																								
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences, Section 1</i> .																								

### Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

### Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

## CHLORINE, FREE continued

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### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Test 'N Tube DPD Free Chlorine Reagent .....	1 vial .....	50/pkg.....	21055-45	
Test 'N Tube Vials .....	1 vial .....	6/pkg.....	22758-06	

### REQUIRED APPARATUS

Caps, white.....	1 cap.....	6/pkg.....	22411-06
COD/TNT Adapter .....	1 .....	each.....	48464-00

### OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 50-75 mg/L, 2 mL .....	20/pkg.....	14268-20
Potassium Iodide Solution, 30 g/L .....	100 mL* MDB.....	343-32
Sodium Arsenite, 5 g/L .....	100 mL* MDB .....	1047-32
Sodium Hydroxide Standard Solution, 1.000 N .....	100 mL* MDB.....	1045-32
Sulfuric Acid Standard Solution, 1.000 N .....	100 mL* MDB.....	1270-32

### OPTIONAL APPARATUS

Beaker, 50 mL.....	each.....	500-41H
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode .....	each.....	51700-10
pH Paper, pH 1 to 11 pH.....	5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
PourRite Ampule Breaker.....	each.....	24846-00
Test Tube Rack .....	each.....	18641-00

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\* Marked Dropper Bottle - contact Hach for larger sizes.



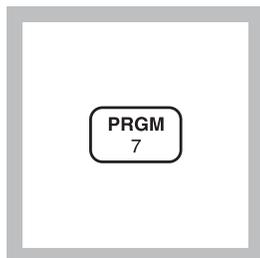


**CHLORINE, TOTAL (0 to 5.00 mg/L)**

For water, wastewater and seawater

**DPD Test 'N Tube™ Method\***

*Note: This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



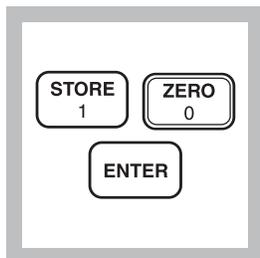
**1.** Enter the stored program number for Test 'N Tube total chlorine (Cl<sub>2</sub>).

Press: **PRGM**

The display will show:

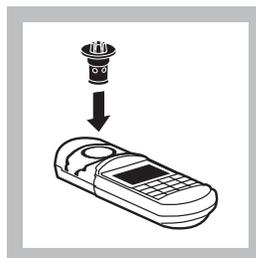
**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



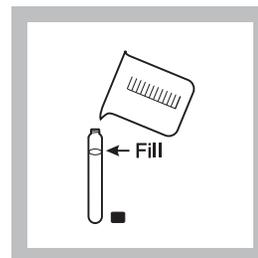
**2.** Press: **10 ENTER**

The display will show **mg/L, Cl<sub>2</sub>** and the **ZERO** icon.



**3.** Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**4.** Fill an empty Test 'N Tube vial with sample (the blank).

*Note: Fill to the top of the Hach logo "oval" mark.*

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*

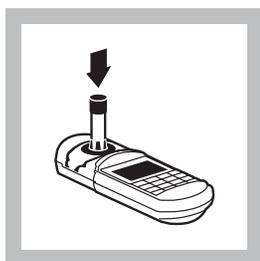
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## CHLORINE, TOTAL, continued



**5.** Wipe the outside of the blank vial with a towel.

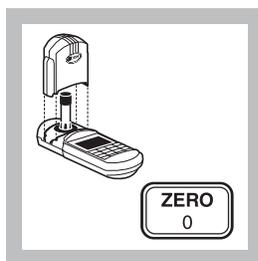
*Note: Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



**6.** Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



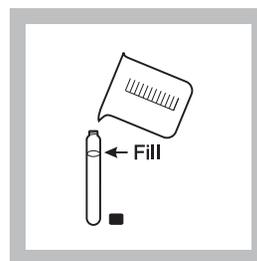
**7.** Cover the vial tightly with the instrument cap.

Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L Cl<sub>2</sub>**

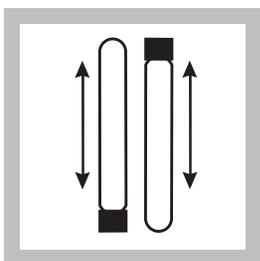
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**8.** Remove the cap from a Total Chlorine DPD-TNT tube. Add 10 mL of sample.

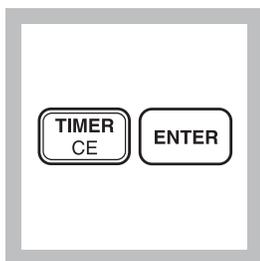
*Note: Fill to the top of the Hach logo "oval" mark.*

*Note: A pink color will develop if chlorine is present.*



**9.** Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

*Note: Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*

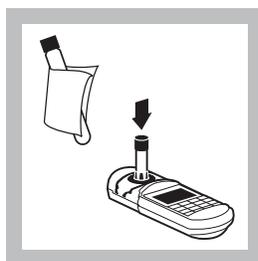


**10.** Press:

**TIMER ENTER**

A three-minute reaction period will begin.

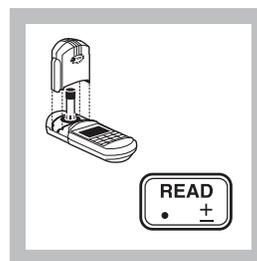
*Note: A pink color will develop if chlorine is present.*



**11.** When the timer beeps, wipe the prepared sample vial with a towel, then place it in the vial adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**12.** Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

## CHLORINE, TOTAL, continued

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### Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free and combined chlorine are strong oxidizing agents and are unstable in natural waters. They react rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

**Avoid plastic containers** since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

### Accuracy Check

#### Standard Additions Method

- a) Snap the top off a High Range Chlorine PourRite™ Ampule Standard Solution.
- b) Use a TenSette® Pipet to add 0.1 mL of the standard to 10 mL of sample (this is the spiked sample). Swirl to mix.
- c) Analyze the spiked sample, beginning at Step 8 of the procedure.
- d) Calculate the concentration of mg/L chlorine added to the sample:  
$$\text{mg/L chlorine added} = \frac{0.1 (\text{vol. standard added}) \times \text{Label value (mg/L Cl}_2\text{)}}{10.1 (\text{sample} + \text{standard volume})}$$
- e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl<sub>2</sub> added (step d).
- f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

## CHLORINE, TOTAL, continued

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained standard deviations of  $\pm 0.14$  mg/L chlorine.

#### Estimated Detection Limit (EDL)

The estimated detection limit for programs 10 is 0.03 mg/L Cl<sub>2</sub>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Correcting for Volume Additions</i> in <i>Section 1</i> ).
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See <i>Correcting for Volume Additions</i> in <i>Section 1</i> ).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, oxidized (Cr <sup>6+</sup> )	<ol style="list-style-type: none"> <li>1. Adjust sample pH to 6-7.</li> <li>2. Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>3. Mix and wait one minute.</li> <li>4. Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>5. Analyze 10 mL of the treated sample as described in the procedure.</li> <li>6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See <i>Interferences</i> in <i>Section 1</i> .

## **CHLORINE, TOTAL, continued**

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### **Summary of Method**

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

### **Pollution Prevention and Waste Management**

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

## CHLORINE, TOTAL, continued

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### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Test 'N Tube DPD Total Chlorine Reagent .....	1 vial.....	25/pkg.....		21056-25
Test 'N Tube Vials .....	1 vial .....	6/pkg.....		22758-06

### REQUIRED APPARATUS

COD/TNT Adapter, DR/800.....	1 .....	each.....		48464-00
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### OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL PourRite ampule, 50-75 mg/L.....	20/pkg.....			14268-20
Potassium Iodide Solution, 30 g/L.....	100 mL* MDB.....			343-32
Sodium Arsenite Solution, 5 g/L .....	100 mL* MDB.....			1047-32
Sodium Hydroxide Standard Solution, 1.00 N .....	100 mL* MDB.....			1045-32
Sulfuric Acid Standard Solution, 1.000 N .....	100 mL* MDB.....			1270-32

### OPTIONAL APPARATUS

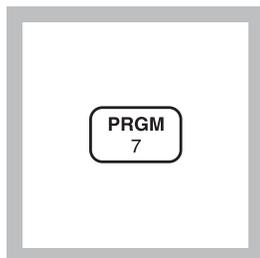
Beaker, 50 mL.....	each.....			500-41H
PourRite Ampule Breaker.....	each.....			24846-00
pH Indicator Paper, pH 1 to 11 .....	5 rolls/pkg.....			391-33
pH Meter, <i>sensio</i> <sup>TM</sup> <b>I</b> , portable, with electrode .....	each.....			51700-10
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....			19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....			21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....			21856-28
Test Tube Rack .....	each.....			18641-00

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\* Marked Dropper Bottle - contact Hach for larger sizes.

**CHROMIUM, HEXAVALENT (0 to 0.60 mg/L Cr<sup>6+</sup>) For water and wastewater****1,5-Diphenylcarbohydrazide Method\* (Powder Pillows or AccuVac Ampuls)**

USEPA accepted for wastewater analyses\*\*

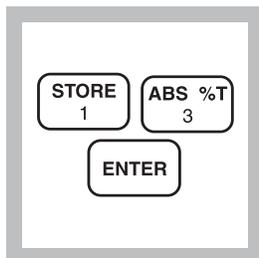
**Using Powder Pillows**

1. Enter the stored program number for hexavalent chromium (Cr<sup>6+</sup>)- powder pillows.

Press: **PRGM**

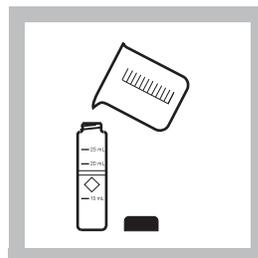
The display will show:

**PRGM ?**

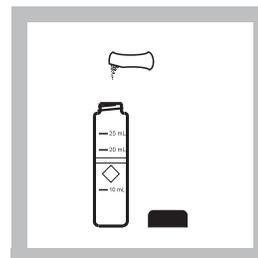


2. Press: **13 ENTER**  
The display will show **mg/L, Cr6** and the **ZERO** icon.

*Note: For alternate forms (CrO<sub>4</sub>, Cr<sub>2</sub>O<sub>7</sub>), press the **CONC** key.*

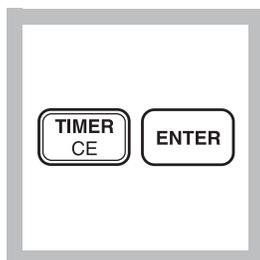


3. Fill a sample cell with 10 mL of sample.

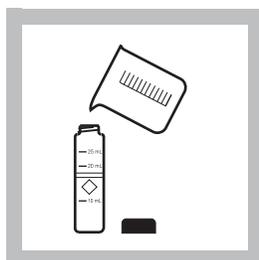


4. Add the contents of one ChromaVer 3 Reagent Powder Pillow to the cell (the prepared sample). Cap the cell and invert several times to mix.

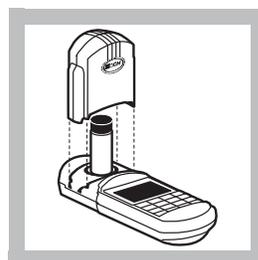
*Note: A purple color will form if Cr<sup>6+</sup> is present.*



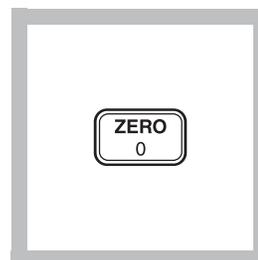
5. Press:  
**TIMER ENTER**  
A five-minute reaction period will begin.



6. Fill another sample cell with 10 mL of sample (the blank).  
*Note: For turbid samples, add the contents of one Acid Reagent Powder Pillow. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



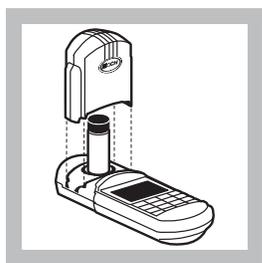
8. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Cr6**

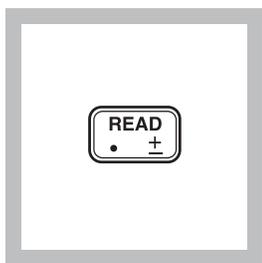
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

\*\* Procedure is equivalent to USGS method I-1230-85 for wastewater.

## CHROMIUM, HEXAVALENT, continued



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

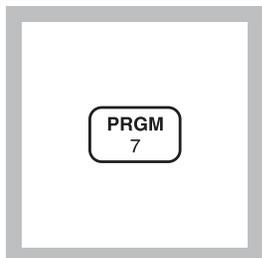


**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

### Using Accuvac Ampuls

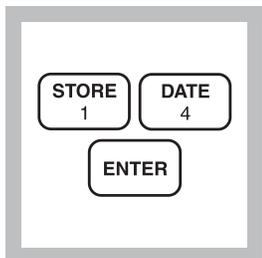


**1.** Enter the stored program number for hexavalent chromium ( $\text{Cr}^{6+}$ )- AccuVac Ampuls.

Press: **PRGM**

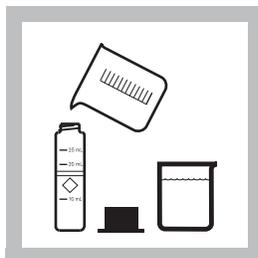
The display will show:

**PRGM ?**



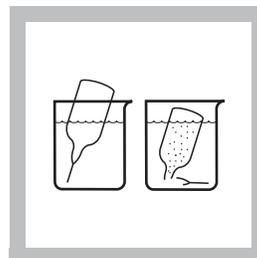
**2.** Press: **14 ENTER**  
The display will show **mg/L, Cr6** and the **ZERO** icon.

*Note: For alternate forms ( $\text{CrO}_4$ ,  $\text{Cr}_2\text{O}_7$ ), press the **CONC** key.*



**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

*Note: For turbid samples, add the contents of one Acid Reagent Powder Pillow to 10 mL of the blank. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



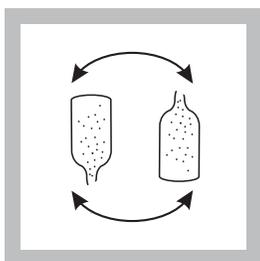
**4.** Fill a ChromaVer 3 Reagent AccuVac Ampul (the prepared sample) with sample.

*Note: Keep the tip immersed while the ampul fills completely.*

*Note: ChromaVer 3 should be white to tan in color. Replace if it is brown or green.*

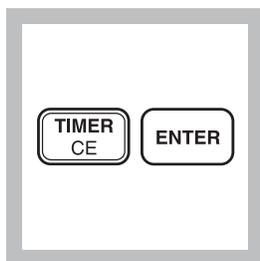
## CHROMIUM, HEXAVALENT, continued

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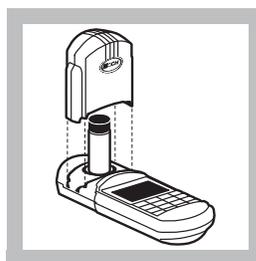


5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

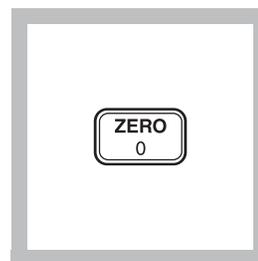
*Note: A purple color will form if hexavalent chromium is present.*



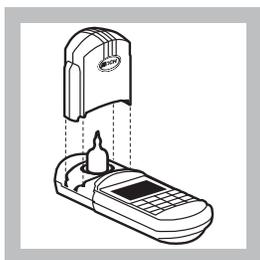
6. Press: **TIMER ENTER**  
A five-minute reaction period will begin.



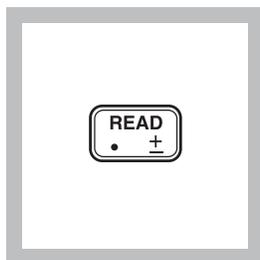
7. When the timer beeps place the blank into the cell holder.



8. Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L Cr<sup>6</sup>**



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**  
The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

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### Sampling and Storage

Collect samples in a cleaned glass or plastic container. Store at 4 °C (39 °F) up to 24 hours. Samples must be analyzed within 24 hours.

### Accuracy Check

#### Standard Additions Method (powder pillows)

- a) Snap the neck off a Hexavalent Chromium PourRite Standard Ampule, 5 mg/L Cr<sup>6+</sup>.

## CHROMIUM, HEXAVALENT, continued

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- b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL samples, respectively. Swirl to mix.
- c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Additions Method (AccuVac Ampuls)

- a) Snap the neck off a Hexavalent Chromium Voluette Standard Ampule, 12.5 mg/L Cr<sup>6+</sup>.
- b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 25-mL samples in beakers. Swirl gently to mix.
- c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 0.50-mg/L Cr<sup>6+</sup> solution by pipetting 10.00 mL of Hexavalent Chromium Standard Solution, 50.0 mg/L Cr<sup>6+</sup>, into a 1000-mL volumetric flask and diluting to the mark with deionized water. Invert repeatedly to mix. Prepare this solution daily. Perform the chromium procedure as described above, using this solution in place of the sample.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.6 mg/L Cr<sup>6+</sup> and two representative lots of powder pillow reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.008$  mg/L Cr<sup>6+</sup>.

In a single laboratory using a standard solution of 0.6 mg/L Cr<sup>6+</sup> and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.005$  mg/L Cr<sup>6+</sup>.

## CHROMIUM, HEXAVALENT, continued

### Estimated Detection Limit (EDL)

The EDL for program 13 (powder pillows) and program 14 (AccuVac Ampuls) is 0.01 mg/L Cr<sup>6+</sup>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

The following substances do not interfere in the test, up to the following concentration:

Substance	Concentration
Mercurous & Mercuric Ions	Interferes slightly
Iron	1 mg/L
Vanadium	1 mg/L. At higher levels vanadium interference can be overcome by waiting ten minutes before reading.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interference* in *Section 1*.

### Summary of Method

Hexavalent chromium is determined by the 1,5-diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-diphenylcarbohydrazide, which reacts to give a purple color which is proportional to the amount of hexavalent chromium present.

### REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required		
	Per Test	Unit	Cat. No.
ChromaVer 3 Chromium Reagent Powder Pillows..	1 pillow .....	100/pkg .....	12710-99
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06

### REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

ChromaVer 3 AccuVac Ampuls .....	1 ampul .....	25/pkg .....	25050-25
Beaker, 50 mL .....	1 .....	each .....	500-41H

## CHROMIUM, HEXAVALENT, continued

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### OPTIONAL REAGENTS

Description	Unit	Cat. No
Acid Reagent Powder Pillows .....	100/pkg.....	2126-99
Chromium, Hexavalent, Standard Solution, 50 mg/L Cr <sup>6+</sup> .....	100 mL.....	810-42
Chromium, Hexavalent, Standard Solution, Voluette Ampule, 12.5 mg/L Cr <sup>6+</sup> , 10 mL .....	16/pkg.....	14256-10
Chromium, Hexavalent, Standard Solution, PourRite Ampule, 5 mg/L Cr <sup>6+</sup> , 2 mL .....	20/pkg.....	26056-20
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

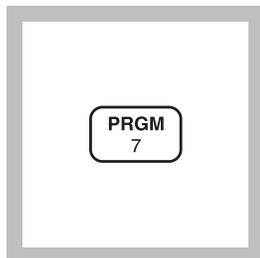
Description	Unit	Cat. No.
AccuVac Snapper Kit.....	each.....	24052-00
Ampule Breaker Kit.....	each.....	21968-00
Flask, volumetric, Class A, 1000 mL .....	each.....	14574-53
pH Paper, 1 to 11 pH units .....	5 rolls/pkg .....	391-33
pH Meter, EC10, portable .....	each.....	50050-00
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet, volumetric, 5.00 mL, Class A .....	each.....	14515-37
Pipet Filler, safety bulb .....	each.....	14651-00
PourRite Ampule Breaker, 2 mL .....	each.....	24846-00

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**CHROMIUM, TOTAL (0 to 0.60 mg/L)**

For water and wastewater

**Alkaline Hypobromite Oxidation Method\* \*\***

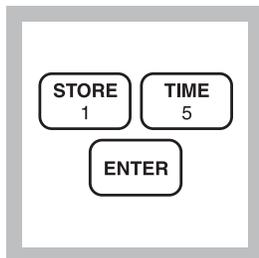
**1.** Enter the stored program number for total chromium (Cr).

Press: **PRGM**

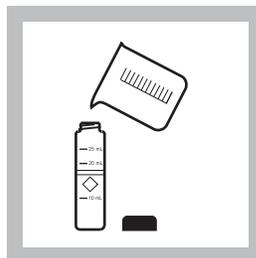
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

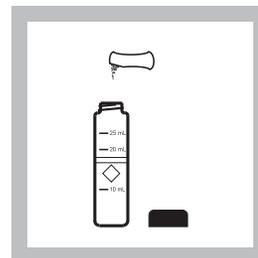


**2.** Press: **15 ENTER**  
The display will show **mg/L, Cr** and the **ZERO** icon.



**3.** Fill a clean sample cell with 25 mL of sample.

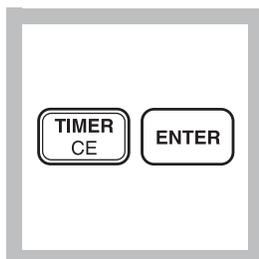
*Note: Adjust the pH of stored samples before analysis.*



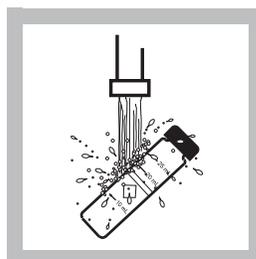
**4.** Add the contents of one Chromium 1 Reagent Powder Pillow (the prepared sample). Cap the cell and invert repeatedly to mix. Remove the cap.



**5.** Place the prepared sample into a boiling water bath.

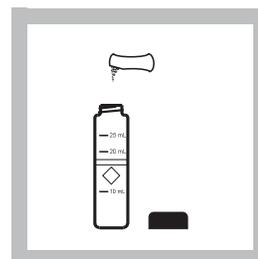


**6.** Press: **TIMER ENTER**  
A five-minute reaction period will begin.



**7.** After the beeper beeps, remove the prepared sample. Cap the cell. Use running tap water to cool the cell to 25 °C.

*Note: Use finger cots to handle the hot sample cell.*

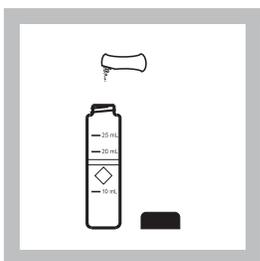


**8.** Add the contents of one Chromium 2 Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.

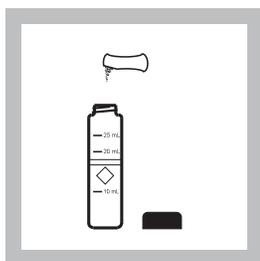
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

\*\* Procedure is equivalent to Standard Method 3500-Cr D for wastewater.

## CHROMIUM, TOTAL, continued



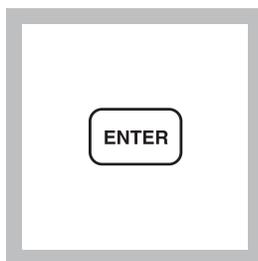
**9.** Add the contents of one Acid Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.



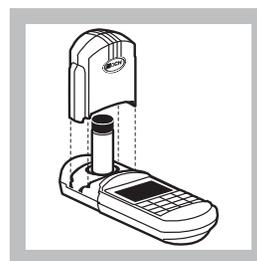
**10.** Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow. Cap the cell and invert repeatedly to mix.

*Note:* A purple color will form if chromium is present.

*Note:* ChromaVer 3 is white to tan in color. Replace brown or green powder. Undissolved powder does not affect accuracy.

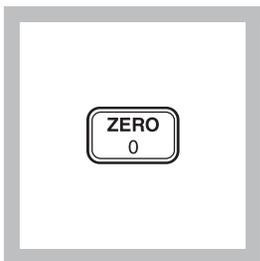


**11.** The display will show: **05:00 TIMER 2**  
Press: **ENTER**  
A five-minute reaction period will begin.



**12.** After the timer beeps, fill another sample cell with 25 mL of sample (the blank). Place it into the cell holder. Tightly cover the sample cell with the instrument cap.

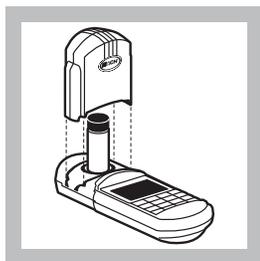
*Note:* For turbid samples, treat the blank as a sample, adding all reagents except the ChromaVer 3.



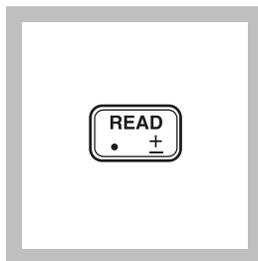
**13.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Cr**

*Note:* If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



**14.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**15.** Press: **READ**  
The cursor will move to the right, then the result in mg/L total chromium (Cr) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

## CHROMIUM, TOTAL, continued

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### Sampling and Storage

Collect samples in acid-washed glass or plastic containers. To preserve samples, adjust the pH to 2 or lower with nitric acid (about 2 mL per liter). Store preserved samples at room temperature up to six months. Adjust the pH to about 4 with 5.0 N Sodium Hydroxide before analysis. Correct the test results for volume additions (see *Section 1*).

### Accuracy Check

#### Standard Additions Method

- a) Fill three sample cells with 25 mL of sample.
- b) Snap the top off a Trivalent Chromium Standard Ampule, 12.5 mg/L as Cr<sup>3+</sup>.
- c) Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of standard to the three sample cells. Cap and invert repeatedly to mix .
- d) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur see *Standard Additions (Section 1)*.

#### Standard Solution Method

Prepare a 0.5 mg/L trivalent chromium standard by diluting 1.00 mL of Trivalent Chromium Standard Solution, 50 mg/L as Cr<sup>3+</sup>, to 100 mL with deionized water. Mix thoroughly. Prepare this solution daily. Perform the chromium procedure as described above. The mg/L Cr reading should be 0.5 mg/L.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.4 mg/L trivalent chromium and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.004$  mg/L chromium.

#### Estimated Detection Limit

The estimated detection limit for program 15 is 0.01 mg/L Cr. For more information on the estimated detection limit, see *Section 1*.

## CHROMIUM, TOTAL, continued

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### Interferences

Interfering Substance	Suggested Treatment
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see <i>pH Interferences</i> in <i>Section 1</i> .
Large amounts of organic material	May inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, see <i>Digestion</i> in <i>Section 2</i> for instruction on sample digestion. Perform the analysis as described on the digested sample.

### Summary of Method

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.

## CHROMIUM, TOTAL, continued

### REQUIRED REAGENTS

Total Chromium Reagent Set (100 Tests) .....	Cat. No.
	22425-00
Includes: (1) 2126-99, (1) 12066-99, (1) 2043-99, (1) 2044-99	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Acid Reagent Powder Pillows .....	1 pillow .....	100/pkg .....	2126-99
ChromaVer 3 Chromium Reagent Powder Pillows ..	1 pillow .....	100/pkg .....	12066-99
Chromium 1 Reagent Powder Pillows .....	1 pillow .....	100/pkg .....	2043-99
Chromium 2 Reagent Powder Pillows .....	1 pillow .....	100/pkg .....	2044-99

### REQUIRED APPARATUS

Hot plate, 4" diameter, 120 V .....	1 .....	each .....	12067-01
OR			
Hot plate, 4" diameter, 240 V .....	1 .....	each .....	12067-02
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06
Water bath and rack .....	1 .....	each .....	1955-55

### OPTIONAL REAGENTS

Chromium, trivalent, Standard Solution, 50 mg/L Cr <sup>3+</sup> .....	100 mL .....	14151-42
Chromium, trivalent, Standard Solution, PourRite ampule, 12.5 mg/L Cr <sup>3+</sup> , 10 mL .....	16/pkg .....	14257-10
Nitric Acid, ACS .....	500 mL .....	152-49
Nitric Acid Solution 1:1 .....	500 mL .....	2540-49
Sodium Hydroxide Standard Solution 5.0 N .....	50 mL* DB .....	2450-26
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Cylinder, graduated, polypropylene, 25 mL .....	each .....	1081-40
Finger Cots .....	2/pkg .....	14647-02
pH Paper, 1 to 11 pH units .....	5 rolls/pkg .....	391-33
pH Meter, <i>sension I</i> , with electrode .....	each .....	51700-10
Pipet, serological, 2 mL .....	each .....	532-36
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00
Ampule Breaker, 10-mL .....	each .....	21968-00

### *For Technical Assistance, Price and Ordering*

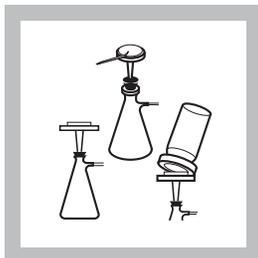
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

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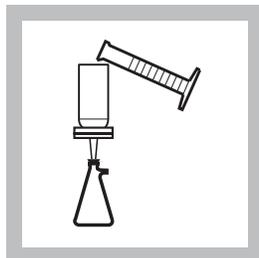
\* Contact Hach for larger sizes.



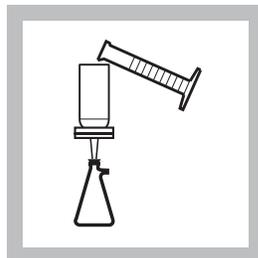
**COLOR, TRUE AND APPARENT (0 to 500 units)****APHA Platinum-Cobalt Standard Method\* For water, wastewater and seawater**

**1.** Assemble the filtering apparatus (membrane filter, filter holder, filter flask, and aspirator).

*Note:* To test for apparent color, do not filter; begin at Step 4 and skip Step 7.



**2.** Rinse the filter by pouring about 50 mL of deionized water through the filter. Discard the rinse water.

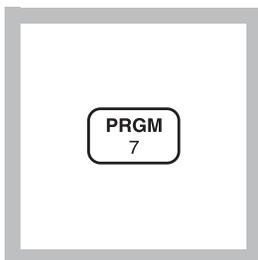


**3.** Pour another 50 mL of deionized water through the filter. Keep this for Step 4.



**4.** Fill a sample cell (the blank) with 25 mL of filtered deionized water. Discard the excess.

*Note:* For apparent color use unfiltered deionized water.

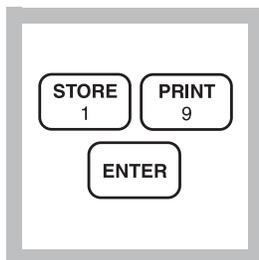


**5.** Enter the stored program number for APHA color.

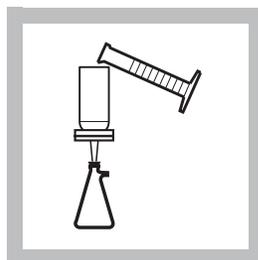
Press: **PRGM**

The display will show:

**PRGM ?**



**6.** Press: **19 ENTER**  
The display will show **PtCo** and the **ZERO** icon.



**7.** Pour about 50 mL of sample through the filter.

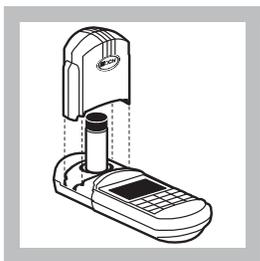


**8.** Fill a second sample cell (the prepared sample) with 25 mL of the filtered sample.

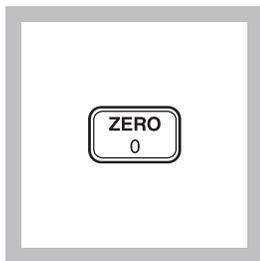
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*

## COLOR, TRUE AND APPARENT, continued

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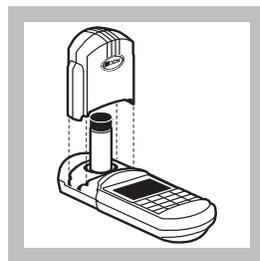


**9.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

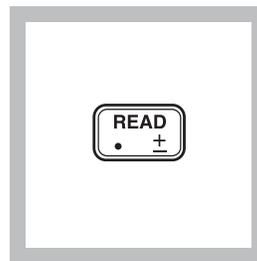


**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 mg/L Pt Co**



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in Platinum-Cobalt color units (Pt-Co) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze the sample as soon as possible after collection for best results. If prompt analysis is impossible, fill bottles completely and cap tightly. Avoid excessive agitation or prolonged contact with air. Samples can be stored for 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before running the test.

### Accuracy Check

#### Standard Solution Method

A 500 Platinum-Cobalt Units Color Standard solution is available for checking test accuracy. A 250 Platinum-Cobalt Units Standard can be made by pipetting 50.0 mL of the 500 Platinum-Cobalt Units Standard into a 100-mL volumetric flask and diluting to volume with deionized water.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 250 Pt-Co color units and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 10$  Pt-Co color units. For more information on Hach's precision statement, see *Section 1*.

## COLOR, TRUE AND APPARENT, continued

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### Estimated Detection Limit

The estimated detection limit for program 19 is 25 Pt-Co color units. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

Color may be expressed as “apparent” or “true” color. The apparent color includes color from dissolved materials plus that from suspended matter. By filtering or centrifuging out the suspended materials, the true color can be determined. The procedure describes true color analysis. If apparent color is desired, it can be determined by measuring an unfiltered water sample. The stored program is used for both forms of color.

---

### REQUIRED REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Units	
Water, deionized .....	50 mL .....	4 L .....	272-56

### REQUIRED APPARATUS

Aspirator, vacuum .....	1 .....	each .....	2131-00
Filter Holder, 47 mm, 300 mL graduated .....	1 .....	each .....	13529-00
Filter, membrane, 47 mm, 0.45 microns .....	1 .....	100/pkg .....	13530-00
Flask, filtering, 500 mL .....	1 .....	each .....	546-49
Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06
Stopper, No. 7, one hole .....	1 .....	6/pkg .....	2119-07

### OPTIONAL REAGENTS

Color Standard Solution, 500 platinum-cobalt units .....	1 L .....	1414-53
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### OPTIONAL APPARATUS

Cylinder, graduated, 50-mL, glass .....	each .....	508-41
Flask, volumetric, Class A, 100 mL .....	each .....	14574-42
Pipet, volumetric, Class A, 50 mL .....	each .....	14515-41
Thermometer, -20 to 110 °C, non-mercury .....	each .....	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

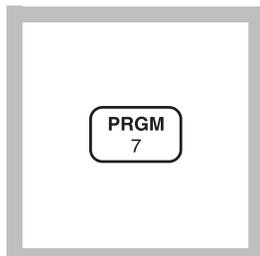
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**COPPER (0 to 5.00 mg/L)**

For water, wastewater and seawater\*

**Bicinchoninate Method\*\* (Powder Pillows or AccuVac Ampuls);  
USEPA approved for reporting wastewater analysis (digestion needed; See Section 2)\*\*\*  
Using Powder Pillows**



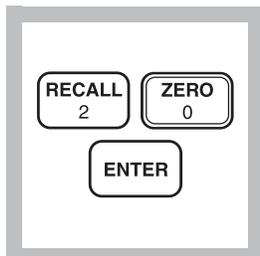
**1.** Enter the stored program number for bicinchoninate copper (Cu)- powder pillows.

Press: **PRGM**

The display will show:

**PRGM ?**

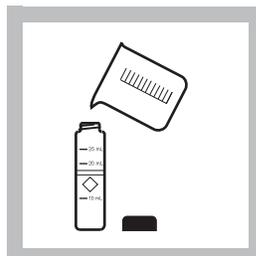
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **20 ENTER**

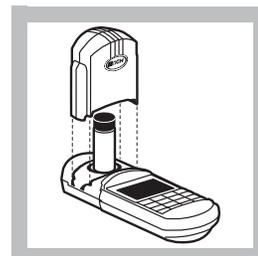
The display will show **mg/L, Cu** and the **ZERO** icon.

*Note: Determination of total copper needs a prior digestion (see Digestion in Section 2).*



**3.** Fill a sample cell with 10 mL of sample (the blank).

*Note: Adjust the pH of acid-preserved samples to 4-6 with 8 N KOH before analysis. Do not exceed pH 6 or copper may precipitate.*



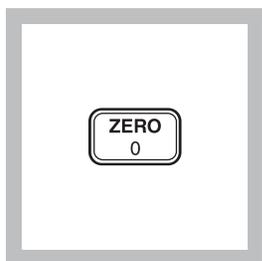
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

\* Pretreatment required; see *Interferences (Using Powder Pillows)*

\*\* Adapted from Nakano, S., *Yakugaku Zasshi*, 82 486-491 (1962) [*Chemical Abstracts*, 58 3390e (1963)]

\*\*\* Powder Pillows only: *Federal Register*, 45 (105) 36166 (May 29, 1980)

## COPPER, continued

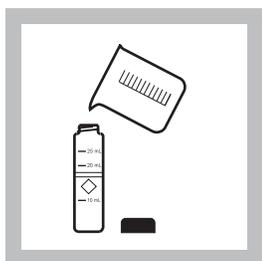


**5. Press: ZERO**

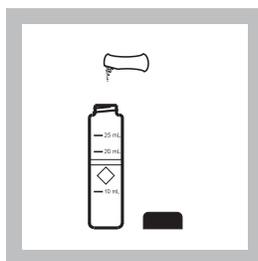
The cursor will move to the right, then the display will show:

**0.00 mg/L Cu**

*Note: If Reagent Blank Correction is on, the display may flash "limit".*

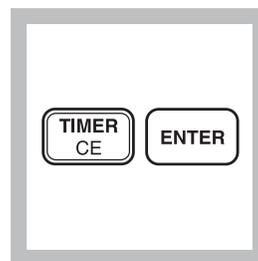


**6. Fill another sample cell with 10 mL of the sample.**



**7. Add the contents of one CuVer 1 Copper Reagent Powder Pillow to the sample cell (the prepared sample). Swirl the cell to mix.**

*Note: If copper is present, A purple color will develop.*

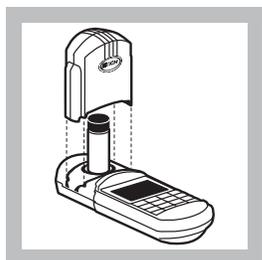


**8. Press:**

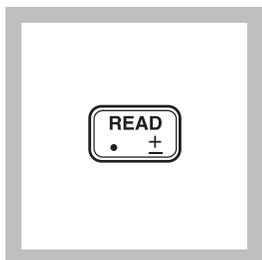
**TIMER ENTER**

A two-minute reaction period will begin.

*Note: Accuracy is not affected by undissolved powder.*



**9. Within 30 minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**



**10. Press: READ**

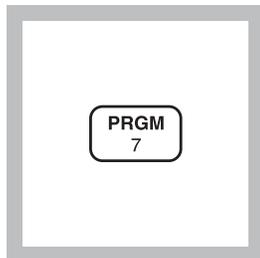
The cursor will move to the right, then the result in mg/L copper will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

## COPPER, continued

### Using AccuVac Ampuls

### Method 8026



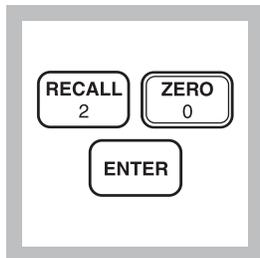
**1.** Enter the stored program number for bicinchoninate copper (Cu)- AccuVac ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

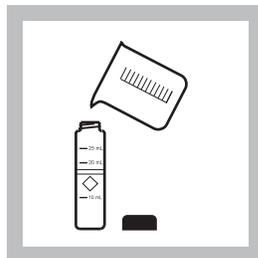


**2.** Press: **20 ENTER**

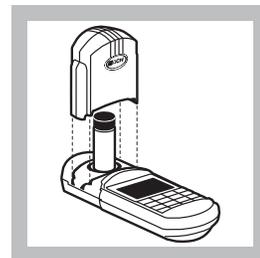
The display will show **mg/L, Cu** and the **ZERO** icon.

*Note: Determination of total copper needs a prior digestion (see Digestion in Section 2).*

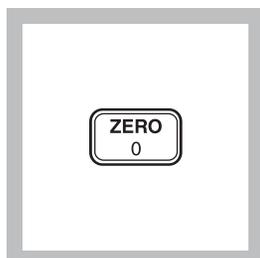
*Note: Adjust the pH of stored samples before analysis.*



**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

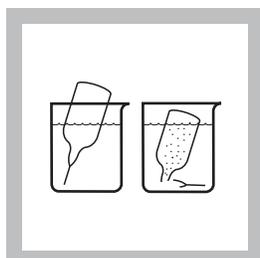


**5.** Press: **ZERO**

The cursor will move to the right, then the display will show:

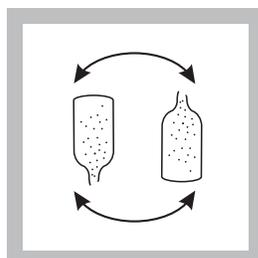
**0.00 mg/L Cu**

*Note: If Reagent Blank Correction is on, the display may flash "limit".*



**6.** Fill a CuVer 2 Copper Reagent AccuVac Ampul with sample.

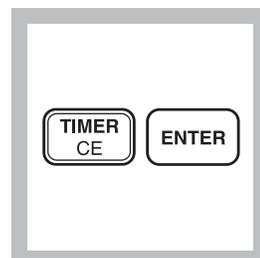
*Note: Keep the tip immersed while the ampul fills completely.*



**7.** Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

*Note: A purple color will form if copper is present.*

*Note: Accuracy is not affected by undissolved powder*



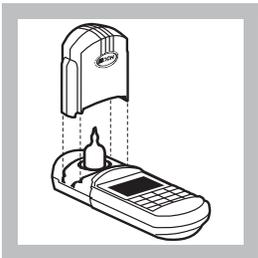
**8.** Press:

**TIMER ENTER**

A two-minute reaction period will begin.

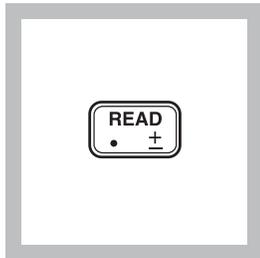
## COPPER, continued

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**9.** After the timer beeps, place the AccuVac ampul in the cell holder. Tightly cover the sample cell with the instrument cap.

*Note:* Step 10 must be completed within 30 minutes after the timer beeps.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L copper (Cu) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see in Section 1).

## COPPER, continued

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### Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4 to 6 with 8 N potassium hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved copper is to be determined, filter the sample before acid addition using the labware listed under *Optional Apparatus*.

### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Copper Voluette Ampule Standard, 75 mg/L as Cu.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to the mixing cylinders. Stopper and mix thoroughly.
- d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50-mL beakers to fill the ampules. For analysis with powder pillows, transfer only 10 mL of the solution to 10-mL sample cells.
- e) Analyze each sample as described in the procedure. The copper concentration should increase about 0.3 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Solution Method

Prepare a 1.00 mg/L copper standard by pipetting 1.00 mL of Copper Standard Solution, 100 mg/L as Cu, into 100-mL volumetric flask. Dilute to volume with deionized water and mix well. Prepare this solution daily. Using this solution as the sample, perform the copper procedure as described above.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 2.25 mg/L Cu

## COPPER, continued

and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Cu.

In a single laboratory, using a standard solution of 2.25 mg/L Cu and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Cu.

### Estimated Detection Limit (EDL)

The EDL for program 20 (Powder Pillows and AccuVac Ampuls) is 0.02 mg/L Cu. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Interferences

### Interfering Substances and Suggested Treatments for Powder Pillows

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution dropwise while swirling to dissolve the turbidity. Continue with Step 3.
Aluminum, Al <sup>3+</sup>	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Cyanide, CN <sup>-</sup>	Prevents full color development. Add 0.2 mL of formaldehyde to the 10-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Iron, Fe <sup>3+</sup>	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Silver, Ag <sup>+</sup>	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

To differentiate free copper from that complexed to EDTA or other complexing agents, use a Free Copper Reagent Powder

## COPPER, continued

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Pillow in place of the CuVer 1 pillow in Step 4. Results in Step 10 will be free copper only. Add a Hydrosulfite Reagent Powder Pillow to the same sample and re-read the result. This result will include the total dissolved copper (free and complexed).

### Interfering Substances and Suggested Treatments for AccuVac Ampuls

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution dropwise until sample pH is above 4. Continue with Step 3.
Aluminum, Al <sup>3+</sup>	Reagents accommodate high levels.
Cyanide, CN <sup>-</sup>	Prevents full color development. Add 1.0 mL of formaldehyde to a 50-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Reagents accommodate high levels
Iron, Fe <sup>3+</sup>	Reagents accommodate high levels
Silver, Ag <sup>+</sup>	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Unlike CuVer 1 Reagent, CuVer 2 Reagent reacts directly with copper which is complexed by chelants such as EDTA. If free copper is to be determined separately from complexed copper, see the Powder Pillow Interference section above.

### Summary of Method

Copper in the sample reacts with a salt of bichinonic acid contained in CuVer 1 or 2 Copper Reagent to form a purple colored complex in proportion to the copper concentration. This method includes procedures for both powder pillow and AccuVac reagents.

## COPPER, continued

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
CuVer 1 Copper Reagent Powder Pillows .....	1 pillow .....	100/pkg.....		21058-69
Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg.....		24019-06

### REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

CuVer 2 Copper Reagent AccuVac Ampuls .....	1 ampul .....	25/pkg.....		25040-25
Beaker, 50 mL.....	1 .....	each.....		500-41H

### OPTIONAL REAGENTS

Copper Standard Solution, 100 mg/L .....	100 mL.....			128-42
Copper Standard Solution, Voluette Ampule, 75 mg/L Cu, 10 mL.....	16/pkg.....			14247-10
CuVer 2 Reagent Powder Pillows .....	100/pkg.....			21882-99
Formaldehyde, 37%, ACS .....	100 mL* MDB.....			2059-32
Free Copper Reagent Powder Pillows .....	100/pkg.....			21186-69
Hydrochloric Acid Solution, 6.0 N.....	500 mL.....			884-49
Hydrosulfite Reagent Powder Pillows.....	100/pkg.....			21188-69
Metals Drinking Water Standard, LR for Cu, Fe, Mn .....	500 mL.....			28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn .....	500 mL.....			28336-49
Nitric Acid, ACS.....	500 mL.....			152-49
Nitric Acid Solution, 1:1 .....	500 mL.....			2540-49
Potassium Chloride Solution, saturated .....	100 mL .....			765-42
Potassium Hydroxide Standard Solution, 8.0 N .....	100 mL* MDB.....			282-32H
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL* MDB.....			2450-32
Water, deionized.....	4 L.....			272-56

### OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit.....	each.....	24052-00
Ampule Breaker Kit.....	each.....	21968-00
Cylinder, graduated, mixing, 25 mL.....	each.....	20886-40
Cylinder, graduated, polypropylene, 25 mL .....	each.....	1081-40
Cylinder, graduated, 100 mL .....	each.....	508-42
Filter Paper, folded, 12.5 cm.....	100/pkg.....	1894-57
Filter Pump .....	each.....	2131-00
Flask, volumetric, 100 mL, Class A .....	each.....	14547-42
Funnel, polypropylene, 65 mm .....	each.....	1083-67
Hot Plate, 4" diameter, 120 V .....	each.....	12067-01
Hot Plate, 4" diameter, 240 V .....	each.....	12067-02
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg.....	391-33
pH Meter, sens <i>ion</i> 1, with electrode .....	each.....	51700-10
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01

\* Contact Hach for larger sizes.

## **COPPER, continued**

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Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00

### ***For Technical Assistance, Price and Ordering***

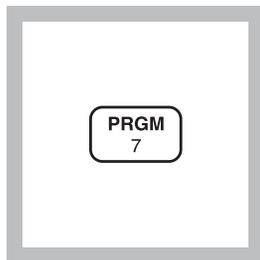
**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**



**COPPER (0 to 210.0 µg/L)**

For water, wastewater and seawater

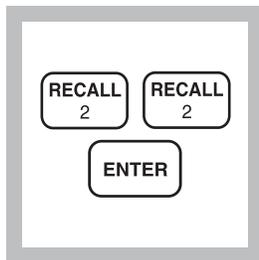
**Porphyrin Method\***

1. Enter the stored program number for copper (Cu), porphyrin method.

Press: **PRGM**

The display will show:

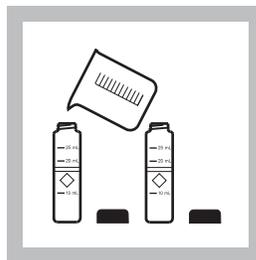
**PRGM ?**



2. Press: **22 ENTER**

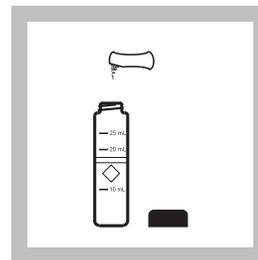
The display will show **µg/L, Cu** and the **ZERO** icon.

*Note: Total copper determination needs a prior digestion; use either the Digesdahl or vigorous digestion (Section 2).*



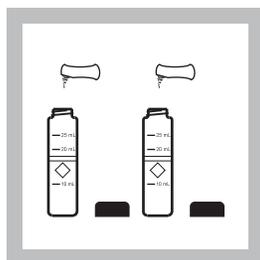
3. Fill two sample cells with 10 mL of sample.

*Note: Wash all glassware with detergent. Rinse with tap water. Rinse again with Nitric Acid Solution, 1:1. Rinse a third time with copper-free, deionized water.*

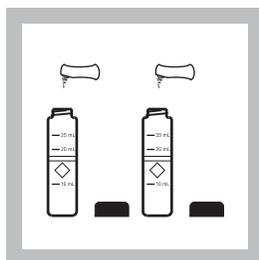


4. Add the contents of one Copper Masking Reagent Powder Pillow to one of the sample cells (the blank). Swirl to dissolve.

*Note: The other sample cell is the prepared sample.*

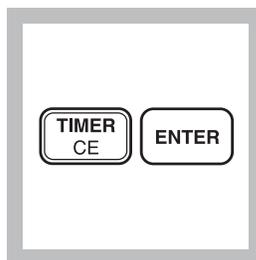


5. Add the contents of one Porphyrin 1 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.



6. Add the contents of one Porphyrin 2 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.

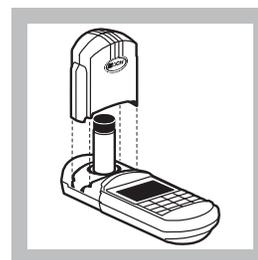
*Note: The yellow color will turn blue momentarily. If any copper is present, the yellow color will return.*



7. Press:

**TIMER ENTER**

A three-minute reaction period will begin.

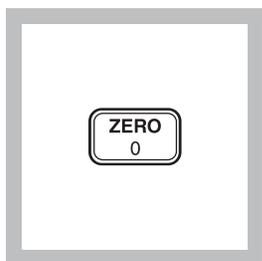


8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

\* Adapted from Ishii and Koh, *Bunseki Kagaku*, 28 473 (1979)

## COPPER, continued

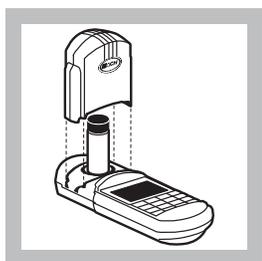
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**9. Press: ZERO**

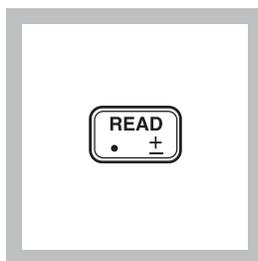
The cursor will move to the right, then the display will show:

**0.0 µg/L Cu**



**10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**

*Note: If samples with high levels of metal are analyzed, a slight metallic deposit or yellow buildup may appear on the sample cell wall. Remove by rinsing with nitric acid. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*



**11. Press: READ**

The cursor will move to the right, then the result in µg/L copper (Cu) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

---

### Sampling and Storage

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature.

Before testing, adjust the pH of the sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution. Correct test results for volume additions; see *Correction for Volume Additions* in Section 1 for more information.

## COPPER, continued

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### Accuracy Check

#### Standard Additions Method

- a) Fill six (3 pairs) 25-mL graduated mixing cylinders with 25 mL of sample. Properly mark each pair of cylinders as “sample” and “blank”.
- b) Using a TenSette Pipet, add 0.1 mL of Copper Standard Solution, 10.0 mg/L Cu, to two of the cylinders. Add 0.2 mL of standard to two more of the cylinders. Add 0.3 mL of standard to the other two cylinders, making a total of six samples (2 for each volume of standard).
- c) Analyze the samples as described above. The copper concentration reading should increase by 40  $\mu\text{g/L}$  for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions in Section 1* for more information.

#### Standard Solution Method

To assure the accuracy of the test, prepare a 100  $\mu\text{g/L}$  copper standard:

- a) Pipet 1.00 mL of Copper Standard Solution, 10.0 mg/L Cu, into a 100-mL volumetric flask.
- b) Dilute to volume with copper-free, reagent-grade water.
- c) Use this standard in place of the sample in the procedure. The reading should be 100  $\mu\text{g/L}$  Cu.
- d) Prepare this solution daily.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 100  $\mu\text{g/L}$  copper and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 3.4$   $\mu\text{g/L}$  copper.

#### Estimated Detection Limit

The estimated detection limit for program 22 is 5.4  $\mu\text{g/L}$  Cu. For more information on the estimated detection limit, see *Section 1*.

## COPPER, continued

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### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Concentration	Substance	Concentration
Aluminum	60 mg/L	Magnesium	10,000 mg/L
Cadmium	10 mg/L	Manganese	140 mg/L
Calcium	15,000 mg/L	Mercury	3 mg/L
Chloride	90,000 mg/L	Molybdenum	11 mg/L
Chromium (Cr <sup>6+</sup> )	110 mg/L	Nickel	60 mg/L
Cobalt	100 mg/L	Potassium	60,000 mg/L
Fluoride	30,000 mg/L	Sodium	90,000 mg/L
Iron (Fe <sup>2+</sup> )	6 mg/L	Zinc	9 mg/L
Lead	3 mg/L		

Chelating agents, such as EDTA, interfere at all levels unless either the Digesdahl or vigorous digestion (*Section 2*) is performed.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment: see pH Interferences in *Section 1*.

### Summary of Method

The porphyrin method is very sensitive to trace amounts of free copper. Due to the sensitivity of the method, a masking agent is used to prepare a “blank” for each sample. The method is free from most interferences and does not require any sample extraction or preconcentration. Interferences from other metals are eliminated by the copper masking reagent. The porphyrin indicator forms an intense, yellow-colored complex proportional to any free copper present in the sample. Total copper may be determined if a digestion is performed prior to analysis.

## COPPER, continued

### REQUIRED REAGENTS

	Cat. No.
Copper Reagent Set, 10-mL samples (100 tests) .....	26033-00
Includes: (1) 26034-49, (2) 26035-49, (2) 26036-49	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Copper Masking Reagent Powder Pillows.....	1 pillow.....	100/pkg .....	26034-49	
Porphyrim 1 Reagent Powder Pillows.....	2 pillows.....	100/pkg .....	26035-49	
Porphyrim 2 Reagent Powder Pillows.....	2 pillows.....	100/pkg .....	26036-49	

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ caps .....	2 .....	6/pkg .....	24019-06
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### OPTIONAL REAGENTS

Copper Standard Solution, 10 mg/L Cu .....	100 mL .....	128-42
Hydrochloric Acid Solution, 1:1 (6 N).....	500 mL .....	884-49
Nitric Acid, ACS .....	500 mL .....	152-49
Nitric Acid Solution, 1:1 .....	500 mL .....	2540-49
Sodium Hydroxide Standard Solution, 5 N.....	1 L .....	2450-53
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Beaker, 100 mL .....	each .....	500-42H
Cylinder, mixing, graduated, 25 mL .....	each .....	20886-40
Flask, volumetric, Class A, 100 mL.....	each .....	14574-42
Hot Plate, 7 x 7 inches, 120 V.....	each .....	23441-00
Hot Plate, 7 x 7 inches, 240 V.....	each .....	23441-02
pH Paper, 1 to 11 pH units .....	5 rolls/pkg .....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode.....	each .....	51700-10
Pipet, Mohr, 5 mL .....	each .....	20934-37
Pipet, TenSette, 0.1 to 1.0 mL.....	each .....	19700-01
Pipet Tips, for 19700-01.....	50/pkg .....	21856-96
Pipet Tips, for 19700-01.....	1000/pkg .....	21856-28
Pipet, volumetric, 1.0 mL, Class A .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00
Watch Glass, Pyrex <sup>®</sup> , 100 mL.....	each .....	578-70

### *For Technical Assistance, Price and Ordering*

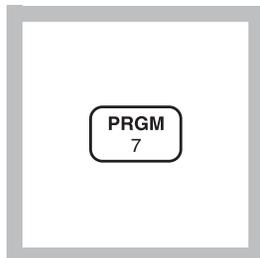
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**CYANIDE (0 to 0.240 mg/L)**

For water, wastewater, and seawater

**Pyridine-Pyrazalone Method\***

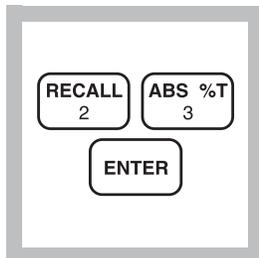
**1.** Enter the stored program number for cyanide (CN).

Press: **PRGM**

The display will show:

**PRGM ?**

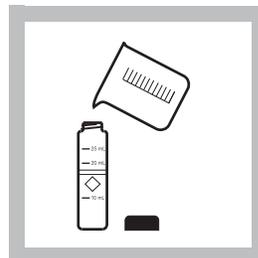
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **23 ENTER**

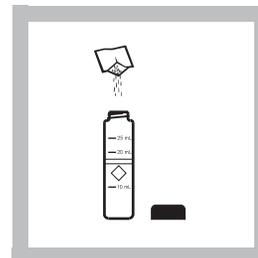
The display will show **mg/L, CN** and the **ZERO** icon.

*Note: Adjust the pH of stored samples before analysis.*



**3.** Fill a sample cell with 10-mL of sample.

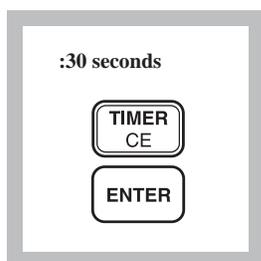
*Note: Samples at less than 23 °C require a longer reaction time and samples at greater than 25 °C give low test results. Sample temperature must be 23-25 °C.*



**4.** Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the sample cell.

\* Adapted from Epstein, Joseph, *Anal. Chem.* 19 (4), 272 (1947)

## CYANIDE, continued



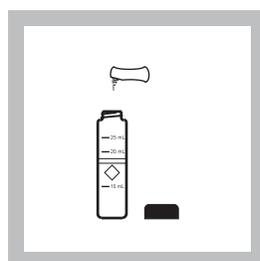
**5. Press: **TIMER**  
**ENTER****

A 30-second reaction period will begin. Shake the sample cell for the 30 seconds.

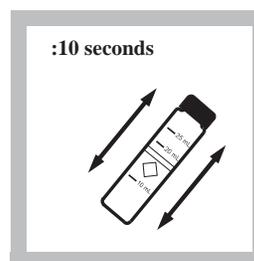


**6. After the first timer beeps, the display will show: **0:30 TIMER 2****  
**Press **ENTER**.**

A 30-second reaction period will begin. Let the sample cell sit undisturbed for this 30-second period.



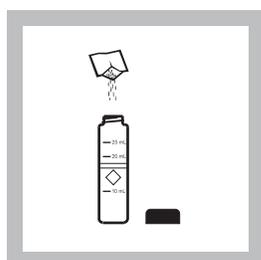
**7. After the timer beeps, add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow.**  
**Cap the sample cell.**



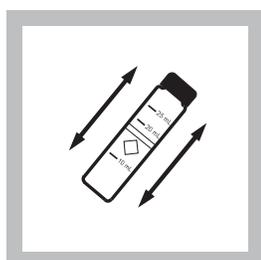
**8. Shake the sample cell for ten seconds.**  
**Immediately proceed with Step 9.**

*Note: Delaying the addition of the CyaniVer 5 Cyanide Reagent Powder for more than 30 seconds after the addition of the CyaniVer 4 Cyanide Reagent Powder will give lower test results.*

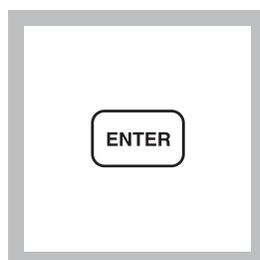
*Note: Accuracy is not affected by undissolved CyaniVer 4 Cyanide Reagent Powder.*



**9. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow.**  
**Cap the cell.**



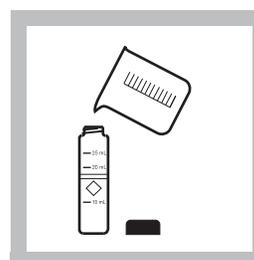
**10. Shake vigorously to completely dissolve the CyaniVer 5 Cyanide Reagent Powder (the prepared sample).**



**11. The display will show: **30:00 Timer 3****  
**Press: **ENTER****

A 30-minute reaction period will begin.

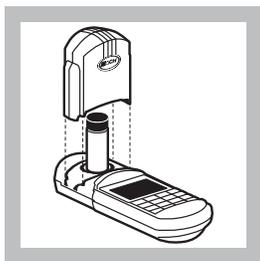
*Note: If cyanide is present, a pink color will develop which then turns blue after a few minutes.*



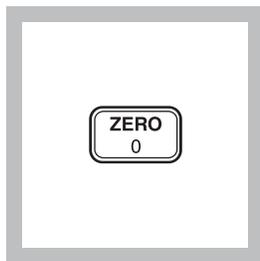
**12. Fill another 10-mL sample cell (the blank) with 10 mL of sample.**

## CYANIDE, continued

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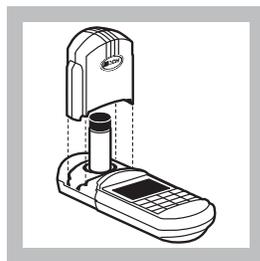
**13.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



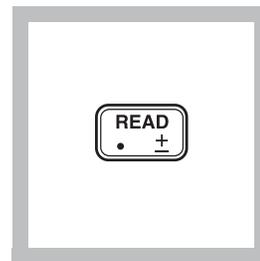
**14.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.000 mg/L CN**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**15.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**16.** Press: **READ**  
The cursor will move to the right, then the result in mg/L cyanide (CN) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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## Sampling and Storage

Collect samples in glass or plastic bottles and analyze as soon as possible.

The presence of oxidizing agents, sulfides and fatty acids can cause cyanide loss during sample storage. Samples containing these substances must be pretreated as described in the following procedures before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

Preserve the sample by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH. Four mL of sodium hydroxide are usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N sodium hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N sodium hydroxide or samples that are highly alkaline due to chlorination treatment processes or distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard

## CYANIDE, continued

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Solution. If significant amounts of preservative are used, correct for the volume added; see *Correction for Volume Additions* in *Section 1* for more information.

### Oxidizing Agents

Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and eliminate their effect, pretreat the sample as follows:

- a) Take a 25-mL portion of the sample and add one drop of m-Nitrophenol Indicator Solution, 10 g/L. Swirl to mix.
- b) Add 2.5 N Hydrochloric Acid Standard Solution dropwise until the color changes from yellow to colorless. Swirl the sample thoroughly after the addition of each drop.
- c) Add two drops of Potassium Iodide Solution, 30 g/L, and two drops of Starch Indicator Solution, to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.
- d) If Step c suggests the presence of oxidizing agents, add two level 1-g measuring spoonfuls of ascorbic acid per liter of sample.
- e) Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat Steps a to c. If the sample turns blue, repeat Steps d and e.
- f) If the 25-mL sample remains colorless, adjust the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution (usually 4 mL/L).
- g) Perform the procedure given under Interferences, Reducing Agents, to eliminate the effect of excess ascorbic acid, before following the cyanide procedure.

### Sulfides

Sulfides quickly convert cyanide to thiocyanate (SCN). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

- a) Place a drop of sample on a disc of hydrogen sulfide test paper that has been wetted with pH 4 Buffer Solution.

## CYANIDE, continued

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- b) If the test paper darkens, add a 1-g measuring spoon of lead acetate to the sample. Repeat Step a. (Purchase lead acetate from a local supplier.)
- c) If the test paper continues to turn dark, keep adding lead acetate until the sample tests negative for sulfide.
- d) Filter the black lead sulfide precipitate using the apparatus listed under Optional Apparatus. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution or neutralize to a pH of 7 for analysis.

### Fatty Acids

*Caution—perform this operation in a hood as quickly as possible.*

When distilled, fatty acids will pass over with cyanide and form soaps under the alkaline conditions of the absorber. If the presence of fatty acid is suspected, do not preserve samples with sodium hydroxide until the following pretreatment is performed. The effect of fatty acids can be minimized as follows:

- a) Acidify 500 mL of sample to pH 6 or 7 with Acetic Acid Solution. (Prepare a 1:10 dilution of Acetate Acid concentration in water.)
- b) Pour the sample into a 1000-mL separatory funnel and add 50 mL of hexane.
- c) Stopper the funnel and shake for one minute. Allow the layers to separate.
- d) Drain off the sample (lower) layer into a 600-mL beaker. If the sample is to be stored, add 5 N Sodium Hydroxide Standard Solution to raise the pH to above 12.

## Accuracy Check

### Standard Solution Method

*Caution—Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.*

Prepare a 100 mg/L cyanide stock solution weekly by dissolving 0.2503 grams of potassium cyanide in deionized water and diluting to 1000 mL.

## CYANIDE, continued

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Immediately before use, prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of the 100 mg/L stock solution to 1000 mL using deionized water. Use this prepared standard in place of sample in Step 3. Results should be 0.10 mg/L CN<sup>-</sup>.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.19 mg/L CN<sup>-</sup> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L CN<sup>-</sup>.

#### Estimated Detection Limit (EDL)

The estimated detection limit for program 23 is 0.008 mg/L CN<sup>-</sup>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

#### Turbidity

Large amounts of turbidity will interfere and cause high readings. If the water sample is highly turbid, it should first be filtered before use in Steps 3 and 12. Filter using the labware listed under Optional Apparatus. The test results should then be recorded as soluble cyanide.

#### Oxidizing and Reducing Agents

Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the CyaniVer 5 Reagent. If chlorine or other oxidizing agents are known to be present, or if reducing agents (such as sulfide or sulfur dioxide) are known to be present, use adequate ventilation and pretreat the sample before testing as follows:

#### Oxidizing Agents

- a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops of acid added.
- b) Add two drops of Potassium Iodide Solution and two drops of Starch Indicator Solution to the sample. Swirl to mix. The sample will turn blue if oxidizing agents are present.

## CYANIDE, continued

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- c) Add Sodium Arsenite Solution drop-wise until the sample turns colorless. Swirl the sample thoroughly after each drop. Count the number of drops.
- d) Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.
- e) Subtract one drop from the amount of Sodium Arsenite Solution added in Step c. Add this amount to the sample and mix thoroughly.
- f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

### Reducing Agents

- a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops added.
- b) Add four drops of Potassium Iodide Solution and four drops of Starch Indicator Solution to the sample. Swirl to mix. The sample should be colorless.
- c) Add Bromine Water drop-wise until a blue color appears. Count the number of drops, and swirl the sample after the addition of each drop.
- d) Take another 25 mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.
- e) Add the total number of drops of Bromine Water counted in Step c to the sample and mix thoroughly.
- f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

### Metals

Nickel or cobalt in concentrations up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L copper and 5 mg/L iron by adding the contents of one HexaVer Chelating Reagent Powder Pillow to the sample and then mixing before adding the CyaniVer 3 Cyanide Reagent Powder Pillow in Step 4. Prepare a reagent blank of deionized water and reagents to zero the instrument in Step 13.

## CYANIDE, continued

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### Acid Distillation

For USEPA reporting purposes, samples must be distilled.

All samples should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The “rotten egg” smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L thiocyanate.

The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate treatment procedure.

- a) Place a drop of the distillate (already diluted to 250 mL) on a disc of hydrogen sulfide test paper that has been wetted with pH 4.0 Buffer Solution.
- b) If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution drop-wise to the distillate until a neutral pH is obtained.
- c) Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat Step a.
- d) If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide.
- e) Filter the black lead sulfide precipitate through filter paper and funnel. This sample should now be neutralized to pH 7 and analyzed for cyanide without delay.

## CYANIDE, continued

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### Distillation Procedures

A detailed procedure for the distillation of cyanide samples is included with the Hach Distillation Apparatus. Three detailed procedures, Free Cyanides, Cyanides Amenable to Chlorination, and Total Cyanides, are included with the four- and ten-position Midi-Dist Distillation System. See the Optional Apparatus listing.

### Summary of Method

The pyridine-pyrazolone method gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes.

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### REQUIRED REAGENTS

	Cat. No.
Cyanide Reagent Set (100 Tests), 10 mL samples .....	24302-00
Includes: (1) 21068-69, (1) 21069-69, (1) 21070-69	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
CyaniVer 3 Cyanide Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	21068-69
CyaniVer 4 Cyanide Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	21069-69
CyaniVer 5 Cyanide Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	21070-69

### REQUIRED APPARATUS

Sample Cell, 10-20-25, w/cap .....	2 .....	6/pkg .....	24019-06
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### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Acetic Acid, Glacial .....	500 mL .....	100-49
Ascorbic Acid.....	100 g .....	6138-26
Bromine Water .....	25 mL .....	2211-20
Buffer Solution, pH 4.0 .....	500 mL .....	12223-49
Hexanes, ACS .....	500 mL .....	14478-49
HexaVer Chelating Reagent Powder Pillows .....	100/pkg .....	243-99
Hydrochloric Acid Standard Solution, 2.5 N .....	100 mL MDB .....	1418-32
Magnesium Chloride Solution .....	1 L .....	14762-53
m-Nitrophenol Indicator.....	100 mL MDB .....	2476-32
Potassium Iodide Solution, 30 g/L .....	100 mL MDB .....	343-32
Sodium Arsenite Solution, APHA .....	100 mL MDB .....	1047-32
Potassium Cyanide, ACS .....	28 g .....	767-14
Sodium Hydroxide Standard Solution, 0.25 N.....	1 L .....	14763-53
Sodium Hydroxide Standard Solution, 5.0 N.....	1 L .....	2450-53

## CYANIDE, continued

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### OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Starch Indicator Solution .....	10 mL MDB .....	349-32
Sulfuric Acid Standard Solution, 19.2 N .....	500 mL .....	2038-49
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Beaker, glass, 600 mL .....	each .....	500-52
Bottle, wash, 500 mL .....	each .....	620-11
Cylinder, graduated, 50 mL .....	each .....	508-41
Cylinder, graduated, 250 mL .....	each .....	508-46
Distillation Apparatus, cyanide accessories .....	each .....	22658-00
Distillation Apparatus, general purpose accessories .....	each .....	22653-00
Distillation Apparatus Heater and Support Apparatus, 115 Vac, 60 Hz .....	each .....	22744-00
Distillation Apparatus Heater and Support Apparatus, 230 Vac, 50 Hz .....	each .....	22744-02
Dropper, plastic .....	each .....	6080-00
Filter Paper, folded, 12.5 cm .....	100/pkg .....	1894-57
Flask, volumetric, Class A, 1000 mL .....	each .....	14574-53
Flask, volumetric, Class A, 250 mL .....	each .....	14574-46
Funnel, poly, 65 mm .....	each .....	1083-67
Funnel, separatory, 500 mL .....	each .....	520-49
Hydrogen Sulfide Test Papers .....	100/pkg .....	25377-33
pH Meter, <i>sension</i> <sup>TM</sup> <b>I</b> , portable .....	each .....	51700-10
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00
Scoop, double ended .....	each .....	12257-00
Spoon, measuring, 1.0 g .....	each .....	510-00
Support Ring, 4 inch .....	each .....	580-01
Support Stand .....	each .....	563-00
Thermometer, -20 to 110 °C, non-mercury .....	each .....	26357-02

### *For Technical Assistance, Price and Ordering*

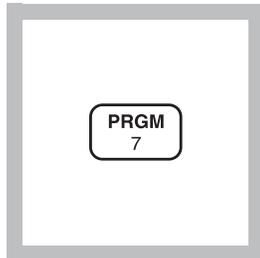
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# CYANURIC ACID (7 to 55 mg/L)

Method 8139  
For water, pools and spas

## Turbidimetric Method



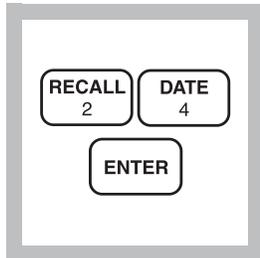
1. Enter the stored program number for cyanuric acid.

Press: **PRGM**

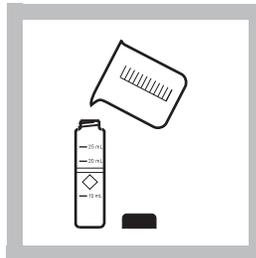
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

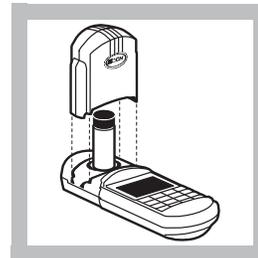


2. Press: **24 ENTER**  
The display will show **mg/L, CYACD** and the **ZERO** icon.

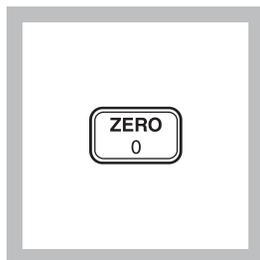


3. Fill a sample cell with 25 mL of sample (the blank).

*Note: Filtering is required for highly turbid samples.*



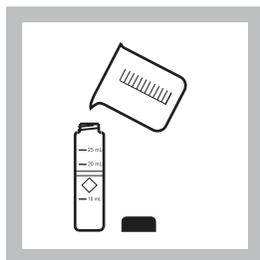
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 mg/L CYACD**

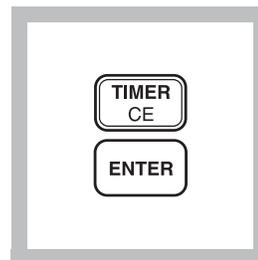
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



6. Fill another cell with 25 mL of sample.



7. Add the contents of one Cyanuric Acid 2 Reagent Powder Pillow (the prepared sample). Swirl to mix.



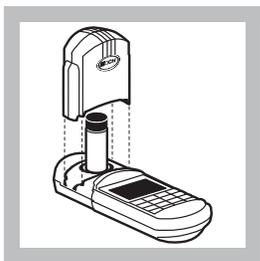
8. Press **TIMER ENTER**  
A three-minute reaction period will begin.

*Note: A white turbidity will form if cyanuric acid is present.*

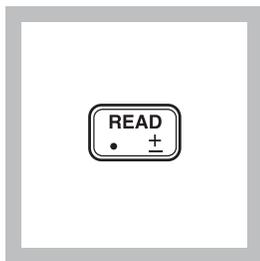
*Note: Accuracy is not affected by undissolved powder.*

## CYANURIC ACID, continued

---



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L cyanuric acid will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

*Note: Clean sample cells with soap, water and a brush soon after each test to prevent a white film from forming.*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

### Accuracy Check

#### Standard Solution Method

- a) Dissolve 1.000 gram of cyanuric acid in 1000 mL of deionized water to make a 1000 mg/L solution. It takes several hours for the cyanuric acid to dissolve. This solution is stable for several weeks.
- b) Dilute 2.00 mL of the 1000 mg/L solution to 100 mL with deionized water to make a 20 mg/L solution. Prepare fresh daily.
- c) Testing the 20 mg/L solution should give test results of about 20 mg/L cyanuric acid.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 25.0 mg/L cyanuric acid and two lots of reagent with the instrument, a single

## CYANURIC ACID, continued

---

operator obtained a standard deviation of  $\pm 1.2$  mg/L cyanuric acid.

### Estimated Detection Limit

The estimated detection limit for program 24 is 7.0 mg/L cyanuric acid. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Turbidity will interfere. Filter turbid samples before running the test.

### Summary of Method

The test for cyanuric acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any cyanuric acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present. Due to the nature of the precipitation reaction, low levels of cyanuric acid (less than 7 mg/L) are not detected by this method.

---

## REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Cyanuric Acid 2 Reagent Powder Pillow.....	1 pillow.....	50/pkg .....	2460-66	
Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06	

## OPTIONAL REAGENTS

Cyanuric Acid .....	25 g .....	7129-24
Water, deionized .....	4 L .....	272-56

## OPTIONAL APPARATUS

Balance, Acculab UI Series.....	each .....	26947-00
Filter Paper, folded 12.5 cm .....	100/pkg .....	1894-57
Flask, volumetric, Class A, 100 mL.....	each .....	14574-42
Flask, volumetric, Class A, 1000 mL.....	each .....	14574-53
Funnel, poly, 65 mm.....	each .....	1083-67
Pipet, Bulb.....	each .....	14651-00
Pipet, volumetric, Class A, 2.00 mL .....	each .....	14515-36

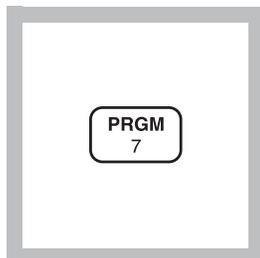
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



## Iron Reduction Method for Oxygen Scavengers\*



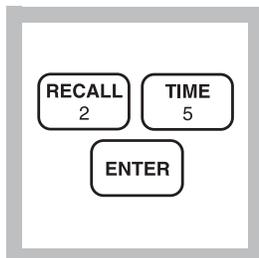
1. Enter the stored program number for diethylhydroxylamine (DEHA).

Press: **PRGM**

The display will show:

**PRGM ?**

*Note:* To determine other oxygen scavengers, multiply the result by the appropriate factor. See Other Oxygen Scavengers following these steps.

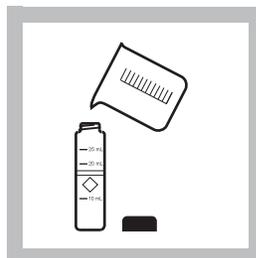


2. Press: **25 ENTER**

The display will show **µg/L, DEHA** and the **ZERO** icon.

*Note:* To prevent contamination from iron deposits, rinse sampling containers and sample cells with 1:1 Hydrochloric Acid Solution. Follow with several rinsings of deionized water.

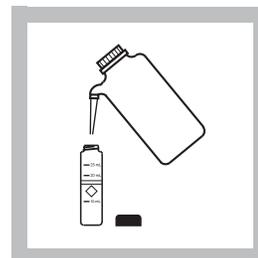
*Note:* Samples must be analyzed immediately.



3. Fill a sample cell with 25 mL of sample (the prepared sample).

*Note:* The sample temperature should be  $25 \pm 3^\circ\text{C}$  ( $77 \pm 5^\circ\text{F}$ ).

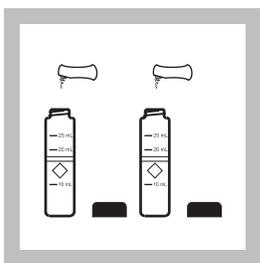
*Note:* When testing for compounds that react quickly with oxygen at room temperature, stopper the cell containing the sample in Steps 5–11.



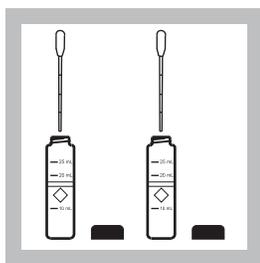
4. Fill a second sample cell with 25 mL of deionized water (the blank).

\* Adapted from Ishii and Koh, *Bunseki Kagaku*, 28 473 (1979)

## DEHA (N,N-DIETHYLHYDROXYLAMINE), continued

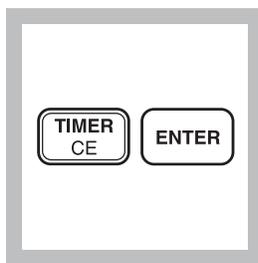


**5.** Add the contents of one DEHA Reagent 1 Powder Pillow to each sample cell. Cap. Swirl to mix.



**6.** Add exactly 0.5 mL of DEHA Reagent 2 Solution to each sample cell. Cap and swirl to mix. Place both sample cells in the dark.

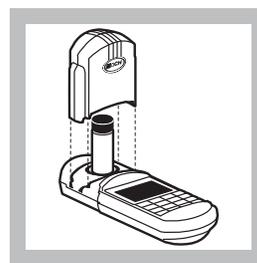
*Note: A purple color will slowly develop if DEHA is present.*



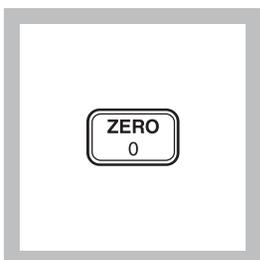
**7.** Immediately, press: **TIMER ENTER**  
A 10-minute reaction period will begin. For hydroquinone, allow only a two-minute reaction period.

*Note: Both sample cells must remain in the dark for the entire reaction period.*

*Note: Temperature and reaction time affect results.*

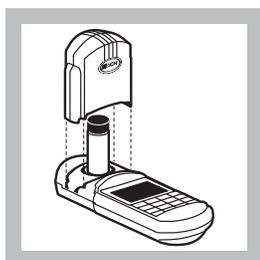


**8.** Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

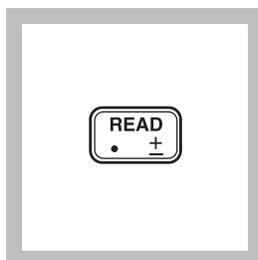


**9.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 µg/L DEHA**



**10.** Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**11.** Press: **READ**  
The cursor will move to the right, then the result in µg/L DEHA will be displayed.

*Note: If the display flashes "limit" it is due to high DEHA levels. Dilute a fresh sample with deoxygenated deionized water and repeat the test. Multiply the result by the dilution factor; see Section 1.*

### **Ferrous Iron Adjustment**

*Note: Repeat the above procedure, but do not add DEHA Reagent 2 (Step 6) to determine the ferrous iron content in the sample. Then press **SETUP**, scroll to "BLANK" and press **ENTER**. The display will show; "BLANK?" Enter the blank value just read. Press **ENTER** to accept the value as the blank to be subtracted from each reading.*

## DEHA (N,N-DIETHYLHYDROXYLAMINE), continued

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### Sampling and Storage

Most oxygen scavengers will react quickly with atmospheric oxygen. Collect samples in acid-rinsed plastic or glass containers, allowing the sample to overflow. Cap the container so there is no head space above the sample. Rinse each sample cell several times with sample, then carefully fill to the fill mark. Analyze the sample immediately.

### Other Oxygen Scavengers

To determine other oxygen scavengers, perform the test as directed above, then multiply the DEHA result by the appropriate factor below:

Oxygen Scavenger	Factor
Erythorbic Acid (Iso-ascorbic acid)	3.5
Hydroquinone	2.5
Methylethylketoxime (MEKO)	4.1
Carbohydrazide	1.3

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 242 µg/L DEHA and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±6.2 µg/L DEHA.

#### Estimated Detection Limit

The estimated detection limit for program 25 is 9 µg/L DEHA. For more information on the estimated detection limit, see *Section 1*.

### Interferences

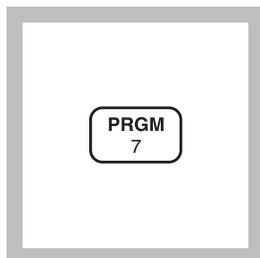
Substances which reduce ferric iron will interfere. Substances which complex iron strongly may also interfere. Light interferes with the color development. The following may also interfere when present in concentrations exceeding those listed below:

Borate (as Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> )	500 mg/L	Molybdenum	80 mg/L
Cobalt	0.025 mg/L	Nickel	0.8 mg/L
Copper	8.0 mg/L	Phosphate	10 mg/L
Hardness (as CaCO <sub>3</sub> )	1000 mg/L	Phosphonates	10 mg/L
Lignosulfonates	0.05 mg/L	Sulfate	1000 mg/L
Manganese	0.8 mg/L	Zinc	50 mg/L



**FLUORIDE (0 to 2.00 mg/L F<sup>-</sup>)**

For water, wastewater and seawater

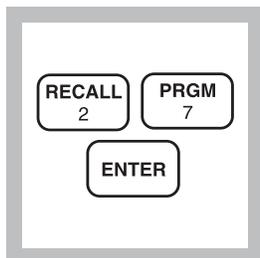
**SPADNS Method\* (Reagent Solution or AccuVac Ampuls)****Using SPADNS Reagent Solution**

1. Enter the stored program number for fluoride (F) powder pillows.

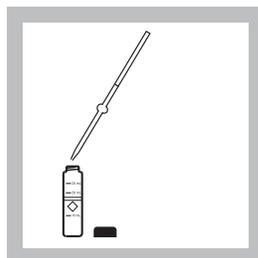
Press: **PRGM**

The display will show:

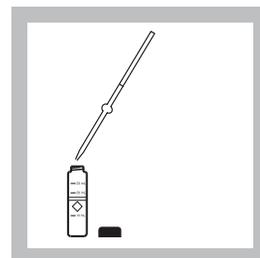
**PRGM ?**



2. Press: **27 ENTER**  
The display will show **mg/L, F** and the **ZERO** icon.

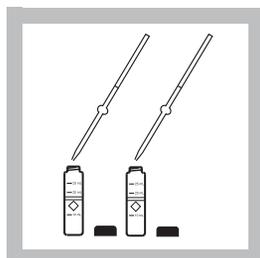


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



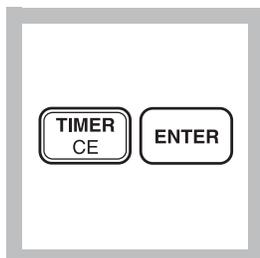
4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

*Note: The sample and blank should be at the same temperature ( $\pm 1$  °C). Temperature adjustments may be made before or after reagent addition.*

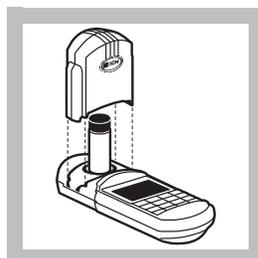


5. Pipet 2.00 mL of SPADNS Reagent into each cell. Swirl to mix.  
*Note: SPADNS Reagent is toxic and corrosive; use care while measuring. Use a pipet filler.*

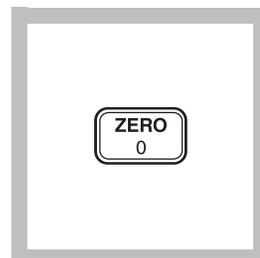
*Note: The SPADNS Reagent must be measured accurately.*



6. Press: **TIMER ENTER**  
A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

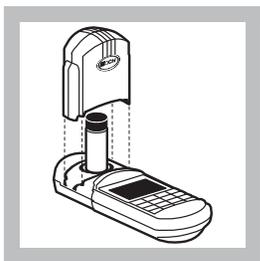


8. Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L F**

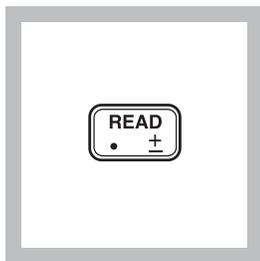
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*. The procedure for this instrument uses an alternate wavelength outside the accepted 550-580 nm range. The reagents used are the same as those in the USEPA accepted method.

## FLUORIDE, continued

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**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

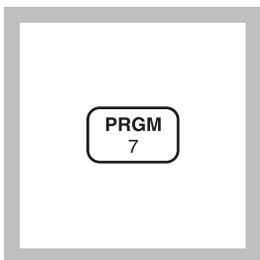


**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

### Using AccuVac Ampuls

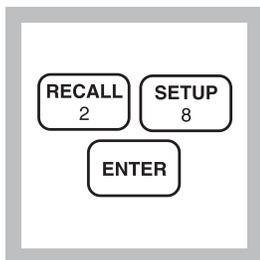


**1.** Enter the stored program number for fluoride (F<sup>-</sup>)- AccuVac Ampuls.

Press: **PRGM**

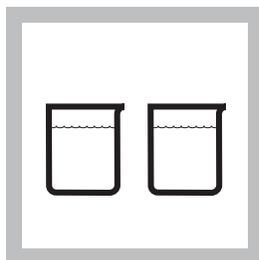
The display will show:

**PRGM ?**

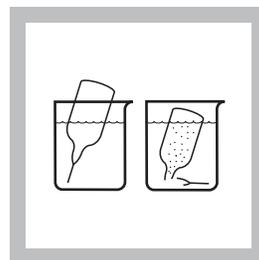


**2.** Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.



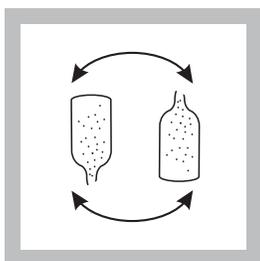
**3.** Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.



**4.** Fill a SPADNS Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

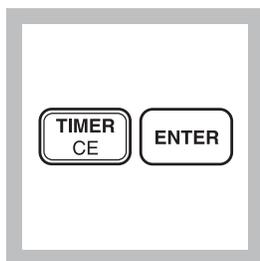
*Note: Keep the tip immersed while the ampule fills completely.*

## FLUORIDE, continued



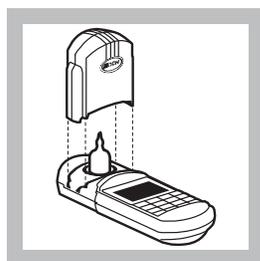
**5.** Quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.

*Note: Do not place finger over the broken tip- the liquid will remain in the ampul.*

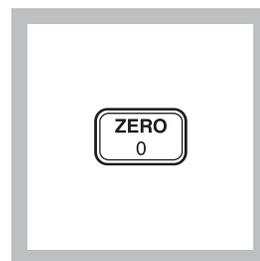


**6.** Press: **TIMER ENTER**

A one-minute reaction period will begin.

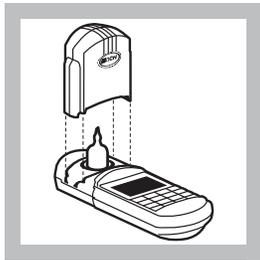


**7.** After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.

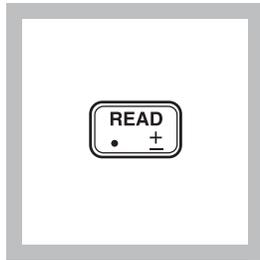


**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.0 mg/L F**



**9.** Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

## FLUORIDE, continued

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### Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

### Accuracy Check

#### Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above

1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the “STD” setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.035$  mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS AccuVac Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.040$  mg/L fluoride.

#### Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F<sup>-</sup>. For more information on derivation and use of Hach’s estimated detection limit, see *Section 1*.

## FLUORIDE, continued

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### Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as CaCO <sub>3</sub> )	5000 mg/L	-0.1 mg/L F <sup>-</sup>
Aluminum	0.1 mg/L	-0.1 mg/L F <sup>-</sup>
Chloride	7000 mg/L	+0.1 mg/L F <sup>-</sup>
Iron, ferric	10 mg/L	-0.1 mg/L F <sup>-</sup>
Phosphate, ortho	16 mg/L	+0.1 mg/L F <sup>-</sup>
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F <sup>-</sup>
Sulfate	200 mg/L	+0.1 mg/L F <sup>-</sup>

SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution to 25 mL of sample for each 2 mg/L of chlorine.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

- a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.
- b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch. Turn the stir control to 5.
- c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each mg/L chloride present.

## FLUORIDE, continued

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- d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.
- e) When the temperature reaches 180 °C (about one hour), turn the still off.
- f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

### Summary of Method

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

### Pollution Prevention and Waste Management

SPADNS Reagent contains sodium arsenite. Final solutions will contain sodium arsenite (D004) in sufficient concentration to be regulated as hazardous waste for Federal RCRA. See *Section 3* for more information on disposal of these materials.

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### REQUIRED REAGENTS (Using Solution)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
SPADNS Reagent for Fluoride .....	4 mL.....	500 mL.....	444-49	
Water, deionized.....	10 mL.....	4 L.....	272-56	

### REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb.....	1.....	each.....	14651-00
Pipet, volumetric, Class A, 10.00 mL.....	1.....	each.....	14515-38
Pipet, volumetric, Class A, 2.00 mL.....	1.....	each.....	14515-36
Sample Cell, 10-20-25 mL w/ cap.....	2.....	6/pkg.....	24019-06
Thermometer, -20 to 110°C, non-mercury.....	1.....	each.....	26357-02

### REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS Fluoride Reagent AccuVac Ampuls.....	2 ampuls.....	25/pkg.....	25060-25
Water, deionized.....	varies.....	4 L.....	272-56

## FLUORIDE, continued

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### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....2 .....each .....500-41H

### OPTIONAL REAGENTS

#### Drinking Water Inorganics Standard

for F<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, PO<sub>4</sub><sup>3-</sup>, and SO<sub>4</sub><sup>2-</sup> ..... 500 mL .....28330-49  
Fluoride Standard Solution, 0.2 mg/L F<sup>-</sup> ..... 500 mL .....405-02  
Fluoride Standard Solution, 0.5 mg/L F<sup>-</sup> ..... 500 mL .....405-05  
Fluoride Standard Solution, 0.8 mg/L F<sup>-</sup> ..... 500 mL .....405-08  
Fluoride Standard Solution, 1.0 mg/L F<sup>-</sup> ..... 1000 mL .....291-53  
Fluoride Standard Solution, 1.0 mg/L F<sup>-</sup> ..... 500 mL .....291-49  
Fluoride Standard Solution, 1.2 mg/L F<sup>-</sup> ..... 500 mL .....405-12  
Fluoride Standard Solution, 1.5 mg/L F<sup>-</sup> ..... 500 mL .....405-15  
Fluoride Standard Solution, 2.0 mg/L F<sup>-</sup> ..... 500 mL .....405-20  
Silver Sulfate, ACS ..... 113 g .....334-14  
Sodium Arsenite Solution ..... 100 mL MDB .....1047-32  
StillVer Distillation Solution ..... 500 mL ..... 446-49

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....each .....24052-00  
Cylinder, graduated, 100 mL.....each .....508-42  
Cylinder, graduated, 250 mL.....each .....508-46  
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz .....each .....22744-00  
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz .....each .....22744-02  
Distillation Apparatus General Purpose Accessories.....each .....22653-00  
pH Meter, *sensIon*<sup>™</sup>**I**, portable, with electrode.....each .....51700-10  
Pipet, TenSette, 1.0 to 10.0 mL.....each .....19700-10  
Pipet Tips, for 19700-10 TenSette Pipet .....50/pkg .....21997-96  
Stopper .....6/pkg .....1731-06

### *For Technical Assistance, Price and Ordering*

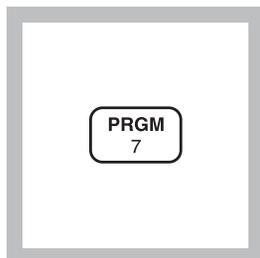
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**FLUORIDE (0 to 2.00 mg/L F<sup>-</sup>)**

For water, wastewater and seawater

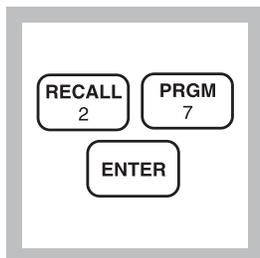
**SPADNS 2 Method<sup>\*</sup> (Reagent Solution or AccuVac Ampuls)****Using SPADNS 2 Reagent Solution**

1. Enter the stored program number for fluoride (F<sup>-</sup>) powder pillows.

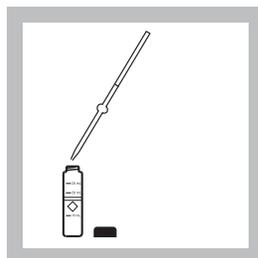
Press: **PRGM**

The display will show:

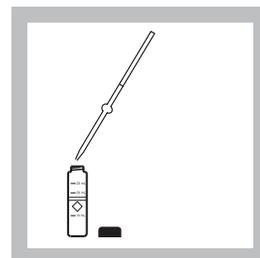
**PRGM ?**



2. Press: **2** **ENTER**  
The display will show **mg/L, F** and the **ZERO** icon.

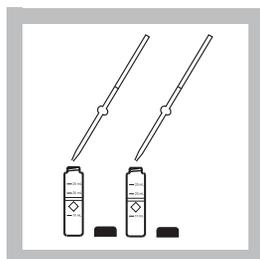


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

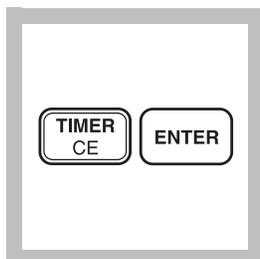
*Note: The sample and blank should be at the same temperature ( $\pm 1$  °C). Temperature adjustments may be made before or after reagent addition.*



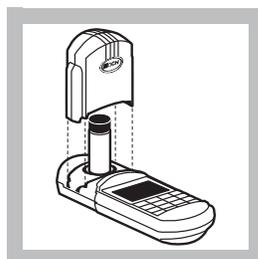
5. Pipet 2.00 mL of SPADNS 2 Reagent into each cell. Swirl to mix.

*Note: SPADNS 2 Reagent is corrosive; use care while measuring. Use a pipet filler.*

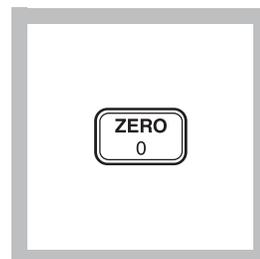
*Note: The SPADNS 2 Reagent must be measured accurately.*



6. Press: **TIMER** **ENTER**  
A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

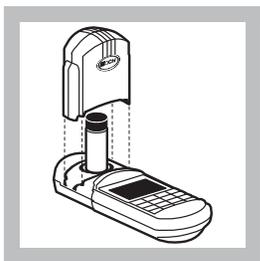


8. Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L F**

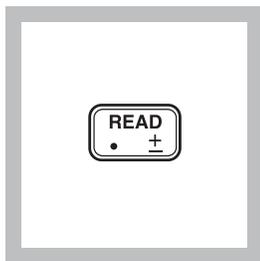
<sup>\*</sup> Adapted from *Standard Methods for the Examination of Water and Wastewater. Per USEPA Rules and Regulations at 40 CFR 136.6, Method Modifications and Analytical Requirements, Hach Method 10225 (SPADNS 2) for the determination of fluoride in water is equivalent to the EPA Reference Method SM 4500-F D. Equivalency data is available upon request.*

## FLUORIDE, continued

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**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

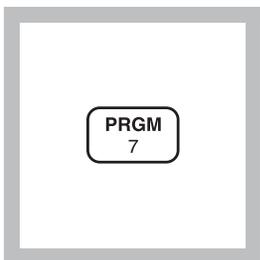


**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

### Using AccuVac Ampuls

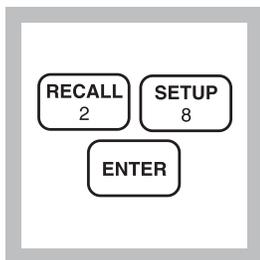


**1.** Enter the stored program number for fluoride (F<sup>-</sup>) AccuVac Ampuls.

Press: **PRGM**

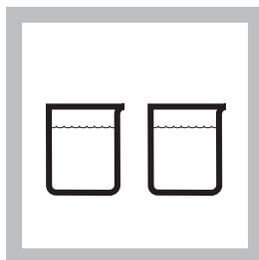
The display will show:

**PRGM ?**

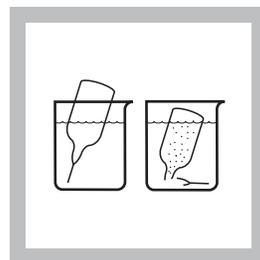


**2.** Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.



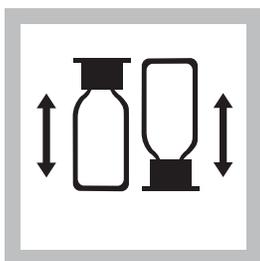
**3.** Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.



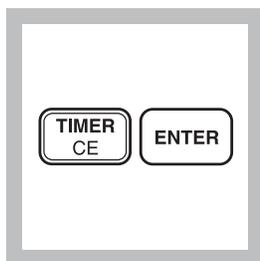
**4.** Fill an SPADNS 2 Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

*Note: Keep the tip immersed while the ampule fills completely.*

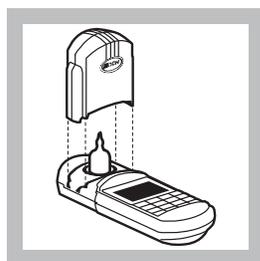
## FLUORIDE, continued



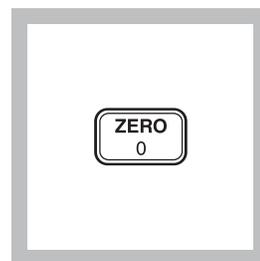
**5.** Cap and quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.



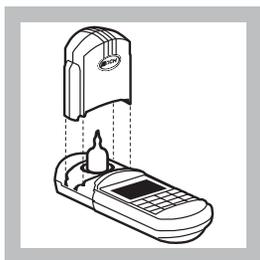
**6.** Press: **TIMER ENTER**  
A one-minute reaction period will begin.



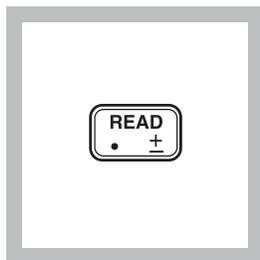
**7.** After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.



**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.0 mg/L F**



**9.** Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L fluoride will be displayed.  
*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

## FLUORIDE, continued

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### Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

### Accuracy Check

#### Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above

1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the “STD” setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.035$  mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 AccuVac Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.040$  mg/L fluoride.

#### Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F<sup>-</sup>. For more information on derivation and use of Hach’s estimated detection limit, see *Section 1*.

## FLUORIDE, continued

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### Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as CaCO <sub>3</sub> )	5000 mg/L	-0.1 mg/L F <sup>-</sup>
Aluminum	0.1 mg/L	-0.1 mg/L F <sup>-</sup>
Chloride	7000 mg/L	+0.1 mg/L F <sup>-</sup>
Iron, ferric	10 mg/L	-0.1 mg/L F <sup>-</sup>
Phosphate, ortho	16 mg/L	+0.1 mg/L F <sup>-</sup>
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F <sup>-</sup>
Sulfate	200 mg/L	+0.1 mg/L F <sup>-</sup>

SPADNS 2 Reagent contains enough non-toxic reducing agent to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, dilute sample with deionized water by a factor that will lower chlorine concentration to below 5 mg/L. Perform the procedure, and multiply results by this factor to obtain mg/L Fluoride.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

- a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.
- b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch. Turn the stir control to 5.
- c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each

## FLUORIDE, continued

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mg/L chloride present.

- d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.
- e) When the temperature reaches 180 °C (about one hour), turn the still off.
- f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

### Summary of Method

The SPADNS 2 Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

### Pollution Prevention and Waste Management

SPADNS 2 Reagent contains a non-toxic proprietary reducing agent in place of sodium arsenite.

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### REQUIRED REAGENTS (Using Solution)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
SPADNS 2 Reagent for Fluoride .....	4 mL.....	500 mL.....	29475-49	
Water, deionized.....	10 mL.....	4 L.....	272-56	

### REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb.....	1.....	each.....	14651-00
Pipet, volumetric, Class A, 10.00 mL.....	1.....	each.....	14515-38
Pipet, volumetric, Class A, 2.00 mL.....	1.....	each.....	14515-36
Sample Cell, 10-20-25 mL w/ cap.....	2.....	6/pkg.....	24019-06
Thermometer, -20 to 110°C, non-mercury.....	1.....	each.....	26357-02

### REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS 2 Fluoride Reagent AccuVac Ampuls.....	2 ampuls.....	25/pkg.....	25270-25
Water, deionized.....	varies.....	4 L.....	272-56

## FLUORIDE, continued

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### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....2 .....each .....500-41H

### OPTIONAL REAGENTS

#### Drinking Water Inorganics Standard

for F<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, PO<sub>4</sub><sup>3-</sup>, and SO<sub>4</sub><sup>2-</sup> ..... 500 mL .....28330-49  
Fluoride Standard Solution, 0.2 mg/L F<sup>-</sup> ..... 500 mL .....405-02  
Fluoride Standard Solution, 0.5 mg/L F<sup>-</sup> ..... 500 mL .....405-05  
Fluoride Standard Solution, 0.8 mg/L F<sup>-</sup> ..... 500 mL .....405-08  
Fluoride Standard Solution, 1.0 mg/L F<sup>-</sup> ..... 1000 mL .....291-53  
Fluoride Standard Solution, 1.0 mg/L F<sup>-</sup> ..... 500 mL .....291-49  
Fluoride Standard Solution, 1.2 mg/L F<sup>-</sup> ..... 500 mL .....405-12  
Fluoride Standard Solution, 1.5 mg/L F<sup>-</sup> ..... 500 mL .....405-15  
Fluoride Standard Solution, 2.0 mg/L F<sup>-</sup> ..... 500 mL .....405-20  
Silver Sulfate, ACS ..... 113 g .....334-14  
StillVer Distillation Solution ..... 500 mL ..... 446-49

### OPTIONAL APPARATUS

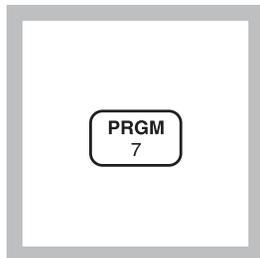
AccuVac Snapper Kit .....each .....24052-00  
Cylinder, graduated, 100 mL.....each .....508-42  
Cylinder, graduated, 250 mL.....each .....508-46  
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz .....each .....22744-00  
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz .....each .....22744-02  
Distillation Apparatus General Purpose Accessories.....each .....22653-00  
pH Meter, *sensio*<sup>TM</sup> 1, portable, with electrode.....each .....51700-10  
Pipet, TenSette, 1.0 to 10.0 mL .....each .....19700-10  
Pipet Tips, for 19700-10 TenSette Pipet .....50/pkg .....21997-96  
Stopper .....6/pkg .....1731-06

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**HARDNESS (0 to 4.00 mg/L Ca and Mg as CaCO<sub>3</sub>) For water, wastewater, seawater****Calcium and Magnesium; Calmagite Colorimetric Method**

**1.** Enter the stored program number for magnesium hardness (as CaCO<sub>3</sub>).

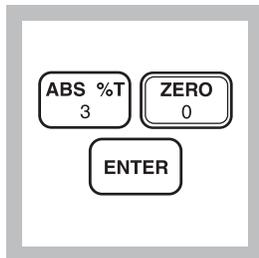
Press: **PRGM**

The display will show:

**PRGM ?**

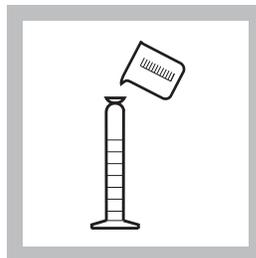
*Note:* Adjust the pH of stored samples before analysis.

*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



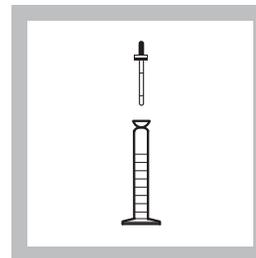
**2.** Press: **30 ENTER**  
The display will show **mg/L, CaCO<sub>3</sub>** and the **ZERO** icon.

*Note:* For alternate forms (Mg, MgCO<sub>3</sub>), press the **CONC** key.

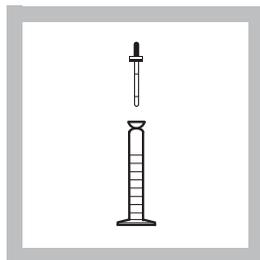


**3.** Pour 100 mL of sample into a 100-mL graduated mixing cylinder.

*Note:* The sample temperature should be 21-29 °C (70-84 °F).

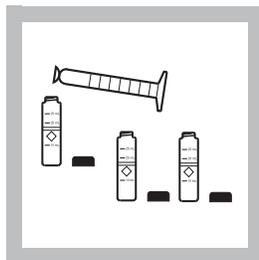


**4.** Add 1.0 mL of Calcium and Magnesium Indicator Solution using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.



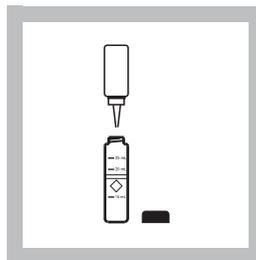
**5.** Add 1.0 mL of Alkali Solution for Calcium and Magnesium Test using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.

*Note:* If the sample turns read after adding Alkali Solution, dilute sample 1:1 and repeat analysis.

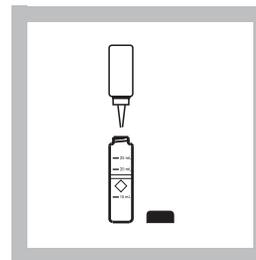


**6.** Pour 10 mL of the solution into each of three sample cells.

*Note:* The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers or sample cells. To test cleanliness, repeat the test multiple times until you obtain consistent results.

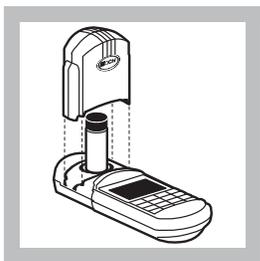


**7.** Add one drop of 1 M EDTA Solution to one cell (the blank). Swirl to mix.

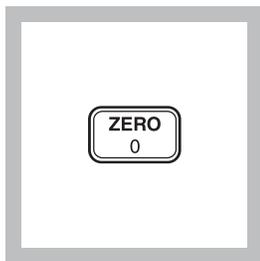


**8.** Add one drop of EGTA Solution to another cell (the prepared sample). Swirl to mix.

## HARDNESS, continued



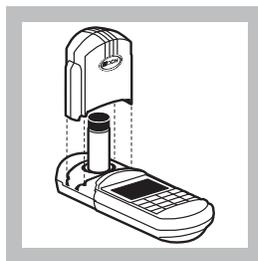
**9.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



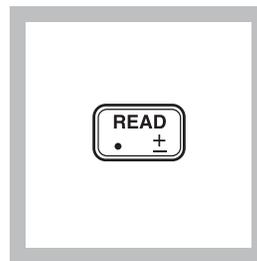
**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L CaCO<sub>3</sub>**

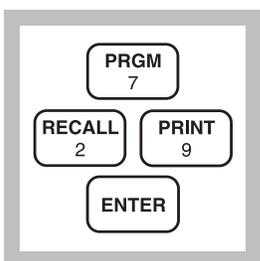
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L magnesium hardness (as CaCO<sub>3</sub>) will be displayed.



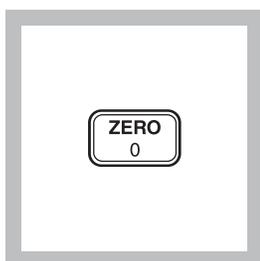
**13.** Without removing the cell, press:

**PRGM 29 ENTER**

The display will show:

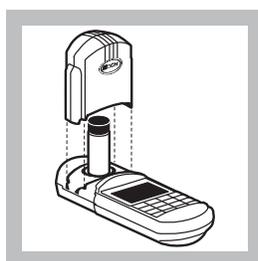
**PRGM ?**

*Note: For alternate forms (Ca) press the **CONC** key.*

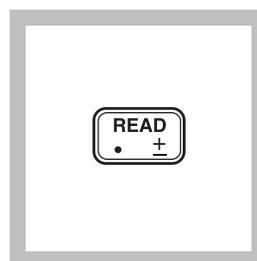


**14.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L CaCO<sub>3</sub>**



**15.** Place the third sample cell into the cell holder.



**16.** Press: **READ**  
The cursor will move to the right, then the result in mg/L calcium hardness (as CaCO<sub>3</sub>) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

*Note: mg/L total hardness = mg/L Ca as CaCO<sub>3</sub> + mg/L Mg as CaCO<sub>3</sub>.*

## Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months. Adjust the sample pH to

## HARDNESS, continued

---

between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Correct the test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

### Accuracy Check

Using a 2.00 mg/L (as CaCO<sub>3</sub>) standard solution as sample, perform the hardness procedure described above. The results should be 2.00 mg/L calcium (as CaCO<sub>3</sub>).

### Method Performance

#### Precision

In a single laboratory using a standard solution of 2.00 mg/L Mg as CaCO<sub>3</sub> and 1.88 mg/L Ca as CaCO<sub>3</sub> with the instrument, a single operator obtained a standard deviation of ± 0.09 mg/L Mg as CaCO<sub>3</sub> and ± 0.08 mg/L Ca as CaCO<sub>3</sub>.

#### Estimated Detection Limit

The estimated detection limit for program 30 is 0.13 mg/L magnesium hardness and 0.08 mg/L calcium hardness. For more information on the estimated detection limit, see *Section 1*.

### Interferences

For the most accurate hardness test result, the test should be rerun on a diluted sample if the calcium is over 1.0 or the magnesium is over 0.25 mg/L as CaCO<sub>3</sub>. No retesting is needed if either is below those respective concentrations.

The following cause a detectable error in test results.

Interfering Substance	Level at Which Substance Interferes
Cr <sup>3+</sup>	0.25 mg/L
Cu <sup>2+</sup>	0.75 mg/L
EDTA, chelated	0.2 mg/L as CaCO <sub>3</sub>
Fe <sup>2+</sup>	1.4 mg/L
Fe <sup>3+</sup>	2.0 mg/L
Mn <sup>2+</sup>	0.20 mg/L
Zn <sup>2+</sup>	0.050 mg/L

Traces of EDTA or EGTA remaining in sample cells from previous tests will give erroneous results. Rinse cells thoroughly before use.

## HARDNESS, continued

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### Summary of Method

The colorimetric method for measuring hardness supplements the conventional titrimetric method because it can measure very low levels of calcium and magnesium. Also some interfering metals (those listed above) in the titrimetric method are inconsequential in the colorimetric method when diluting the sample to bring it within the range of this test.

The indicator dye, calmagite, forms a purplish-blue color in a strongly alkaline solution and changes to red when it reacts with free calcium or magnesium. Calcium is chelated with EGTA to destroy any red color due to calcium and then the sample is chelated with EDTA to destroy the red color due to both calcium and magnesium. Measuring the red color in the different stages of chelation gives results as the calcium and magnesium hardness concentrations.

---

### REQUIRED REAGENTS

	Cat. No.
Hardness Reagent Set (100 Tests) .....	23199-00
Includes: (1) 22417-32, (1) 22418-32, (1) 22419-26, (1) 22297-26	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Alkali Solution for Calcium and Magnesium Test ....	1 mL.....	100 mL	MDB.....	22417-32
Calcium and Magnesium Indicator Solution .....	1 mL.....	100 mL	MDB.....	22418-32
EDTA Solution, 1 M.....	1 drop.....	50 mL.....		22419-26
EGTA Solution .....	1 drop.....	50 mL.....		22297-26

### REQUIRED APPARATUS

Cylinder, 100-mL mixing .....	1 .....	each.....	1896-42
Dropper, measuring, 0.5 and 1.0 mL .....	2 .....	20/pkg.....	21247-20
Sample Cell, 10-20-25 mL, w/cap .....	3 .....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

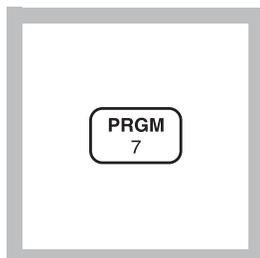
Calcium Standard Solution, 2.0 mg/L as CaCO <sub>3</sub> .....	946 mL.....	20581-16
Nitric Acid, ACS.....	500 mL.....	152-49
Nitric Acid Solution, 1:1 .....	500 mL.....	2540-49
Sodium Hydroxide Standard Solution 5.0 N .....	100 mL MDB.....	2450-32

### OPTIONAL APPARATUS

pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode .....	each.....	51700-10
Thermometer, -20 to 110 °C.....	each.....	26357-02

**HYDRAZINE (0 to 500 µg/L)**

For boiler water/feedwater, water and seawater

**p-Dimethylaminobenzaldehyde Method\*****Using Reagent Solution**

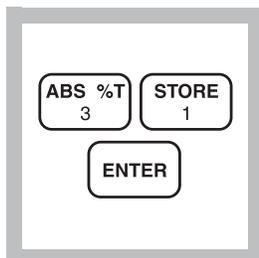
**1.** Enter the stored program number for hydrazine (N<sub>2</sub>H<sub>4</sub>).

Press: **PRGM**

The display will show:

**PRGM ?**

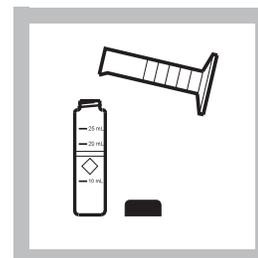
*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



**2.** Press: **31 ENTER**  
The display will show **µg/L, N2H4** and the **ZERO** icon.

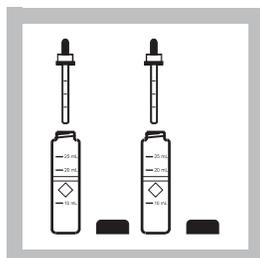


**3.** Pour 10.0 mL of deionized water into a sample cell (the blank) using a graduated cylinder.

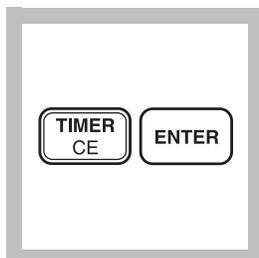


**4.** Pour 10.0 mL of sample into a second sample cell (the sample) using a graduated cylinder.

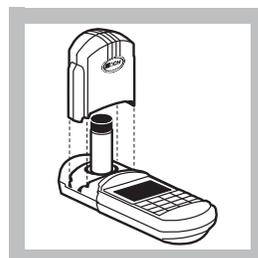
*Note: The sample temperature should be 21 ± 4 °C (70 ± 7 °F).*



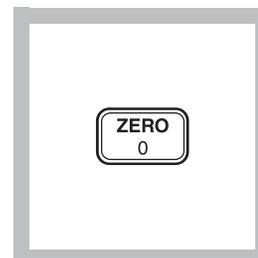
**5.** Add 0.5 mL of HydraVer 2 Hydrazine Reagent to each sample cell. Cap. Invert to mix.



**6.** Press: **TIMER ENTER**  
A 12-minute reaction period will begin.  
*Note: Complete Steps 7-9 within 3 minutes.*  
*Note: A yellow color will form if hydrazine is present. The blank will be a faint yellow color due to the HydraVer 2 reagent.*



**7.** Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

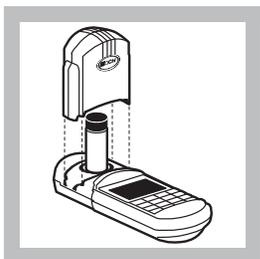


**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0 µg/L N2H4**

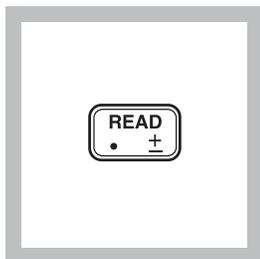
\* Adapted from ASTM Manual of Industrial Water, D1385-78, 376 (1979)

## HYDRAZINE, continued

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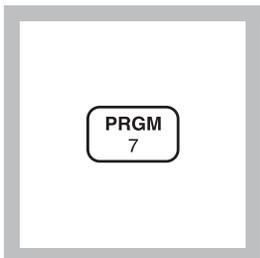


**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in  $\mu\text{g/L}$  hydrazine will be displayed.

### Using AccuVac Ampuls



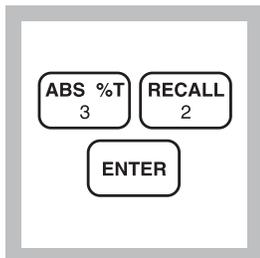
**1.** Enter the stored program number for hydrazine ( $\text{N}_2\text{H}_4$ )-AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

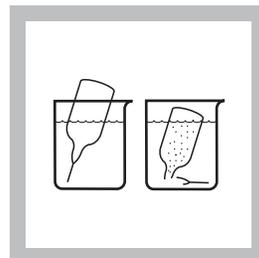
*Note: Samples must be analyzed immediately and cannot be preserved for later analysis.*



**2.** Press: **32 ENTER**  
The display will show  $\mu\text{g/L}$ , **N2H4** and the **ZERO** icon.



**3.** Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second 50-mL beaker.



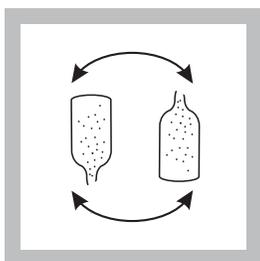
**4.** Fill a Hydrazine AccuVac Ampul with sample. Fill a second Hydrazine AccuVac Ampul with deionized water (the blank).

*Note: Keep the tip immersed while the ampul fills completely.*

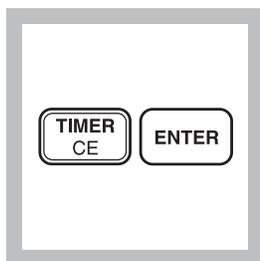
*Note: The sample temperature should be  $21 \pm 4$  °C ( $70 \pm 7$  °F).*

## HYDRAZINE, continued

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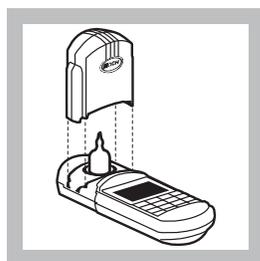
**5.** Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.



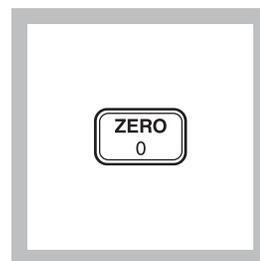
**6.** Press:  
**TIMER ENTER**  
A 12-minute reaction period will begin.

*Note: Complete Steps 7-9 during this period.*

*Note: A yellow color will develop if hydrazine is present. The blank will be a faint yellow color due to the reagent.*

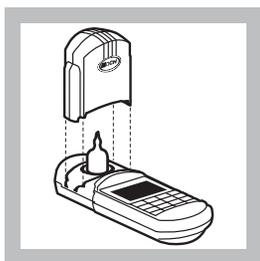


**7.** Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

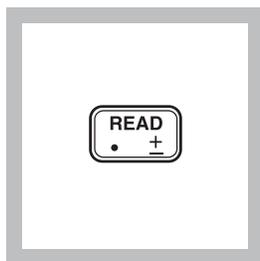


**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 µg/L N2H4**



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Immediately after the timer beeps, press **READ**.

The cursor will move to the right, then the result in µg/L hydrazine will be displayed.

---

### Sampling and Storage

Collect samples in glass or plastic containers. Fill the containers completely and cap them tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

## HYDRAZINE, continued

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### Accuracy Check

#### Standard Solution Method

To assure the accuracy of the test, prepare the following solutions:

- a) Prepare a 25 mg/L hydrazine stock solution by dissolving 0.1016 g of hydrazine sulfate in 1000 mL of oxygen-free deionized water. Use Class A glassware. Prepare this stock solution daily.
- b) Prepare a 100 µg/L hydrazine working solution by diluting 4.00 mL of the 25 mg/L stock solution to 1000 mL with deionized oxygen-free water. Prepare just before analysis.
- c) Use the working solution in place of the sample in Step 4. The result should be 100 µg/L hydrazine.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 250 µg/L hydrazine (N<sub>2</sub>H<sub>4</sub>) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±9 µg/L hydrazine.

In a single laboratory using a standard solution of 250 µg/L hydrazine (N<sub>2</sub>H<sub>4</sub>) and two lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±3 µg/L hydrazine.

#### Estimated Detection Limit

The estimated detection limit for program 31 is 16 µg/L N<sub>2</sub>H<sub>4</sub>, and the estimated detection limit for program 32 is 10 µg/L N<sub>2</sub>H<sub>4</sub>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

For highly colored or turbid samples, prepare a blank by oxidizing the hydrazine in a portion of the sample. This can be accomplished with a 1:1 mixture of deionized water and household bleach. Add two drops of this mixture to 40 mL of sample contained in a graduated mixing cylinder and invert to mix. Use this solution in Step 3, in place of deionized water, to prepare the blank.

Ammonia has no effects up to 10 mg/L ammonia. At 20 mg/L, a

## HYDRAZINE, continued

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positive interference occurs.

Morpholine does not interfere up to 10 mg/L.

### Summary of Method

Hydrazine reacts with the p-dimethylaminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration.

---

### REQUIRED REAGENTS (Using Reagent Solution)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
HydraVer 2 Hydrazine Reagent .....	1 mL	.....100 mL	MDB	.....1790-32
Water, deionized .....	10 mL	.....	4 L	.....272-56

### REQUIRED APPARATUS (Using Reagent Solution)

Cylinder, graduated, 25 mL .....	1	.....each	.....508-40
Sample Cells, 10-, 20- and 25 mL, w/ caps.....	2	.....6/pkg	.....24019-06

### REQUIRED REAGENTS (Using AccuVac Ampuls)

Hydrazine Reagent AccuVac Ampul.....	2	.....25/pkg	.....25240-25	
Water, deionized .....	10 mL	.....	4 L	.....272-56

### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....	2	.....each	.....500-41H
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### OPTIONAL REAGENTS

Hydrazine Sulfate, ACS .....	100 g	.....742-26
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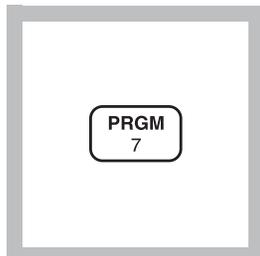
### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each	.....24052-00
Balance, Analytical, 115 V, 0.1 mg .....	each	.....28014-01
Balance, Analytical, 220 V, 0.1 mg .....	each	.....28014-02
Cylinder, graduated, mixing, 25 mL .....	each	.....1896-40
Flask, volumetric, 100 mL, Class A.....	each	.....14574-42
Flask, volumetric, 1000 mL, Class A.....	each	.....14574-53
Pipet, serological, 1 mL.....	each	.....9190-02
Pipet, TenSette, 0.1 to 1.0 mL .....	each	.....19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	.....21856-96
Pipet, volumetric, Class A, 1.00 mL .....	each	.....14515-35
Pipet, volumetric, Class A, 4.00 mL .....	each	.....14515-04
Pipet Filler, safety bulb .....	each	.....14651-00
Thermometer, -20 to 110 °C, non-mercury .....	each	.....26357-02
Weighing Boat, 67/46 mm, 8.9 cm sq.....	500/pkg	.....21790-00



**IRON, FERROUS (0 to 3.00 mg/L)**

For water, wastewater, and seawater

**1,10 Phenanthroline Method\*** (Powder Pillows or AccuVac Ampuls)**Using Powder Pillows**

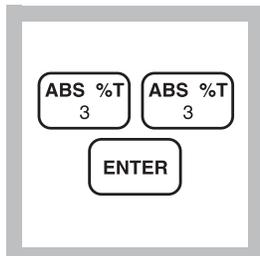
1. Enter the stored program number for Ferrous iron ( $\text{Fe}^{2+}$ )-powder pillows.

Press: **PRGM**

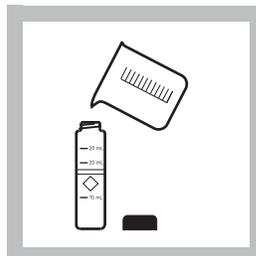
The display will show:

**PRGM ?**

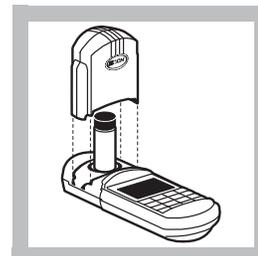
*Note: Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined.*



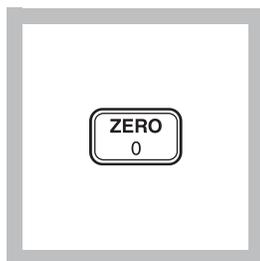
2. Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.



3. Fill a sample cell with 25 mL of sample (the blank).

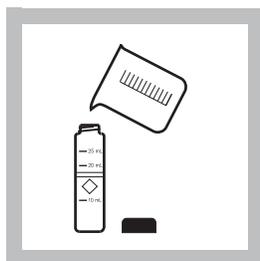


4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

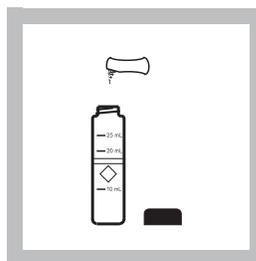


5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**

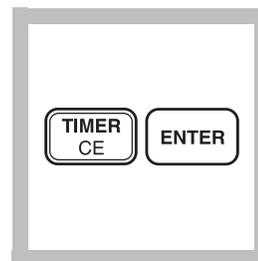


6. Fill another sample cell with 25 mL of sample.



7. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

*Note: Undissolved powder does not affect accuracy.*



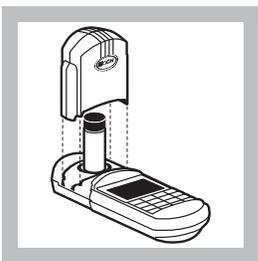
8. Press: **TIMER ENTER**  
A three-minute reaction period will begin.

*Note: An orange color will form if ferrous iron is present.*

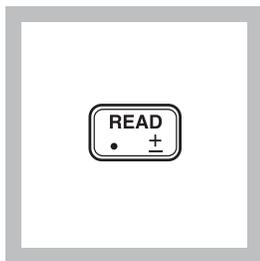
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## IRON, FERROUS, continued

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**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

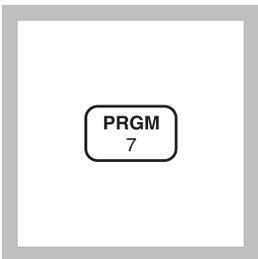


**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

### Using AccuVac Ampuls



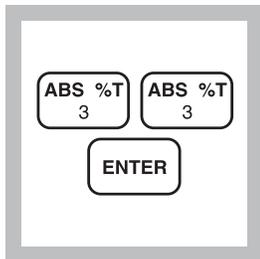
**1.** Enter the stored program number for ferrous iron ( $\text{Fe}^{2+}$ ) AccuVac ampuls.

Press: **PRGM**

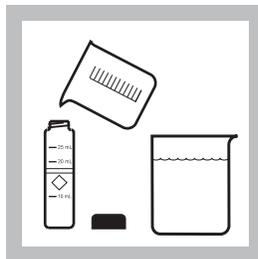
The display will show:

**PRGM ?**

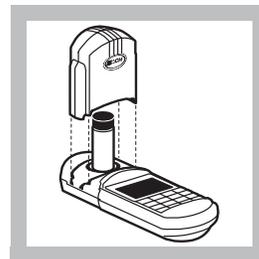
*Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.*



**2.** Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.

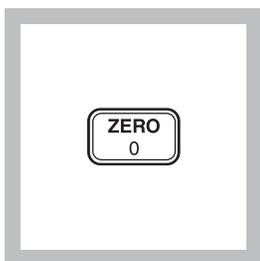


**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



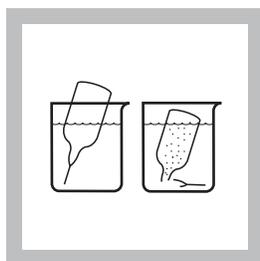
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## IRON, FERROUS, continued



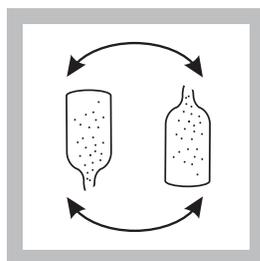
**5. Press: ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**



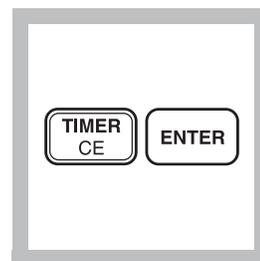
**6. Fill a Ferrous Iron AccuVac Ampul with sample.**

*Note: Keep the tip immersed while the ampul fills completely.*



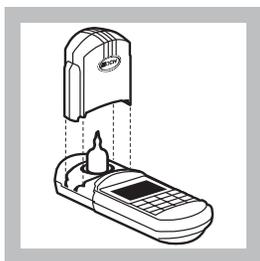
**7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.**

*Note: Undissolved powder does not affect accuracy.*

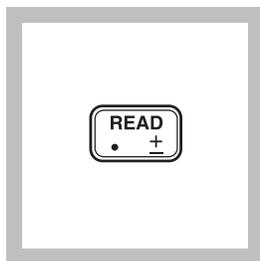


**8. Press: TIMER ENTER**  
A three-minute reaction period will begin.

*Note: An orange color will form if ferrous iron is present.*



**9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.**



**10. Press: READ**  
The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

## IRON, FERROUS, continued

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### Sampling and Storage

Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

### Accuracy Check

#### Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L Fe<sup>2+</sup>) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.00 mg/L standard solution. Prepare immediately before use.

Run the test using the 1.00 mg/L Fe<sup>2+</sup> Standard Solution by following either the powder pillow or AccuVac procedure. Results should be between 0.90 mg/L and 1.10 mg/L Fe<sup>2+</sup>.

### Method Performance

#### Precision

In a single laboratory using an iron standard solution of 2.00 mg/L Fe<sup>2+</sup> and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L Fe<sup>2+</sup>.

In a single laboratory using a standard solution of 2.00 mg/L Fe<sup>2+</sup> and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L Fe<sup>2+</sup>.

#### Estimated Detection Limit

The estimated detection limit for program 33 (powder pillows and AccuVac Ampuls) is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe<sup>3+</sup>) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

## IRON, FERROUS, continued

---

### REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Quantity Required		Cat. No.
	Per Test	Units	
Ferrous Iron Reagent Powder Pillows.....	1 pillow.....	100/pkg .....	1037-69
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06

### REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

Ferrous Iron Reagent AccuVac Ampuls.....	1 ampul.....	25/pkg .....	25140-25
Beaker, 50 mL .....	1 .....	each .....	500-41H

### OPTIONAL REAGENTS

Ferrous Ammonium Sulfate, hexahydrate, ACS.....	113 g .....	11256-14
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each .....	24052-00
Balance, analytical, 115 V, 0.1 mg .....	each .....	28014-01
Balance, analytical, 230 V, 0.1 mg .....	each .....	28014-02
Clippers, for opening powder pillows .....	each .....	968-00
Flask, volumetric, 100 mL, Class A.....	each .....	14574-42
Flask, volumetric, 1000 mL, Class A.....	each .....	14574-53
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00
Weighing Boat, 67/46 mm, 8.9 cm square .....	500/pkg .....	21790-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

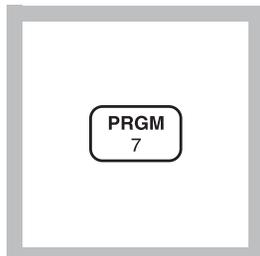


**IRON, TOTAL (0 to 3.00 mg/L)**

For water, wastewater, and seawater

**FerroVer Method (Powder Pillows or AccuVac Ampuls)**

USEPA approved for reporting wastewater analysis (digestion is required; see Section 2\*)



1. Enter the stored program number for iron (Fe) powder pillows.

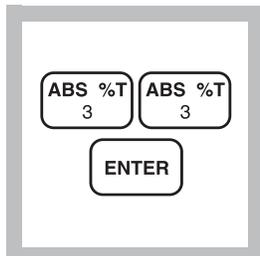
Press: **PRGM**

The display will show:

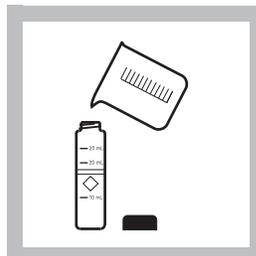
**PRGM ?**

*Note: Determination of total iron requires a digestion prior to analysis (see Section 2).*

*Note: Adjust pH of stored samples before analysis.*

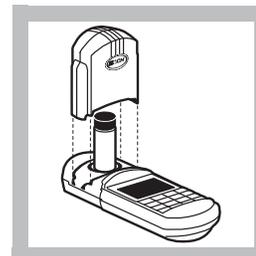


2. Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.

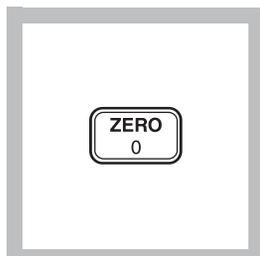


3. Fill a clean sample cell with 10 mL of sample (the blank).

*Note: For turbid samples, treat the blank with one 0.1-gram scoop of RoVer Rust Remover. Swirl to mix.*

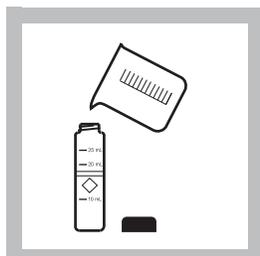


4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**

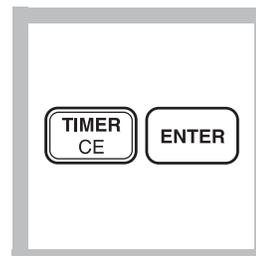


6. Fill another sample cell with 10 mL of sample.



7. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to dissolve the reagent powder.

*Note: Accuracy is not affected by undissolved powder.*



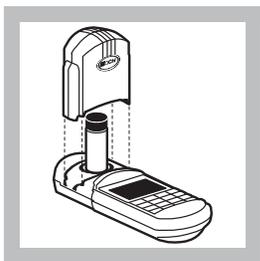
8. Press: **TIMER ENTER**  
A three-minute reaction period will begin.

*Note: An orange color will form if iron is present.*

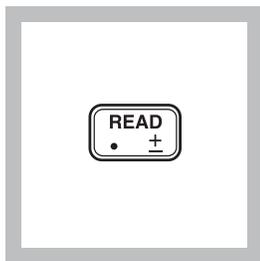
*Note: Samples containing visible rust should be allowed to react at least five minutes.*

\* Federal Register, 45 (126) 43459 (June 27, 1980). See also 40 CFR, part 136.3, Table IB.

## IRON, TOTAL, continued



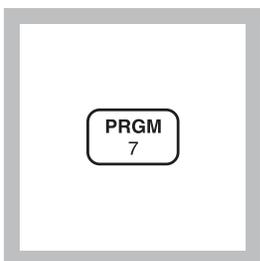
**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

### Using AccuVac Ampuls



**1.** Enter the stored program number for iron (Fe), AccuVac ampuls.

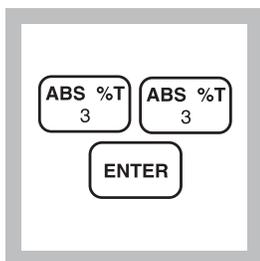
Press: **PRGM**

The display will show:

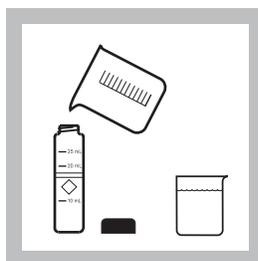
**PRGM ?**

*Note:* Adjust pH of stored samples before analysis.

*Note:* Determination of total iron requires a digestion prior to analysis (see Section 2).

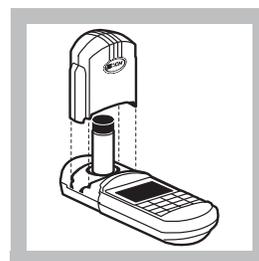


**2.** Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.



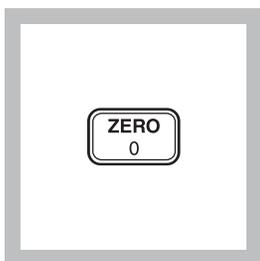
**3.** Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

*Note:* For turbid samples, treat the blank with one 0.1 g scoop of RoVer Rust Remover. Swirl to mix.



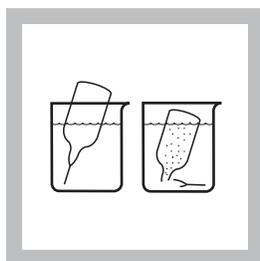
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## IRON, TOTAL, continued

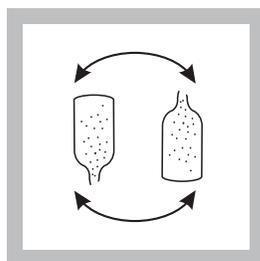


- 5. Press: ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**



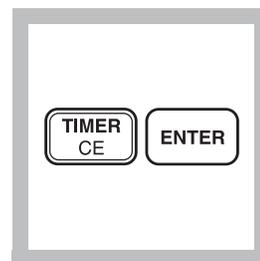
- 6. Fill a FerroVer AccuVac Ampul with sample.**  
*Note: Keep the tip immersed while the ampul fills completely.*



- 7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.**

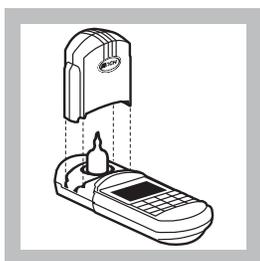
*Note: An orange color will form if iron is present.*

*Note: Accuracy is not affected by undissolved powder.*

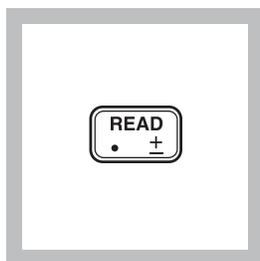


- 8. Press: TIMER ENTER**  
A three-minute reaction period will begin.

*Note: Samples containing visible rust should be allowed to react at least five minutes.*



- 9. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.**



- 10. Press: READ**  
The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

## IRON, TOTAL, continued

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### Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information. If only dissolved iron is to be determined, filter the sample before adding the acid.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a 50 mg/L Iron PourRite Ampule Standard Solution.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples and mix thoroughly.
- c) For analysis using AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers to facilitate filling of the ampuls. For analysis with powder pillows, transfer only 10 mL of solution to the 10-mL sample cells.
- d) Analyze each standard addition sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

#### Standard Solution Method

Prepare a 1.0-mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, dilute 1.00 mL of an Iron PourRite Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

Run the test following the procedure for powder pillows or AccuVac Ampuls. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

## IRON, TOTAL, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L.

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L Fe.

#### Estimated Detection Limit (EDL)

The EDL for program 33 is 0.03 mg/L Fe. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

#### Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Calcium, Ca <sup>2+</sup>	No effect at less than 10,000 mg/L as CaCO <sub>3</sub>
Chloride, Cl <sup>-</sup>	No effect at less than 185,000 mg/L.
Copper, Cu <sup>2+</sup>	No effect. Masking agent is contained in FerroVer Iron Reagent.
High Iron Levels	Inhibits color development. Dilute sample and retest to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion (see Section 2). After digestion, adjust sample to pH 3-5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as CaCO <sub>3</sub> .
Molybdate, Molybdenum	No effect at 25 mg/L as Mo.
High Sulfide Levels, S <sup>2-</sup>	<ol style="list-style-type: none"><li>1. Treat in fume hood or well-ventilated area. Add 5 mL HCl to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes.</li><li>2. Cool. Adjust pH to 3-5 with NaOH. Re-adjust volume to 100 mL with deionized water.</li><li>3. Analyze.</li></ol>

## IRON, TOTAL, continued

Interfering Substance	Interference Level and Treatment
Turbidity	<ol style="list-style-type: none"> <li>1. Add 0.1 g scoop of RoVer Rust Remover to the blank in Step 3. Swirl to mix.</li> <li>2. Zero the instrument with this blank.</li> <li>3. If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes.</li> <li>4. Filter through a glass filter or centrifuge.</li> <li>5. Use filtered sample in Steps 3 and 6.</li> </ol>
Sample pH (extreme)	Adjust pH to 3-5. See <i>Interferences</i> in Section 1.
Highly Buffered Samples	Adjust pH to 3-5. See <i>Interferences</i> in Section 1.

### Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. This reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat No.
FerroVer Iron Reagent Powder Pillows .....	1 pillow .....	100/pkg.....	21057-69
Sample cell, 10-20-25 mL, with screw cap .....	1 .....	6/pkg.....	24019-06

### REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls .....	1 ampul .....	25/pkg.....	25070-25
Beaker, 50 mL.....	1 .....	each.....	500-41H

### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Ammonium Hydroxide, ACS .....	500 mL.....	106-49
Drinking Water Standard, Metals, LR (Cu, Fe, Mn) .....	500 mL.....	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn) .....	500 mL.....	28336-49
Hydrochloric Acid Standard Solution, 6 N.....	500 mL.....	884-49
Hydrochloric Acid, ACS.....	500 mL.....	134-49
Iron Standard Solution, 100 mg/L .....	100 mL.....	14175-42
Iron Ampule Standard, 50 mg/L .....	20/pkg.....	14254-20
Nitric Acid, ACS.....	500 mL.....	152-49
Nitric Acid Solution, 1:1 .....	500 mL.....	2540-49
RoVer Rust Remover .....	454 g.....	300-01
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB.....	2450-32
Water, deionized.....	4 L.....	272-56

## IRON, TOTAL, continued

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### OPTIONAL APPARATUS

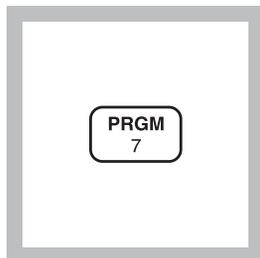
AccuVac Snapper Kit .....	each .....	24052-00
Ampule Breaker, PourRite Ampules .....	each .....	24846-00
Clippers, Shears 7 <sup>1</sup> / <sub>4</sub> " .....	each .....	23694-00
Cylinder, graduated, poly, 25 mL .....	each .....	1081-40
Cylinder, graduated, poly, 100 mL .....	each .....	1081-42
Digesdahl Digestion Apparatus, 115 V .....	each .....	23130-20
Digesdahl Digestion Apparatus, 230 V .....	each .....	23130-21
Filter Discs, glass, 47 mm .....	100/pkg .....	2530-00
Filter Holder, membrane .....	each .....	2340-00
Filter Pump .....	each .....	2131-00
Flask, Erlenmeyer, 250 mL .....	each .....	505-46
Flask, filtering, 500 mL .....	each .....	546-49
Flask, volumetric, Class A, 50 mL .....	each .....	14574-41
Flask, volumetric, Class A, 100 mL .....	each .....	14574-42
Hot Plate, 4" diameter, 120 VAC .....	each .....	12067-01
Hot Plate, 4" diameter, 240 VAC .....	each .....	12067-02
pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> 1, portable, with electrode .....	each .....	51700-10
pH Indicator Paper, 1 to 11 pH .....	each .....	391-33
Pipet Filler, safety bulb .....	each .....	14651-00
Pipet, serological, 2 mL .....	each .....	532-36
Pipet, serological, 5 mL .....	each .....	532-37
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Spoon, measuring, 0.1 g .....	each .....	511-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**IRON (0 to 1.300 mg/L)****FerroZine Method\***

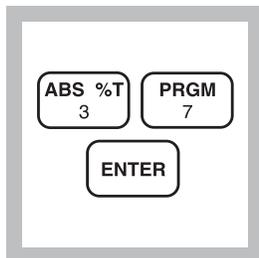
**1.** Enter the stored program number for iron (Fe).

Press: **PRGM**

The display will show:

**PRGM ?**

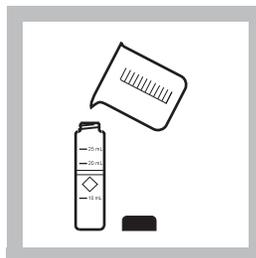
*Note:* Adjust the pH of stored samples before analysis.



**2.** Press: **37 ENTER**

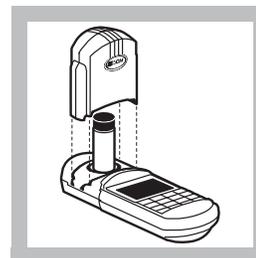
The display will show **mg/L, Fe** and the **ZERO** icon.

*Note:* Total iron determinations need a prior digestion; use any of the three procedures given in Digestion (Section 2).

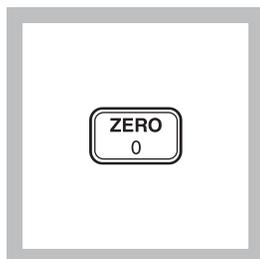


**3.** Fill a sample cell with 25-mL of sample (the blank).

*Note:* Rinse glassware with a 1:1 Hydrochloric Acid Solution and deionized water before use to avoid errors due to iron deposits on the glass.



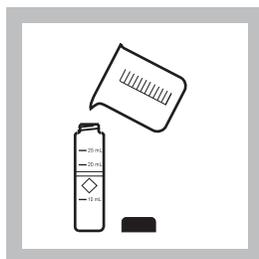
**4.** Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**5.** Press: **ZERO**

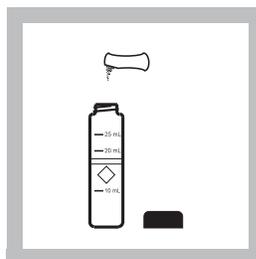
The cursor will move to the right, then the display will show:

**0.000 mg/L Fe**



**6.** Fill another sample cell with 25 mL of sample.

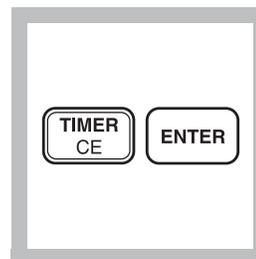
*Note:* If the sample contains rust, see Interferences below.



**7.** Add the contents of one FerroZine Iron Reagent Solution Pillow to the cell (the prepared sample). Cap and invert to mix.

*Note:* Do not allow the clippers to come into contact with the contents of the pillow.

*Note:* If preferred, use 0.5 mL of FerroZine Iron Reagent Solution in place of the solution pillow.



**8.** Press:

**TIMER ENTER**

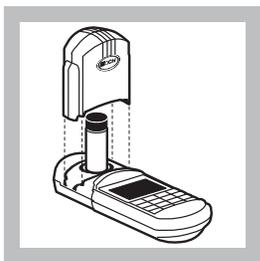
A five-minute reaction period will begin.

*Note:* A violet color will develop if iron is present.

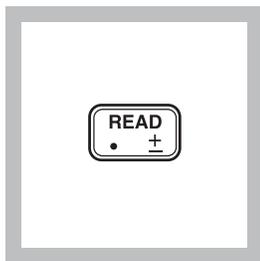
\* Adapted from Stookey, L.L., Anal. Chem., 42 (7) 779 (1970)

## IRON, continued

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**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection and before the addition of nitric acid.

Before testing, adjust the sample pH to 3–5 with ammonium hydroxide, ACS. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see *Correction for Volume Additions* in Section 1 for more detailed information.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off an Iron Voluette Ampule Standard, 25 mg/L Fe.
- b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10.
- c) Swirl to mix and allow another five-minute reaction period, then measure the iron concentration as in Step 10.
- d) Add two additional 0.1-mL standard increments, taking a

## IRON, continued

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concentration reading after allowing the five-minute reaction period for each increment.

- e) Each 0.1 mL of standard added should cause a 0.1 mg/L increase in the concentration reading.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. This solution should be prepared daily. Analyze the working solution according to the above procedure.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.80 mg/L iron and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.004$  mg/L iron.

#### Estimated Detection Limit

The estimated detection limit for program 37 is 0.011 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

## IRON, continued

### Interferences

Interfering Substance	Interference Levels and Treatments
Strong chelants, EDTA	Interfere at all levels. Use the FerroVer or TPTZ methods to test these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results
Copper	May give slightly high results
Hydroxides	Boil the sample, with the FerroZine Iron Reagent from Step 7 added to it for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the sample volume to 25 mL with deionized water. OR Use any of the digestions in <i>Section 2</i> .
Magnetite (black iron oxide) or Ferrites	<ol style="list-style-type: none"><li>1. Fill a 25-mL graduated cylinder with 25 mL of sample.</li><li>2. Transfer this sample into a 125-mL erlenmeyer flask.</li><li>3. Add the contents of one FerroZine Iron Reagent Solution Pillow and swirl to mix.</li><li>4. Place the flask on a hot plate or over a flame and bring to a boil.</li><li>5. Continue boiling gently for 20 to 30 minutes. <i>Note: Do not allow to boil dry.</i> <i>Note: A purple color will develop if iron is present.</i></li><li>6. Return the boiled sample to the 25-mL graduated cylinder. Rinse the erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder.</li><li>7. Return the sample volume to the 25-mL mark with deionized water.</li><li>8. Pour this solution into a sample cell. Swirl to mix.</li><li>9. Proceed with Step 9.</li></ol> OR Use any of the digestions in <i>Section 2</i> .
Rust	Boil the sample, with the FerroZine Iron Reagent from Step 7 for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the volume to 25 mL with deionized water. OR Use any of the digestions in <i>Section 2</i> .

### Summary of Method

The FerroZine Iron Reagent forms a purple colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and can be used to analyze samples containing magnetite (black iron oxide) or ferrites after treatment as described in Interferences.

## IRON, continued

### REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
FerroZine Iron Reagent Solution Pillows.....	1 pillow.....	50/pkg.....	2301-66	
Clippers, for opening pillows.....	1.....	each.....	968-00	
Sample Cell, 10-20-25, w/cap.....	2.....	6/pkg.....	24019-06	

### OPTIONAL REAGENTS

Ammonium Hydroxide, ACS.....	500 mL	106-49	Drinking
Water Standard, Metals, LR (Cu, Fe, Mn).....	500 mL.....	28337-49	
Hydrochloric Acid Solution, 1:1 (6N).....	500 mL.....	884-49	
FerroZine Iron Reagent Solution.....	500 mL.....	2301-49	
Iron Standard Solution, 100 mg/L Fe.....	100 mL.....	14175-42	
Iron Standard Solution, Voluette Ampule, 25 mg/L Fe, 10 mL.....	16/pkg.....	14253-10	
Nitric Acid, ACS.....	500 mL.....	152-49	
Nitric Acid Solution, 1:1.....	500 mL.....	2540-49	
Water, deionized.....	4 L.....	272-56	

### OPTIONAL APPARATUS

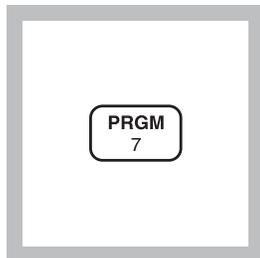
Ampule Breaker Kit.....	each.....	21968-00	
Clippers, shears, 7¼-inch.....	each.....	20658-00	
Cylinder, graduated, 25 mL.....	each.....	508-40	
Dropper, calibrated, 0.5-mL & 1.0-mL mark.....	6/pkg.....	23185-06	
Flask, erlenmeyer, 125 mL.....	each.....	505-43	
Flask, volumetric, 250 mL, Class A.....	each.....	14574-46	
Hot plate, 3 ½" diameter, 120 V.....	each.....	12067-01	
Hot plate, 3 ½" diameter, 240 V.....	each.....	12067-02	
pH Indicator Paper, 1 to 11 pH.....	5 rolls/pkg.....	391-33	
Pipet, serological, 2 mL.....	each.....	532-36	
pH Meter, <i>sensio</i> <sup>TM</sup> <i>1</i> , portable, with electrode.....	each.....	51700-10	
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01	
Pipet Tips, for 19700-01 TenSette Pipet.....	50/pkg.....	21856-96	Pipet
Tips, for 19700-01 TenSette Pipet.....	1000/pkg.....	21856-28	
Pipet, volumetric, 1.00 mL, Class A.....	each.....	14515-35	
Thermometer, -20 to 110 °C, non-mercury.....	each.....	26357-02	
Water Bath, with sample cell rack.....	each.....	1955-55	

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**IRON, TOTAL (0 to 1.80 mg/L) For cooling water with molybdenum-based treatment****FerroMo™ Method\***

**1.** Enter the stored program number for iron (Fe).

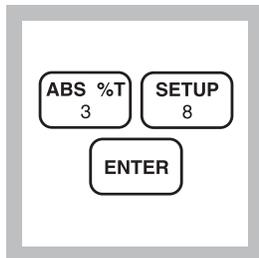
Press: **PRGM**

The display will show:

**PRGM ?**

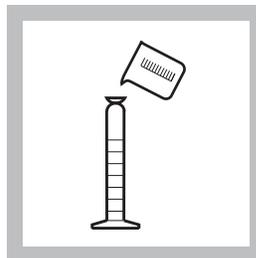
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

*Note:* Adjust the pH of stored samples before analysis.



**2.** Press: **38 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.

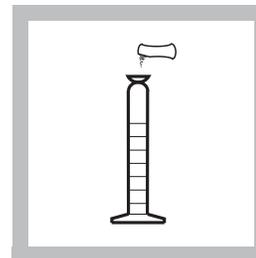
*Note:* Determination of total iron requires digestion; see Section 2.



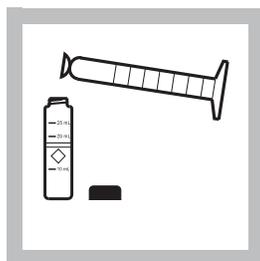
**3.** Fill a 50-mL graduated mixing cylinder with 50 mL of sample.

*Note:* Sample pH is important in the test; see Interferences.

*Note:* Rinse glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. This removes iron deposits which can cause slightly high results.



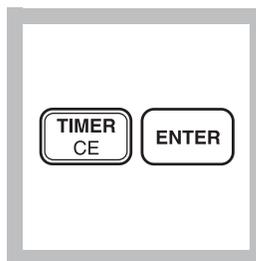
**4.** Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the graduated cylinder. Stopper and invert several times to mix. Remove the stopper. This is the prepared sample.



**5.** Transfer 25 mL of the prepared sample to a sample cell.

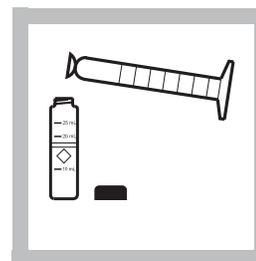


**6.** Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the sample cell. Cap the cell and shake for 30 seconds. This is the prepared sample.



**7.** Press:  
**TIMER ENTER**  
A three-minute reaction period will begin.

*Note:* A blue color will develop if iron is present.

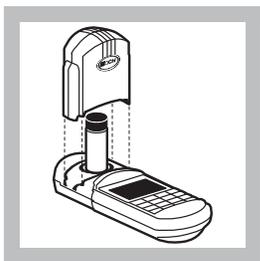


**8.** Fill a second sample cell with 25 mL of the prepared sample from Step 4 (the blank).

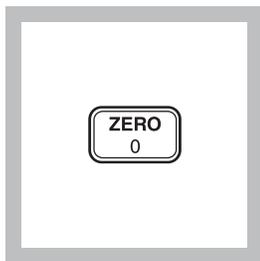
\* Adapted from G. Frederic Smith Chemical Company, *The Iron Reagents*, 3rd ed. (1980).

## IRON, TOTAL, continued

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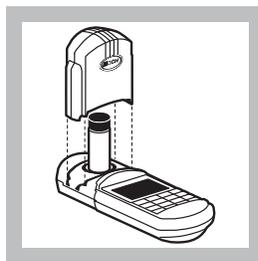
**9.** Insert the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

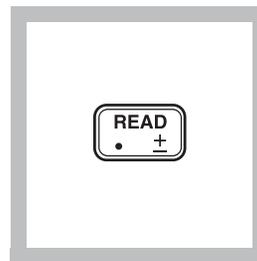
**0.00 mg/L Fe**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**11.** After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: For samples containing high levels of molybdate ( $\geq 100$  mg/L), read the sample immediately after zeroing the blank.*



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with hydrochloric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. If reporting only dissolved iron, filter the sample immediately after collection and before adding the acid.

Before analysis, adjust the sample pH to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume; see *Correction for Volume Additions* in Section 1.

### Accuracy Check

#### Standard Additions Method

- Snap the top off an Iron PourRite Ampule Standard Solution, 25 mg/L Fe.
- Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 50-mL samples. Swirl gently to mix.
- Analyze each sample as described above. The iron concentration should increase by 0.1 mg/L for each 0.2

## IRON, TOTAL, continued

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mL of standard added.

- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more Information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. Prepare this solution daily. Analyze this working solution according to the above procedure. Results should be between 0.36 and 0.44 mg/L Fe.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.00 mg/L Fe and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.006$  mg/L Fe.

#### Estimated Detection Limit

The estimated detection limit for program 38 is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Interferences

A sample pH of less than 3 or greater than 4 after reagent addition may inhibit color formation, cause the developed color to fade, or result in turbidity. Adjust the sample pH before reagent addition to between 3 and 5 using a pH meter or pH paper. Drop by drop, add an appropriate amount of acid (1.0 N Sulfuric Acid Solution) or base (1.0 N Sodium Hydroxide Standard Solution). Make volume corrections if significant amounts of acid or base are used (see *Correction for Volume Additions* in *Section 1*).

### Summary of Method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron (rust) to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering

## IRON, TOTAL, continued

agent. The indicator reacts with the ferrous iron in the sample, buffered between pH 3-4, resulting in a deep blue-purple color.

### REQUIRED REAGENTS

	Cat. No.
FerroMo Reagent Set (100 tests) .....	25448-00
Includes: (4) 25437-68, (2) 25438-66	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
FerroMo Iron Reagent 1 Powder Pillows .....	1 pillow .....	25/pkg .....	25437-68
FerroMo Iron Reagent 2 Powder Pillows .....	1 pillow .....	50/pkg .....	25438-66

### REQUIRED APPARATUS

Clippers, for opening powder pillows.....	1 .....	each.....	968-00
Cylinder, graduated, mixing, 50 mL.....	1 .....	each.....	1896-41
Sample Cell, 10-20-25 mL, w/cap.....	2 .....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

Hydrochloric Acid Solution, 6.0 N (1:1).....	500 mL.....	884-49
Hydrochloric Acid, ACS.....	500 mL.....	134-49
Iron Standard Solution, 100 mg/L Fe .....	100 mL.....	14175-42
Iron Standard Solution, PourRite Ampule, 25 mg/L Fe, 2 mL .....	20/pkg.....	24629-20
Sodium Hydroxide Standard Solution, 1.0 N .....	100 mL MDB.....	1045-32
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB.....	2450-32
Sulfuric Acid Standard Solution, 1.0 N .....	100 mL MDB.....	1270-32
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....	24846-00
Flask, volumetric, Class A, 250 mL .....	each.....	14574-46
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>Sension</i> <sup>TM</sup> I, portable, with electrode .....	each.....	51700-10
Pipet Filler, safety bulb .....	each.....	14651-00
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 Pipet .....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 1.00 mL.....	each.....	14515-35

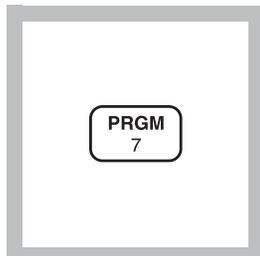
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**IRON, TOTAL (0 to 1.80 mg/L)**

For water, wastewater, and seawater

**TPTZ Method\* (Powder Pillows or AccuVac Ampuls)****Using Powder Pillows**

**1.** Enter the stored program number for iron (Fe)- powder pillows.

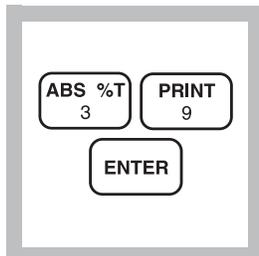
Press: **PRGM**

The display will show:

**PRGM ?**

*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

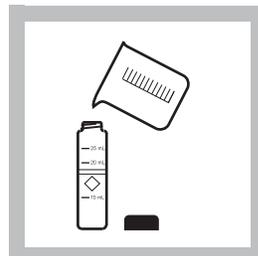
*Note:* Adjust the pH of stored samples before analysis.



**2.** Press: **39 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon.

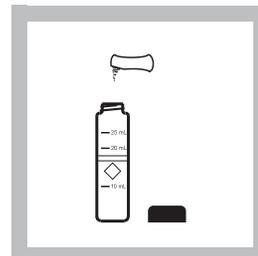
*Note:* Total iron determination needs a prior digestion. Use any of the procedures in Digestion (Section 2).



**3.** Fill a sample cell with 10 mL of sample.

*Note:* Sample pH is important in this test; see Interferences.

*Note:* Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.

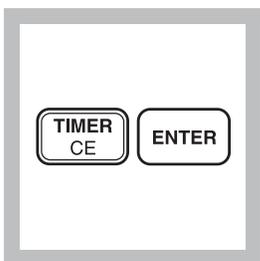


**4.** Add the contents of one TPTZ Iron Reagent Powder Pillow (the prepared sample). Cap and shake the cell for 30 seconds.

*Note:* A blue color will develop if iron is present.

\* Adapted from G. Frederic Smith Chemical Co., *The Iron Reagents*, 3rd ed. (1980).

## IRON, TOTAL, continued



5. Press:

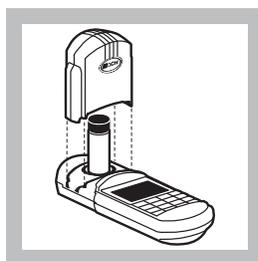
**TIMER ENTER**

A three-minute reaction period will begin.

*Note: Continue with Steps 6 to 8 while the timer is running.*

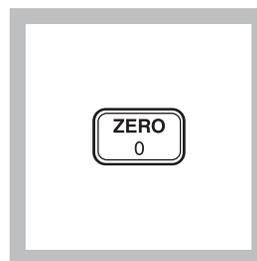


6. Fill a second sample cell with 10 mL of sample (the blank).



7. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: Press EXIT to zero the instrument while the timer is running.*

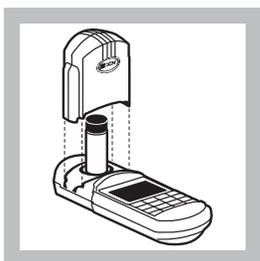


Press: **ZERO**

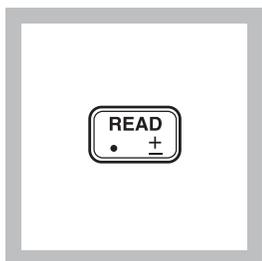
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**

If Reagent Blank Correction is on, the display may flash “limit”. See Section 1.



8. After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.



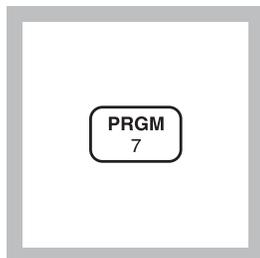
9. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

## IRON, TOTAL, continued

### Using AccuVac Ampuls



**1.** Enter the stored program number for iron (Fe)- AccuVac Ampuls.

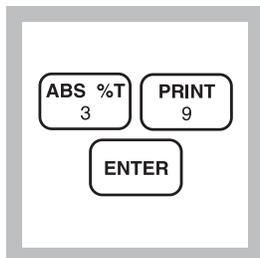
Press: **PRGM**

The display will show:

**PRGM ?**

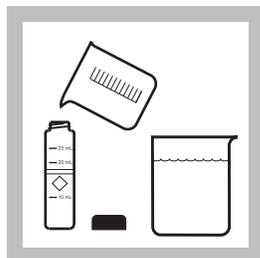
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

*Note:* Adjust the pH of stored samples before analysis.



**2.** Press: **39 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.

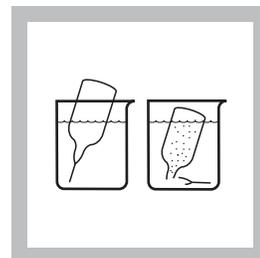
*Note:* Total iron determination needs a prior digestion. Use any of the three procedures in Digestion (Section 2).



**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

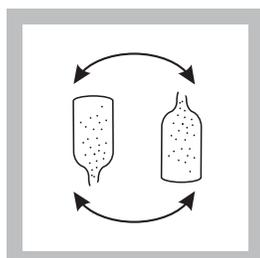
*Note:* Sample pH is important in this test; see Interferences.

*Note:* Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.



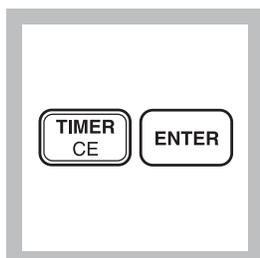
**4.** Fill a TPTZ Iron AccuVac Ampul with sample.

*Note:* Keep the tip immersed while the ampul fills completely.

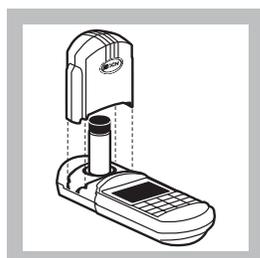


**5.** Invert the ampul (the prepared sample) repeatedly to mix. Wipe off any liquid or fingerprints.

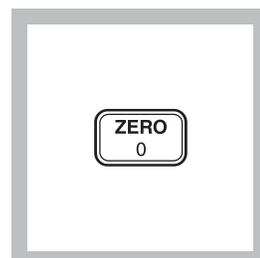
*Note:* A blue color will develop if iron is present.



**6.** Press: **TIMER ENTER**  
A three-minute reaction period will begin.



**7.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

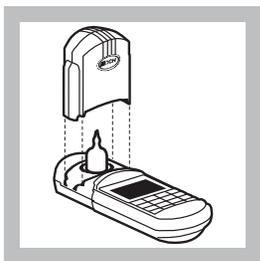
**0.00 mg/L Fe**

*Note:* Press **EXIT** to zero the instrument while the timer is running.

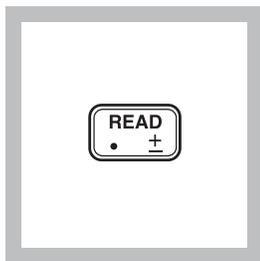
*Note:* If Reagent Blank Correction is on, the display may flash "limit". See Section 1.

## IRON, TOTAL, continued

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**9.** When the timer beeps, place the prepared sample into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Store samples preserved in this manner up to six months at room temperature. If reporting only dissolved iron, filter sample immediately after collection and before addition of nitric acid.

Before testing, adjust the pH of the stored sample to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1.

### Accuracy Check

#### Standard Additions Method (Powder Pillows)

- a) Snap the neck off a PourRite Iron Ampule Standard, 25 mg/L Fe.
- b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10. Swirl to mix.
- c) Measure the iron concentration as in Step 10. The measurement does not require the three-minute waiting period.

## IRON, TOTAL, continued

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- d) Add two additional 0.1-mL aliquots of standard, measuring the concentration after each addition. The iron concentration should increase by 0.25 mg/L for each 0.1-mL addition of standard.
- e) If these increases do not occur, see *Standard Additions in Section 1* for more information.

### Standard Additions Method (AccuVac Ampuls)

- a) Use a graduated cylinder to measure 25.0 mL of sample into each of three 50-mL beakers.
- b) Snap the neck off an Iron Ampule Standard, 25 mg/L Fe.
- c) Using a TenSette Pipet, add 0.1, 0.2 and 0.3 mL of standard, respectively, to the 50-mL beakers. Swirl to mix.
- d) Fill a TPTZ AccuVac Ampul from each beaker.
- e) Measure the concentration of each ampul according to the procedure. The iron concentration should increase by 0.1 mg/L for each 0.1 mL addition of standard.
- f) If these increases do not occur, see *Standard Additions in Section 1* for more information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Using Class A glassware, pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. Stopper and invert repeatedly to mix. Prepare this solution daily.

## Method Performance

### Precision

In a single laboratory using a standard solution of 1.00 mg/L Fe and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L Fe.

In a single laboratory using a standard solution of 1.00 mg/L Fe and one representative lot of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.022$  mg/L Fe.

## IRON, TOTAL, continued

### Estimated Detection Limit

The estimated detection limit for program 39 is 0.04 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Cadmium	Greater than 4.0 mg/L
Chromium ( <sup>3+</sup> )	Greater than 0.25 mg/L
Chromium ( <sup>6+</sup> )	Greater than 1.2 mg/L
Cobalt	Greater than 0.05 mg/L
Color or turbidity	If the sample is turbid, add one 0.1-g scoop of RoVer Rust Remover to the blank in Step 6 (Step 3 for AccuVac procedure). Swirl to mix.
Copper	Greater than 0.6 mg/L
Cyanide	Greater than 2.8 mg/L
Manganese	Greater than 50.0 mg/L
Mercury	Greater than 0.4 mg/L
Molybdenum	Greater than 4.0 mg/L
Nickel	Greater than 1.0 mg/L
Nitrite Ion	Greater than 0.8 mg/L
pH	A sample pH of < 3 or > 4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH to 3–5 before adding reagent using a pH meter or pH paper and adding (dropwise) an appropriate amount of iron-free acid or base (i.e., 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution). Make a volume correction if significant volumes of acid or base are used.

### Summary of Method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron.

## IRON, TOTAL, continued

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
TPTZ Iron Reagent Powder Pillows, .....	1 pillow	100/pkg	26087-99	
Sample Cell, 10-20-25 mL, w/cap .....	1	6/pkg	24019-06	

### REQUIRED REAGENTS (Using AccuVac Ampuls)

TPTZ Iron Reagent AccuVac Ampuls .....	1 ampul	25/pkg	25100-25
--	---------	--------	----------

### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....	1	each	500-41H
Sample Cell, 10-20-25 mL, w/cap .....	1	6/pkg	24019-06

### OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn) .....	500 mL	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn) .....	500 mL	28336-49
Hydrochloric Acid Solution, 1:1, 6.0 N .....	500 mL	884-49
Iron Standard Solution, 100 mg/L Fe .....	100 mL	14175-42
Iron Standard Solution, Ampule, 25 mg/L Fe, 2 mL .....	20/pkg	24629-20
Nitric Acid, ACS .....	500 mL	152-49
Nitric Acid Solution, 1:1 .....	500 mL	2540-49
RoVer Rust Remover .....	454 g	300-01
Sodium Hydroxide Standard Solution, 1.0 N .....	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB	2450-32
Sulfuric Acid Standard Solution .....	100 mL MDB	1270-32
Water, deionized .....	4 L	272-56

### OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit .....	each	24052-00
Ampule Breaker, Ampules .....	each	24846-00
Cylinder, graduated, 25 mL .....	each	1081-40
Dropper, graduated, 0.5 and 1.0 mL marks .....	20/pkg	21247-20
Flask, volumetric, Class A, 250 mL .....	each	14574-46
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg	391-33
pH Meter, <i>sension</i> <sup>TM</sup> 1, portable, with electrode .....	each	51700-10
Pipet Filler, safety bulb .....	each	14651-00
Pipet, serological, 2 mL .....	each	532-36
Pipet TenSette, 0.1 to 1.0 mL .....	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL .....	each	14515-35

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

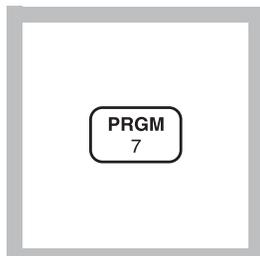
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**MANGANESE, High Range (0 to 20.0 mg/L)**

For water and wastewater

**Periodate Oxidation Method\*** USEPA approved for reporting wastewater analysis  
(digestion is required; see Section 2)\*\*

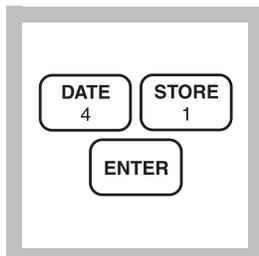


**1.** Enter the stored program number for manganese, periodate oxidation method.

Press: **PRGM**

The display will show:

**PRGM ?**



**2.** Press: **41 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon.

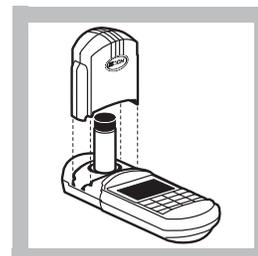
*Note:* For alternate forms ( $KMnO_4$ ,  $MnO_4^-$ ), press the **CONC** key.



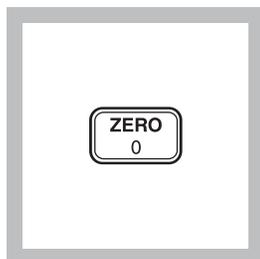
**3.** Fill a sample cell with 10 mL of sample (the blank).

*Note:* For total manganese determination perform a digestion (see Section 2).

*Note:* Adjust the pH of stored samples before analysis.



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



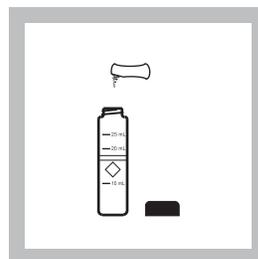
**5.** Press: **ZERO**

The cursor will move to the right, then the display will show:

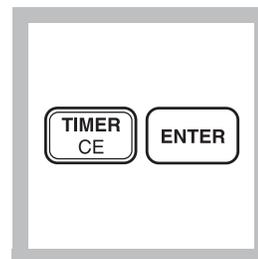
**0.0 mg/L Mn**



**6.** Remove the cell from the instrument. Add the contents of one Buffer Powder Pillow, citrate type, to the cell. Cap the cell and invert until the powder is dissolved. Remove cap.



**7.** Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap the sample cell. Invert for 10 seconds to mix.



**8.** Press: **TIMER ENTER**

A two-minute reaction period will begin.

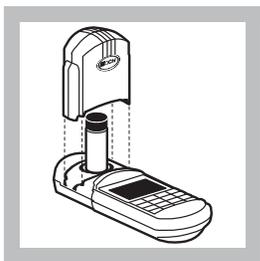
*Note:* A violet color will form if manganese is present.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

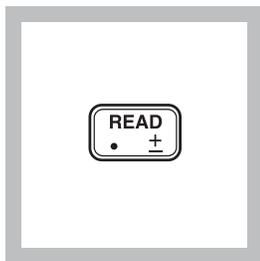
\*\* *Federal Register*, 44 (116) 34193 (June 14, 1979).

## MANGANESE, High Range, continued

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**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1 for more information. If only dissolved Mn is to be determined, filter before acid addition.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.
- b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.
- c) Transfer only 10 mL of each solution to the 10-mL sample cells.
- d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

## MANGANESE, High Range, continued

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- e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

### Standard Solution Method

Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or Class A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.18$  mg/L Mn.

#### Estimated Detection Limit

The estimated detection limit for program 41 is 0.2 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

### Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.

## MANGANESE, High Range, continued

### REQUIRED REAGENTS

High Range Manganese Reagent Set (100 tests) 10 mL .....	Cat. No.	24300-00
Includes: (1) 21076-69, (1) 21077-69		

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Buffer Powder Pillows, citrate type for Manganese .....	1 pillow .....	100/pkg .....	21076-69
Sodium Periodate Powder Pillows for Manganese .....	1 pillow .....	100/pkg .....	21077-69

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06
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### OPTIONAL REAGENTS

Drinking Water Standard, Metals, HR (Cu, Fe, Mn) .....	500 mL .....	28336-49
Hydrochloric Acid, 6 N .....	500 mL .....	884-49
Manganese Standard Solution, 1000 mg/L Mn .....	100 mL .....	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL .....	16/pkg .....	14258-10
Nitric Acid, ACS .....	500 mL .....	152-49
Nitric Acid Solution 1:1 .....	500 mL .....	2540-49
Sodium Hydroxide Solution, 5.0 N .....	100 mL MDB .....	2450-32
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit .....	each .....	21968-00
Flask, Erlenmeyer, 250 mL .....	each .....	505-46
Flask, volumetric, Class A, 50 mL .....	each .....	14574-41
Flask, volumetric, Class A, 100 mL .....	each .....	14574-42
Flask, volumetric, Class A, 1000 mL .....	each .....	14574-53
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> I, portable, with electrode .....	each .....	51700-10
Pipet, serological, 5 mL .....	each .....	532-37
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet, TenSette, 1.0 to 10.0 mL .....	each .....	19700-10
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet Tips, for 19700-10 TenSette Pipet .....	50/pkg .....	21997-96
Pipet, volumetric, Class A, 5.00 mL .....	each .....	14515-37
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Pipet Filler, safety bulb .....	each .....	14651-00

### *For Technical Assistance, Price and Ordering*

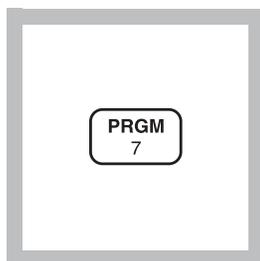
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## MANGANESE, Low Range (0 to 0.700 mg/L)

For water and wastewater

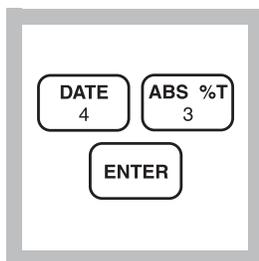
## PAN Method\*



1. Enter the stored program number for low range manganese.

Press: **PRGM**

The display will show:  
**PRGM ?**

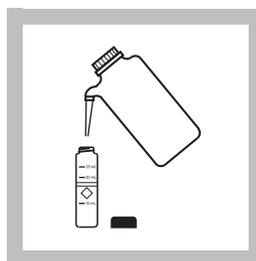


2. Press: **43 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon.

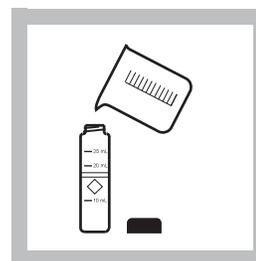
*Note:* For alternate forms ( $MnO_4$ ,  $KMnO_4$ ), press the **CONC** key.

*Note:* Total manganese determination requires a prior digestion; see Digestion (Section 2).

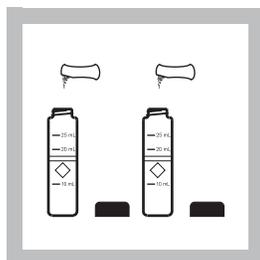


3. Fill a sample cell with 10 mL of deionized water (the blank).

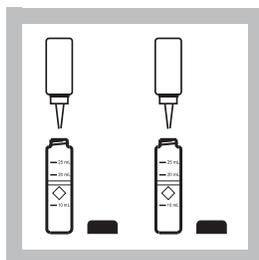
*Note:* Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with deionized water.



4. Fill another sample cell with 10 mL of sample (the prepared sample).



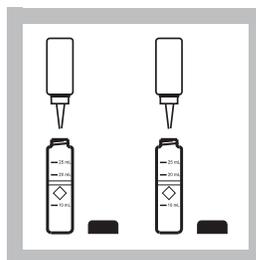
5. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Swirl to mix.



6. Add 12 drops of Alkaline-Cyanide Reagent Solution to each cell. Swirl to mix.

*Note:* A cloudy solution may form in some samples after reagent addition. The turbidity should dissipate after Step 8.

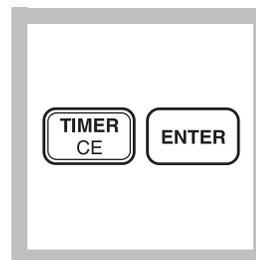
*Note:* A Tensette Pipet may be used to dispense 0.4 mL of the Alkaline Cyanide Reagent.



7. Add 12 drops of PAN Indicator Solution, 0.1%, to each sample cell. Swirl to mix.

*Note:* An orange color will develop in the sample if manganese is present.

*Note:* A Tensette Pipet may be used to dispense 0.4 mL of the PAN Indicator Solution.

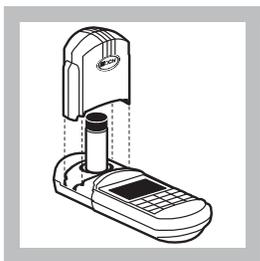


8. Press:  
**TIMER ENTER**  
A two-minute reaction period will begin.

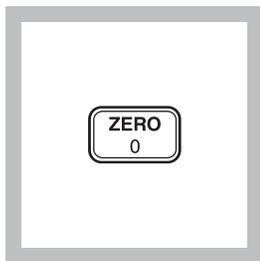
\* Adapted from Goto, K., et al., Talanta, 24, 752-3 (1977).

## MANGANESE, LR, continued

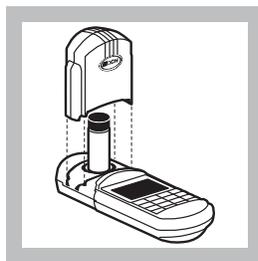
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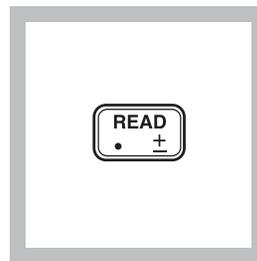
**9.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.000 mg/L Mn**



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L manganese will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

*Note: See Waste Management below for proper disposal of cyanide wastes.*

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### Sampling and Storage

Collect samples in a clean glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 4.0-5.0 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1.

### Accuracy Check

#### Standard Additions Method

*Note: Volume accuracy is very important when performing standard additions with 10-mL volumes. The fill mark on the 10-mL sample cell is not intended to measure standard addition volumes.*

- a) Fill three 10-mL graduated mixing cylinders with 10.0 mL of sample.
- b) Snap the neck off a Manganese Voluette Ampule Standard, 10 mg/L Mn.

## MANGANESE, LR, continued

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- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and mix each thoroughly.
- d) Analyze each sample as described in the procedure. The manganese concentration should increase 0.1 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions in Section 1* for more information.

*Note:* An alternative to the above procedure is to pipet 10.0 mL of sample into dry sample cells before performing standard additions. A volumetric pipet or a TenSette Pipet can be used to deliver the sample volume.

### Standard Solution Method

Prepare a 0.5 mg/L manganese standard solution as follows:

- a) Pipet 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask.
- b) Dilute to the mark with deionized water. Prepare this solution daily.
- c) Pipet 10.00 mL of the solution from Step b into a 100-mL volumetric flask.
- d) Dilute to the mark with deionized water. This second dilution is equivalent to 0.5 mg/L Mn.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.5 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.013$  mg/L Mn.

#### Estimated Detection Limit

The estimated detection limit for program 43 is 0.020 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

## MANGANESE, LR, continued

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### Interferences

The following do not interfere up to the indicated concentrations:

Substance	Suggested Treatment For Levels That Interfere
Aluminum	20 mg/L
Cadmium	10 mg/L
Cobalt	20 mg/L
Copper	50 mg/L
Hardness	300 mg/L.
Iron	If the sample contains more than 5 mg/L iron, allow 10 minutes for complete color development. Instead of performing Step 8, set the timer for 10 minutes by pressing <b>TIMER</b> twice. Then press <b>1000</b> . Press <b>ENTER</b> to start the timer.
Lead	0.5 mg/L
Magnesium	For samples containing hardness greater than 300 mg/L CaCO <sub>3</sub> , add four drops of Rochelle Salt Solution to the sample after addition of the Ascorbic Acid Powder Pillow.
Nickel	40 mg/L
Zinc	15 mg/L

### Waste Management

The alkaline cyanide solution contains cyanide. Cyanide solutions should be collected for disposal as reactive (D003) waste. Store all cyanide solutions in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. In case of a spill, clean up the area as outlined below:

1. Use a fume hood or self-contained breathing apparatus.
2. While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
3. Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
4. Flush the solution down the drain with a large excess of water.

### Summary of Method

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to Mn<sup>2+</sup>. An alkaline-cyanide reagent is added to mask any potential

## MANGANESE, LR, continued

interferences. PAN Indicator is then added to combine with the  $Mn^{2+}$  to form an orange-colored complex.

### REQUIRED REAGENTS

	Cat. No.
Manganese Reagent Set (50 tests).....	26517-00
Includes: (1) 21223-26, (1) 14577-99, (1) 21224-26	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Alkaline-Cyanide Reagent .....	30 drops	50 mL	SCDB	21223-26
Ascorbic Acid Powder Pillows .....	2 pillows	100	/pkg	14577-99
PAN Indicator Solution, 0.1% .....	42 drops	50 mL	SCDB	21224-26
Water, deionized .....	10 mL	4	L	272-56

### REQUIRED APPARATUS

Cylinder, graduated, 25 mL.....	1	each	508-40
Sample Cell, 10-20-25 mL, w/cap .....	2	6/pkg	24019-06

### OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn).....	500 mL	28337-49
Hydrochloric Acid Solution, 1:1 (6 N).....	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn.....	100 mL	12791-42
Manganese Standard Sol'n, Ampule, 25 mg/L Mn, 2 mL .....	20/pkg.	21128-20
Nitric Acid Solution, 1:1 .....	500 mL	2540-49
Rochelle Salt Solution.....	29 mL	DB 1725-33
Sodium Hydroxide Solution, 50% .....	500 mL	2180-49
Nitric Acid, ACS .....	500 mL	152-49

### OPTIONAL APPARATUS

Ampule Breaker, Ampule.....	each	24846-00
Beaker, glass, 1000 mL .....	each	500-53
Cylinder, graduated, mixing, 10 mL .....	each	20886-38
Dropper, plastic, calibrated, 1.0 mL.....	20/pkg	21247-20
Flask, volumetric, Class A, 1000 mL.....	each	14574-53
Flask, volumetric, Class A, 100 mL.....	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL .....	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.0 mL .....	each	14515-38
Pipet, volumetric, Class A, 5.0 mL .....	each	14515-37
Pipet Filler, safety bulb .....	each	14651-00

#### *For Technical Assistance, Price and Ordering*

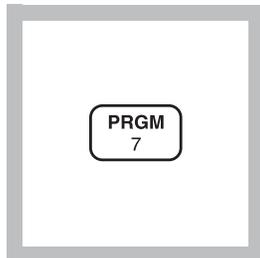
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**MOLYBDENUM, MOLYBDATE, Low Range (0 to 3.00 mg/L)****Ternary Complex Method**

For boiler and cooling tower waters



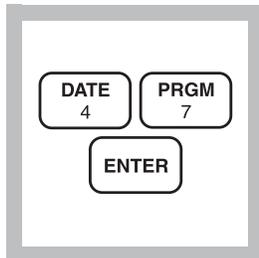
1. Enter the stored program number for molybdate molybdenum.

Press: **PRGM**

The display will show:

**PRGM ?**

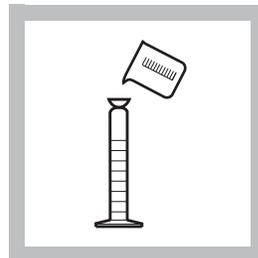
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **47 ENTER**

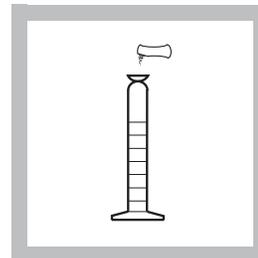
The display will show **mg/L, Mo6** and the **ZERO** icon.

*Note: For alternate forms (MoO<sub>4</sub>), press the **CONC** key.*



3. Fill a 25-mL mixing graduated cylinder with 20 mL of the sample.

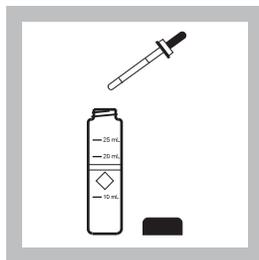
*Note: Filter turbid samples using the labware listed under *Optional Apparatus*.*



4. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the graduated cylinder. Stopper. Invert the graduated cylinder several times to dissolve the reagents.

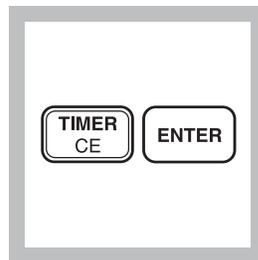


5. Pour 10 mL of the solution into a sample cell.



6. Add 0.5 mL of Molybdenum 2 Reagent to the sample cell. Swirl to mix. This is the prepared sample.

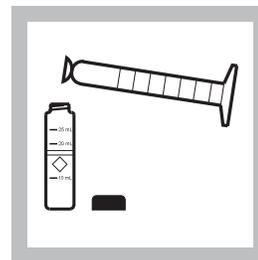
*Note: Molybdenum will cause a green color to form.*



7. Press:

**TIMER ENTER**

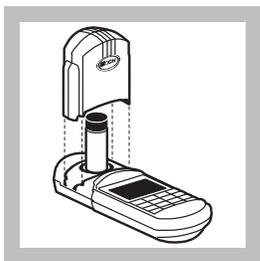
A two-minute reaction period will begin.



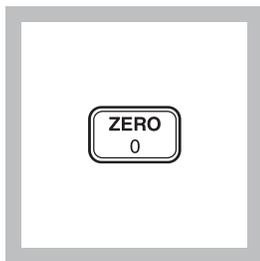
8. Fill a second sample cell with 10 mL of solution from the graduated cylinder (the blank).

## MOLYBDENUM, MOLYBDATE, LR, continued

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**9.** Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

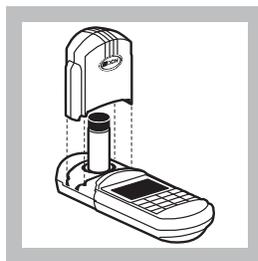


**10.** Press: **ZERO**

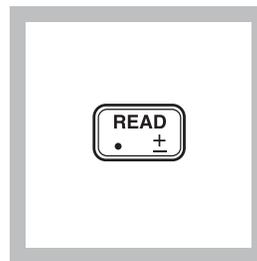
The cursor will move to the right, then the display will show:

**0.00 mg/L Mo<sup>6</sup>**

*Note: If Reagent Blank Correction is on, the display may flash "limit" (see Section 1).*



**11.** Place the developed sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**

The cursor will move to the right, then the result in mg/L molybdate molybdenum will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

---

### Sampling and Storage

Collect samples in glass or plastic bottles.

### Accuracy Check

#### Standard Addition Method

- a) Add 25 mL of sample to three 25-mL mixing cylinders.
- b) Snap the neck off a Molybdenum PourRite Ampule Standard Solution, 75 mg/L Mo<sup>6+</sup>.
- c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix thoroughly.
- d) Analyze 20 mL of each spiked sample as described in the procedure. The molybdenum concentration reading should increase by 0.3 mg/L for each 0.1 mL addition of standard.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Solution Method

Prepare a 2.0-mg/L molybdenum standard solution by pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-

## MOLYBDENUM, MOLYBDATE, LR, continued

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mL graduated mixing cylinder. Dilute to the mark with deionized water and mix thoroughly. Analyze 20 mL of this solution according to the procedure.

### Method Performance

#### Precision

In a single laboratory using standard solutions of 2.00 mg/L Mo<sup>6+</sup> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Mo<sup>6+</sup>.

#### Estimated Detection Limit

The estimated detection limit for program 47 is 0.07 mg/L Mo<sup>6+</sup>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interference studies were conducted by preparing a molybdenum standard solution (2 mg/L Mo<sup>6+</sup>) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered an interference.

Table 1 Negative Interferences

Ion	Level above which it interferes (mg/L)
Iron	200
Copper	98
Chromium (Cr <sup>6+</sup> )	4.5 <sup>1</sup>
Chloride	1,400
AMP (Phosphonate)	15
Phosphonohydroxyacetic Acid	32
Bisulfate	3,300
Nitrite	350
Aluminum	2
Acrylates	790
Alum	7
Lignin Sulfonate	105
Orthophosphate	4,500
Bicarbonate	5,650
EDTA	1,500
Borate	5,250
Ethylene Glycol	2% (by volume)
Sulfite	6,500
Diethanoldithiocarbamate	32

## MOLYBDENUM, MOLYBDATE, LR, continued

Table 1 Negative Interferences (continued)

Ion	Level above which it interferes (mg/L)
<b>Positive Interferences</b>	
Carbonate	1,325
Silica	600
Benzotriazole	210
Morpholine	6

<sup>1</sup> Read molybdenum concentration immediately after the completion of the two-minute reaction period.

Table 2 No Interference

Ion	Highest Concentration Tested (mg/L)
Zinc	400
Calcium	720
Magnesium	8,000
Manganese	1,600
Chlorine	7.5
PBTC (phosphonate)	500
Sulfate	12,800
Bisulfite	9,600
Nickel	250

Phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). For these samples, multiply the value obtained in step 12 by 0.9 to obtain the actual molybdenum concentration. As the concentration of HEDP increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require pretreatment. Adjust the sample pH to 3-5 (use a pH meter or pH paper) by adding drops of an acid or base such as 1.0 N Sulfuric Acid Standard Solution, or 1.0 N Sodium Hydroxide Standard Solution. If a significant volume of acid or base is used, correct the result by dividing the total volume (sample + acid + base) by the original volume and multiplying the test result by this factor.

## MOLYBDENUM, MOLYBDATE, LR, continued

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Large interferences are caused by some biocides used in cooling tower samples. Hach recommends testing the ternary complex procedure on molybdenum standards containing the specific biocides in use to determine if the ternary complex method will work with these samples.

After many samples have been analyzed, the sample cells may show a slight blue color. Rinse with Hydrochloric Acid Solution, 1:1, to eliminate the build-up.

### Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex.

---

### REQUIRED REAGENTS

Molybdenum Reagent Set, 20 mL sample (100 tests) .....24494-00  
Includes: (1) 23524-49, (1) 23525-12

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Molybdenum 1 Reagent for 20 mL sample size .....	1 pillow .....	100/pkg .....	23524-49	
Molybdenum 2 Reagent Solution.....	0.5 mL .....	.50 mL MDB .....	23525-12	

### REQUIRED APPARATUS

Cylinder, mixing, graduated, 25 mL ..... 1 .....each .....1896-40  
Sample Cell, 10-20-25 mL, w/cap ..... 2 .....6/pkg .....24019-06

### OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1, 6.0 N ..... 500 mL .....884-49  
Molybdenum Standard Solution, Ampule  
75 mg/L Mo<sup>6+</sup>, 2 mL .....20/pkg .....25575-20  
Molybdenum Standard Solution, 10 mg/L Mo<sup>6+</sup> ..... 100 mL .....14187-42  
Sodium Hydroxide Standard Solution, 1.0 N..... 100 mL MDB .....1045-32  
Water, deionized .....4 L .....272-56

## MOLYBDENUM, MOLYBDATE, LR, continued

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### OPTIONAL APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Cylinder, mixing, graduated, 50 mL.....		each.....	1896-41
Filter Paper, folded, 12.5 cm.....		100/pkg.....	1894-57
Funnel, poly, 65 mm.....		each.....	1083-67
pH Paper, 1-11 pH units.....		5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL.....		each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet.....		50/pkg.....	21856-96
Pipet, volumetric, 10.00 mL, Class A.....		each.....	14515-38
Pipet Filler, safety bulb.....		each.....	14651-00
PourRite Ampule Breaker.....		each.....	24846-00

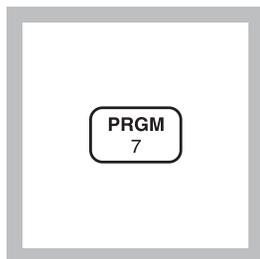
### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**MOLYBDENUM, MOLYBDATE, High Range (0 to 40.0 mg/L)****Mercaptoacetic Acid Method\***

For water and wastewater

**Using Powder Pillows**

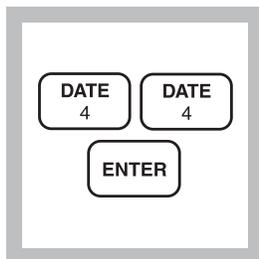
**1.** Enter the stored program number for high range molybdenum-powder pillows

Press: **PRGM**

The display will show:

**PRGM ?**

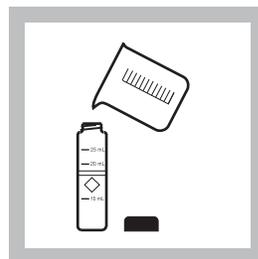
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon.

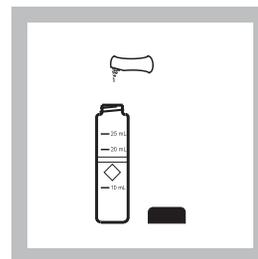
*Note: For alternate form (MoO<sub>4</sub>), press the **CONC** key.*



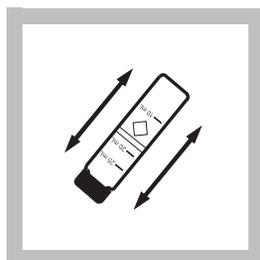
**3.** Fill a sample cell with 10 mL of sample.

*Note: Filter turbid samples.*

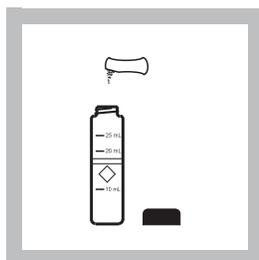
*Note: Adjust pH of stored samples before analysis.*



**4.** Add the contents of one MolyVer 1 Reagent Powder Pillow. Cap the cell and invert several times to mix.

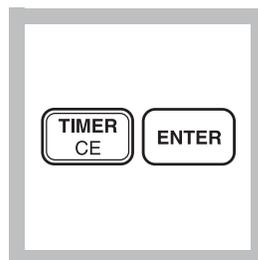


**5.** Add the contents of one MolyVer 2 Reagent Powder Pillow. Cap the cell and invert several times to mix.



**6.** Add the contents of one MolyVer 3 Reagent Powder Pillow. Cap the cell and invert several times to mix. This is the prepared sample.

*Note: Accuracy is not affected by undissolved powder.*

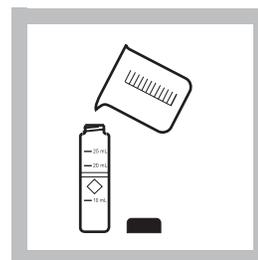


**7.** Press:

**TIMER ENTER**

A five-minute reaction period will begin.

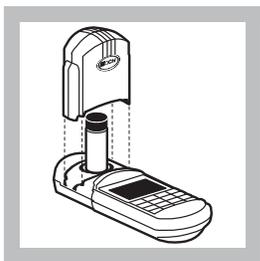
*Note: Molybdenum will cause a yellow color to form.*



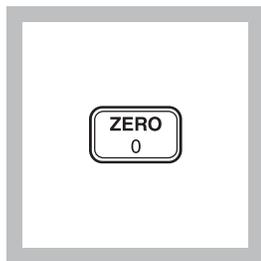
**8.** After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).

\* Adapted from Analytical Chemistry, 25(9) 1363 (1953).

## MOLYBDENUM, MOLYBDATE, HR, continued



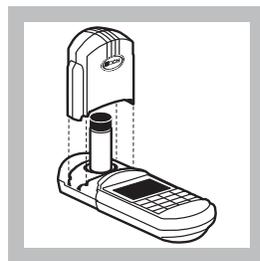
**9.** Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



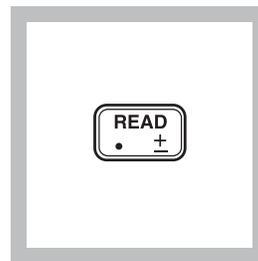
**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.0 mg/L Mo6**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



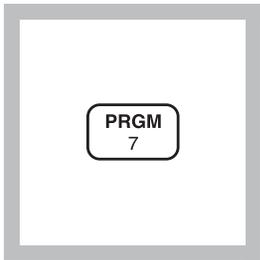
**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L molybdenum (or alternate form) will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagents is highly recommended. See Accuracy Check.*

### Using AccuVac Ampuls



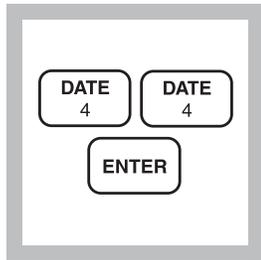
**1.** Enter the stored program number for high range molybdenum using AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **44 ENTER**  
The display will show **mg/L, Mo6** and the **ZERO** icon.

*Note: For alternate form (MoO<sub>4</sub>), press the CONC key.*

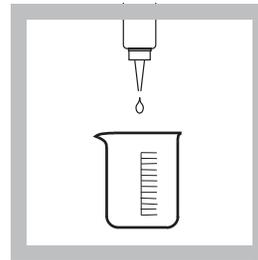


**3.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

*Note: Filter turbid samples.*

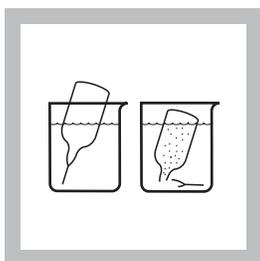
*Note: Adjust the pH of stored samples before analysis.*

### Method 10046



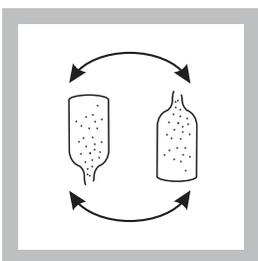
**4.** Add 4 drops of 0.4 M CDTA Solution to the beaker. Swirl to mix.

## MOLYBDENUM, MOLYBDATE, HR, continued



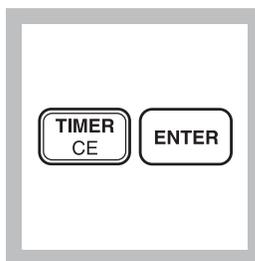
5. Fill a MolyVer 6 Reagent AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampul fills.*



6. Invert the ampul repeatedly to mix. Wipe off any liquid or fingerprints.

*Note: Undissolved reagent will not affect the result.*

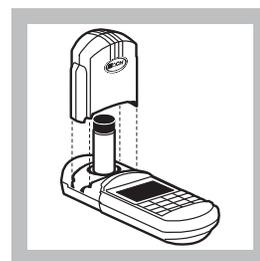


7. Press:

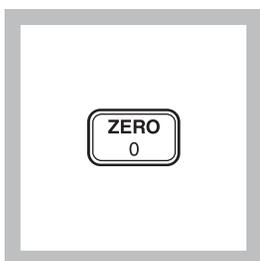
**TIMER ENTER**

A five-minute reaction period will begin.

*Note: If molybdenum is present a yellow color will develop.*



8. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

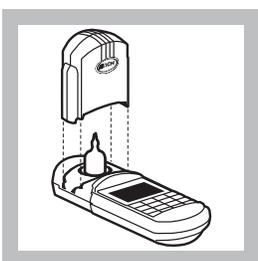


9. Press: **ZERO**

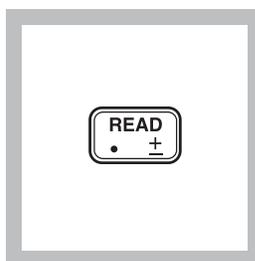
The cursor will move to the right, then the display will show:

**0.0 mg/L Mo6**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the AccuVac Ampul in the cell holder. Tightly cover the ampul with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

### Sampling and Storage

Collect samples in clean plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Volume Additions (Section 1)* for more information.

## MOLYBDENUM, MOLYBDATE, HR, continued

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### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo<sup>6+</sup>.
- c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.
- d) For analysis with AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.
- e) Analyze each standard addition sample as described in the procedure. The molybdenum concentration reading should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions in Section 1* for troubleshooting information.

#### Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo<sup>6+</sup>. Follow the procedure for powder pillows or AccuVac Ampuls. Results should be between 9.0 and 11.0 mg/L Mo<sup>6+</sup>.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 20.0 mg/L Mo<sup>6+</sup> and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.3 mg/L Mo<sup>6+</sup>.

## MOLYBDENUM, MOLYBDATE, HR, continued

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In a single laboratory using a standard solution of 20.0 mg/L Mo<sup>6+</sup> and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L Mo<sup>6+</sup>.

### Estimated Detection Limit

The estimated detection limit for program 44 is 0.2 mg/L Mo<sup>6+</sup>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 50 mg/L
Chromium	Greater than 1000 mg/L
Copper	Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the five-minute reaction period is complete.
Iron	Greater than 50 mg/L
Nickel	Greater than 50 mg/L
Nitrite	Interference from up to 2000 mg/L as NO <sub>2</sub> <sup>-</sup> can be eliminated by adding one Sulfamic Acid Powder Pillow to the sample.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see <i>Section 1, pH Interferences</i> .

### Summary of Method

#### Powder Pillows

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 1 contains a buffer to control the pH in addition to a chelating agent to mask interferences. MolyVer 3 provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

#### AccuVac Ampuls

The CDTA Solution masks metal interferences. The MolyVer 6 reagent provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

## MOLYBDENUM, MOLYBDATE, HR, continued

### REQUIRED REAGENTS (for Powder Pillows)

	Cat. No.
Molybdenum Reagent Set, 10 mL (100 tests) .....	26041-00
Includes: (1) 26042-99, (1) 26043-99, (1) 26044-99	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
MolyVer 1 Reagent Powder Pillows .....	1 pillow	.....	100/pkg	26042-99
MolyVer 2 Reagent Powder Pillows .....	1 pillow	.....	100/pkg	26043-99
MolyVer 3 Reagent Powder Pillows .....	1 pillow	.....	100/pkg	26044-99

### REQUIRED REAGENTS (for AccuVac Ampuls)

MolyVer 6 Molybdenum AccuVac Reagent Set (25 tests) .....				25220-98
Includes: (1) 25220-25, (1) 26154-36				
CDTA Solution 0.4M .....	4 drops	.....	15 mL SCDB	26154-36
MolyVer 6 Reagent AccuVac Ampuls .....	1 ampul	.....	25/pkg	25220-25

### REQUIRED APPARATUS (for Powder Pillows)

Sample Cell, 10-20-25 mL, w/cap .....	2	.....	6/pkg	24019-06
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### REQUIRED APPARATUS (for AccuVac Ampuls)

Beaker, 50 mL .....	2	.....	each	500-41H
Sample Cell, 10-20-25 mL, w/cap .....	1	.....	6/pkg	24019-06

### OPTIONAL REAGENTS

Molybdenum Standard Solution, 10 mg/L Mo <sup>6+</sup> .....	100 mL	.....	14187-42
Molybdenum Standard Solution, Voluette Ampule, 500 mg/L Mo <sup>6+</sup> , 10 mL .....	16/pkg	.....	14265-10
Nitric Acid, ACS .....	500 mL	.....	152-49
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB	.....	2450-32
Sulfamic Acid Powder Pillows .....	100/pkg	.....	1055-99
Water, deionized .....	4 L	.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each	.....	24052-00
Ampule Breaker Kit .....	each	.....	21968-00
Cylinder, graduated, mixing, 25 mL .....	each	.....	20886-40
Filter Paper, folded, 12.5 cm .....	100/pkg	.....	1894-57
Flask, Erlenmeyer, 250 mL .....	each	.....	505-46
Funnel, poly, 65 mm .....	each	.....	1083-67
Pipet, serological, 5 mL .....	each	.....	532-37
Pipet, TenSette, 0.1 to 1.0 mL .....	each	.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg	.....	21856-28

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# Nitrogen, Free Ammonia and Chloramine (Mono)

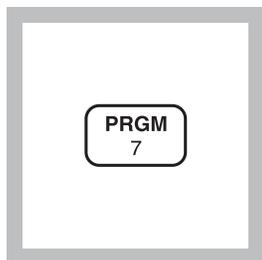
Method 10200

## Indophenol Method\*

(0–4.50 mg/L Cl<sub>2</sub> and 0–0.50 mg/L NH<sub>3</sub>-N)

For finished chloraminated drinking water

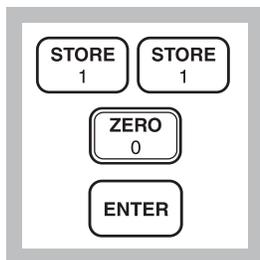
*Note: For the most accurate chloramine results, determine a reagent blank for each new lot of reagent using deionized water instead of sample. Subtract the blank value from the final chloramine result.*



1. Enter the user program number for monochloramine.

Press: **PRGM**

The display will show:  
**PRGM?**



2. Press:

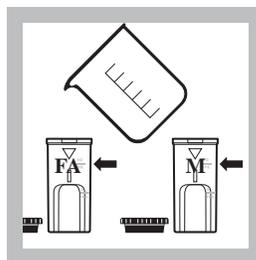
**110 ENTER**

The display will show

**mg/L Cl<sub>2</sub>**

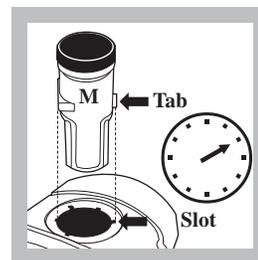
and the zero icon.

*Note: For alternate forms, press the **CONC** key.*



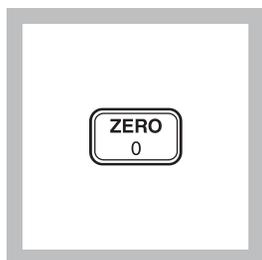
3. Fill two cells with 10 mL of sample

Label one cell “Free Ammonia” and one cell “Monochloramine”.



4. Place the Monochloramine cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

Tightly cover the sample cell with the instrument cap.

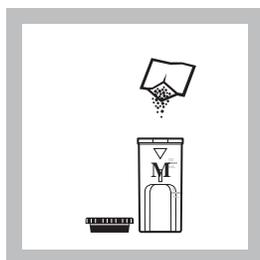


5. Press: **ZERO**

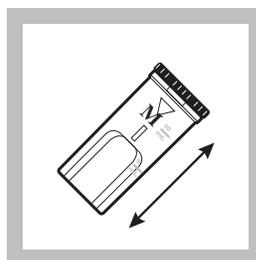
The cursor will move to the right, then the display will show:

0.00 mg/L Cl<sub>2</sub>

Remove the cell from the instrument.

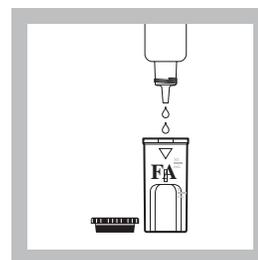


6. Add the contents of one pillow of Monochlor F to the cell for the Monochloramine measurement.



7. Cap the cell and shake for 20 seconds to dissolve the reagent.

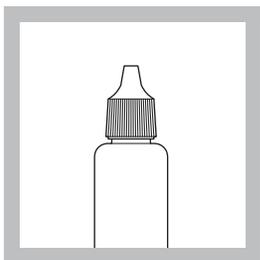
A green color will form if monochloramine is present.



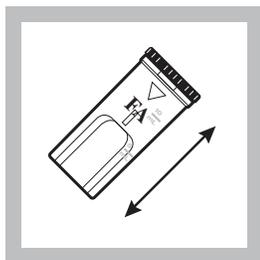
8. Add one drop of Free Ammonia Reagent Solution to the cell for Free Ammonia measurement.

\* U.S. Patent 6,315,950

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

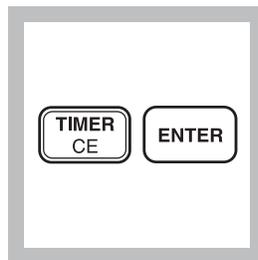


**9.** Cap the reagent bottle to maintain reagent performance and stability.



**10.** Cap the cell and mix.

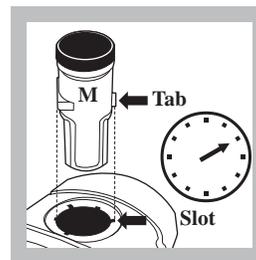
*Note: If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 296.*



**11.** Press: **TIMER ENTER**

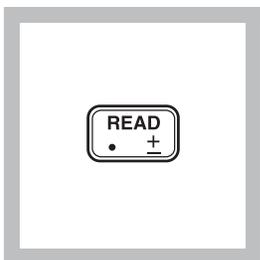
A five-minute reaction period will begin.

*Note: The color development time depends on the sample temperature. See Table 1. For accurate results allow the full reaction period to occur.*



**12.** When the timer expires, place the Monochloramine cell into the instrument so that the cell tab is in the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

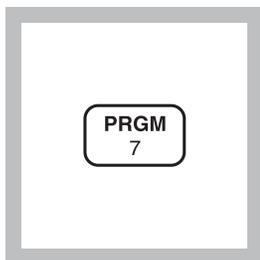
Tightly cover the sample cell with the instrument cap.



**13.** Press: **READ**

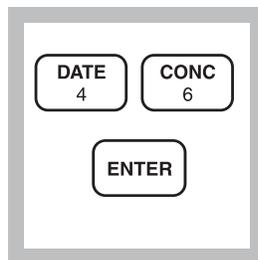
The cursor will move to the right, then the result in mg/L Monochloramine (as  $\text{Cl}_2$  or chosen units) will be displayed.

Leave the cell in the instrument.



**14.** Enter the stored program number for Free Ammonia.

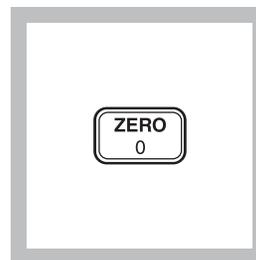
Press: **PRGM**  
The display will show PRGM?



**15.** Press: **46 ENTER**

The display will show  $\text{NH}_3\text{-N}$  and the zero icon.

*Note: For alternate forms, press the CONC key.*



**16.** With the Monochloramine sample still in the cell holder, press **ZERO**.

The cursor will move to the right, then the display will show: 0.00 mg/L  $\text{NH}_3\text{-N}$ .

Remove the cell from the instrument.

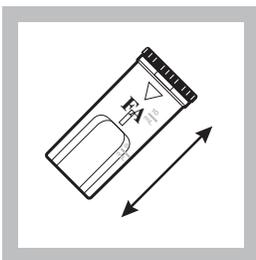
## Nitrogen, Free Ammonia and Chloramine (Mono), continued



**17.** Add the contents of one pillow of Monochlor F to the cell for the Free Ammonia measurement.

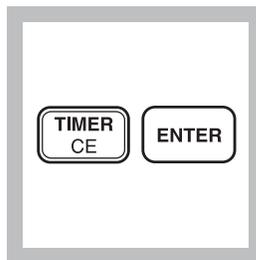
Cap and shake the cell about 20 seconds to dissolve the reagent.

*Note: The reaction period indicated in step 11 must be complete before the addition of Monochlor F to the cell for free ammonia measurement.*



**18.** Cap and shake the cell about 20 seconds to dissolve the reagent.

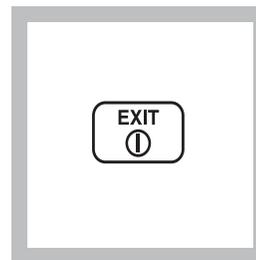
A green color will form if ammonia or monochloramine is present.



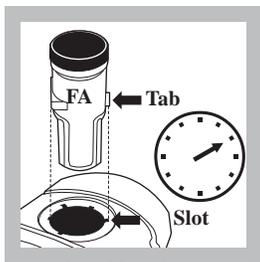
**19.** Press: **TIMER ENTER**

A five-minute reaction period will begin.

*Note: The color development time depends on the sample temperature. See Table 1.*

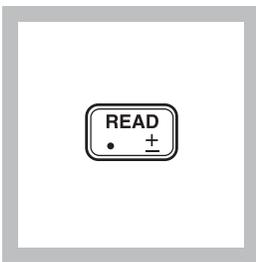


**20.** After the timer has expired, press: **EXIT**



**21.** Place the Free Ammonia cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

Tightly cover the sample cell with the instrument cap.



**22.** Press: **READ**

The cursor will move to the right, then the result in mg/L free ammonia as nitrogen ( $\text{NH}_3\text{-N}$ ) or chosen units will be displayed.

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

---

### Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

### Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (68–73 °F). Adjust both reaction periods according to Table 3.

Table 3 Reaction Period

Sample Temperature		Reaction Periods (Minutes)
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	5
20	73	5
23	75	2.5
25	77	2
>25	>77	2

### Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination when at or below the stated concentration.

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

---

Substance	Level Tested
Aluminum	0.2 mg/L Al
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO <sub>3</sub> -N
Nitrite	1 mg/L NO <sub>2</sub> -N
Phosphate	2 mg/L -PO <sub>4</sub>
Silica	100 mg/L SiO <sub>2</sub>
Sulfate	1600 ppm as CaCO <sub>3</sub>
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become turbid (cloudy) after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

*Note: The sample for Monochloramine measurement does not need pretreatment.*

1. Measure 10 mL of sample into the cell for Free Ammonia measurement.
2. Add the contents of one Hardness Treatment Reagent Powder Pillow (Cat. No. 28823-46) to the sample.
3. Cap the cell and invert until the reagent is dissolved.
4. Remove the cap.

Continue with the analysis at step 2 using the pretreated sample as the Free Ammonia cell.

### Accuracy Check (Monochloramine, Program 110)

1. Prepare the following monochloramine standard fresh before use.
2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.
3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH<sub>3</sub>-N into the flask.

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

---

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.
5. Pipet 50.0 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.
6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.
7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir plate.
9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.
10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl<sub>2</sub>) monochloramine standard.

Use this standard within 1 hour of preparation.

*Important Note: Because of the strong buffer used in the preparation of this standard, it cannot be used for accuracy verification of the Free Ammonia test.*

### Accuracy Check (Free Ammonia Test, Program 46)

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

### Standard Additions Method

1. Measure 50 mL of sample into three 50-mL mixing cylinders.
2. Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH<sub>3</sub>-N to the three samples. Mix well.

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

---

3. Analyze each spiked sample, following all steps of the Monochloramine and Free Ammonia procedure. The ammonia nitrogen concentration should increase 0.02 mg/L for each 0.1 mL of standard added.
4. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

### Standard Solution Method

Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as  $\text{NH}_3\text{-N}$ , to 100 mL with dilution water. Analyze the standard solution, following all steps of the Monochloramine and Free Ammonia procedure.

### Method Performance

#### Monochloramine Test

##### Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L  $\text{Cl}_2$  and representative lots of reagent, a single operator obtained a standard deviation of  $\pm 0.12$  mg/L  $\text{Cl}_2$ .

##### Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L  $\text{Cl}_2$ .

#### Free Ammonia Test

##### Precision

In a single laboratory using a solution containing 1.80 mg/L  $\text{Cl}_2$  plus 0.20 mg/L ammonia nitrogen ( $\text{NH}_3\text{-N}$ ) and two representative lots of reagent with the DR/890, a single operator obtained a standard deviation of  $\pm 0.01$  mg/L N for seven replicates.

##### Estimated Detection Limit

The estimated detection limit for program 46 is 0.02 mg/L N.

For more information on the estimated detection limit, see *Section 1 of the DR/850 or DR/890 Procedure Manual*.

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

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### Summary of Method

Monochloramine ( $\text{NH}_2\text{Cl}$ ) and “free ammonia” ( $\text{NH}_3$  and  $\text{NH}_4^+$ ) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite.

### Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests) Includes: (1) 28022-99, (1) 28773-36.....			28797-00
Free Ammonia Reagent Solution.....	1 drop	4 mL SCDB	28773-36
Monochlor F Reagent Pillows .....	2 pillows	100/pkg	28022-99

### REQUIRED APPARATUS

Sample Cell, 1-cm/10-mL, with cap.....	2	2/pkg	48643-02
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### OPTIONAL REAGENTS

Buffer, pH 8.3, Powder Pillows .....	25/pkg		898-68
Chlorine Solution, Voluette <sup>®</sup> Ampule .....	16/pkg		14268-10
Hardness Treatment Reagent Pillows (1 per test).....	50/pkg		28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as $\text{NH}_3\text{-N}$ .....	500 mL		153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as $\text{NH}_3\text{-N}$ , 10 mL.....	16/pkg		14791-10
Nitrogen Ammonia Standard Solution, 100 mg/L as $\text{NH}_3\text{-N}$ .....	500 mL		24065-10

## Nitrogen, Free Ammonia and Chloramine (Mono), continued

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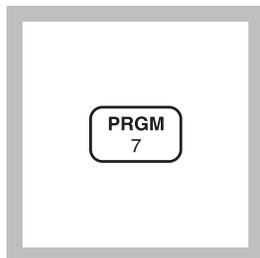
### OPTIONAL APPARATUS

Description	Per Test	Unit	Cat. No.
Ampule Breaker Kit .....		each	21968-00
Beaker, 100 mL, Polypropylene.....		each	1080-42
Beaker, 100 mL, Glass .....		each	500-42H
Cylinder, 50 mL, mixing .....		each	20886-41
Flask, Volumetric, Class A, 100 mL .....		each	14574-42
Pipet Filler, Safety Bulb .....		each	14651-00
Pipet, TenSette <sup>®</sup> , 0.1 to 1.0 mL.....		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg		21856-96
Pipet, Mohr, Glass, 10 mL .....		each	20934-38
Pipet, Volumetric, Class A, 2.0 mL.....		each	14515-36
Pipet, Volumetric, Class A, 50.00 mL.....		each	14515-41
Scissors.....		each	28831-00
Stir Bar, Octagonal .....		each	20953-53
Stirrer, Magnetic.....		each	23436-00
Thermometer, -10 to 110 °C.....		each	1877-01
Wipers, Disposable Kimwipes <sup>®</sup> , 30 x 30 cm, 280/box.....		box	20970-01



**NICKEL (0 to 1.000 mg/L)**

For water and wastewater

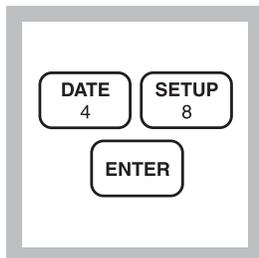
**PAN Method\***

**1.** Enter the stored program number for nickel (Ni), PAN method.

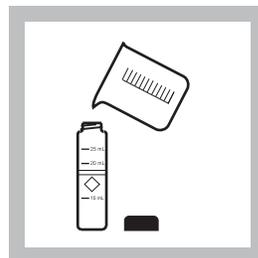
Press: **PRGM**

The display will show:

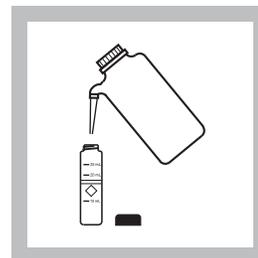
**PRGM ?**



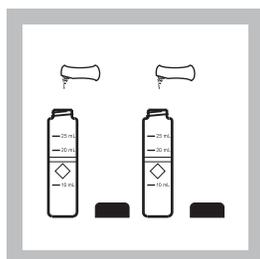
**2.** Press: **48 ENTER**  
The display will show **mg/L, Ni** and the **ZERO** icon.



**3.** Fill a sample cell with 25 mL of sample (the prepared sample).  
*Note: If sample is less than 10 °C (50 °F), warm to room temperature before analysis. Adjust the pH of stored samples.*

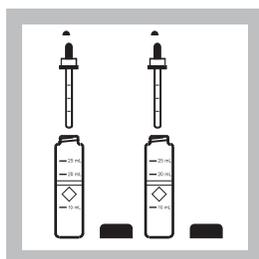


**4.** Fill a second sample cell with 25 mL of deionized water (the blank).



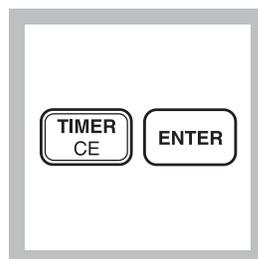
**5.** Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell. Cap. Invert several times to mix.

*Note: If sample contains iron ( $Fe^{3+}$ ), all the powder must be dissolved completely before continuing with Step 6.*



**6.** Add 1.0 mL of 0.3% PAN Indicator Solution to each cell. Cap. Invert several times to mix.

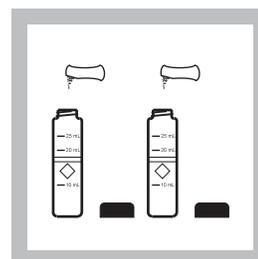
*Note: Use the plastic dropper provided.*



**7.** Press:  
**TIMER ENTER**

A 15-minute reaction period will begin.

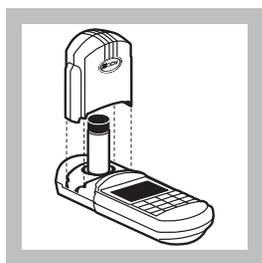
*Note: The sample solution color may vary from yellowish-orange to dark red. The blank should be yellow.*



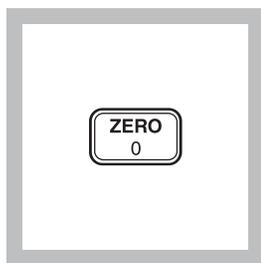
**8.** After the timer beeps, add the contents of one EDTA Reagent Powder Pillow to each cell. Cap. Invert several times to dissolve the reagent.

\* Adapted from Watanabe, H., Talanta, 21 295 (1974)

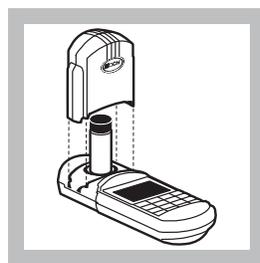
## NICKEL, continued



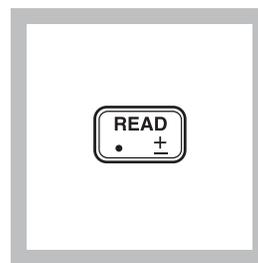
**9.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.000 mg/L Ni**



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L nickel will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

### Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions, see *Correcting for Volume Additions*, (Section 1) for more information.

### Accuracy Check

#### Standard Solution Method

Prepare a 0.5 mg/L nickel standard solution by diluting 10.0 mL of a 5 mg/L working stock solution to 100 mL in a 100-mL volumetric flask. The working stock solution should be prepared daily by diluting 5.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with deionized water.

Or, using the TenSette Pipet, add 0.2 mL of a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni, into a 100-mL volumetric flask. Dilute to volume with deionized water. This is a 0.6 mg/L standard solution.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.50 mg/L nickel and two representative lots of reagent with the instrument,

## NICKEL, continued

---

a single operator obtained a standard deviation of  $\pm 0.008$  mg/L nickel.

### Estimated Detection Limit

The estimated detection limit for program 48 is 0.013 mg/L Ni. For more information on the estimated detection limit, see *Section 1*.

### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level
Al <sup>3+</sup>	32 mg/L
Ca <sup>2+</sup>	1000 mg/L as (CaCO <sub>3</sub> )
Cd <sup>2+</sup>	20 mg/L
Cl <sup>-</sup>	8000 mg/L
Co	Causes a positive interference at all levels.
Cr <sup>3+</sup>	20 mg/L
Cr <sup>6+</sup>	40 mg/L
Cu <sup>2+</sup>	15 mg/L
F <sup>-</sup>	20 mg/L
Fe <sup>3+</sup>	10 mg/L
Fe <sup>2+</sup>	Interferes directly and must not be present.
K <sup>+</sup>	500 mg/L
Mg <sup>2+</sup>	400 mg/L
Mn <sup>2+</sup>	25 mg/L
Mo <sup>6+</sup>	60 mg/L
Na <sup>+</sup>	5000 mg/L
Pb <sup>2+</sup>	20 mg/L
Zn <sup>2+</sup>	30 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and required sample pretreatment; see *pH Interferences (Section 1)*.

Chelating agents, such as EDTA, interfere. Use either the Digesdahl or vigorous digestion (*Section 2*) to eliminate this interference.

### Summary of Method

After buffering the sample and masking any Fe<sup>3+</sup> with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator.

## NICKEL, continued

The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt.

### REQUIRED REAGENTS

	Cat. No.
Nickel Reagent Set, 25 mL sample (100 tests) .....	22426-00
Includes: (2) 7005-99, (4) 21501-66, (2) 21502-32	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
EDTA Reagent Powder Pillows.....	2 pillows		100/pkg	7005-99
Phthalate-Phosphate Reagent Powder Pillows .....	2 pillows		50/pkg	21501-66
P.A.N. Indicator Solution, 0.3%.....	2 mL		100 mL MDB	21502-32
Water, deionized.....	10 mL		4 L	272-56

### REQUIRED APPARATUS

Clippers, for opening powder pillows.....	1		each	968-00
Cylinder, graduated, mixing, 25 mL.....	1		each	20886-40
Sample Cell, 10-20-25, w/caps.....	2		6/pkg	24019-06

### OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L Ni .....		100 mL		14176-42
Nickel Standard Solution, Voluette Ampule, 300 mg/L Ni, 10 mL.....		16/pkg		14266-10
Nitric Acid, ACS.....		500 mL		152-49
Nitric Acid Solution, 1:1.....		500 mL		2540-49
Sodium Hydroxide Standard Solution, 5.0 N .....		100 mL	MDB	2450-32

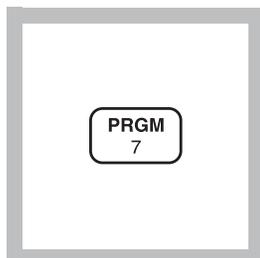
### OPTIONAL APPARATUS

Ampule Breaker Kit.....			each	21968-00
Flask, volumetric, Class A, 100 mL .....			each	14574-42
Flask, volumetric, Class A, 1000 mL .....			each	14574-53
pH Paper, 1 to 11 pH units.....		5 rolls/pkg		391-33
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode .....			each	51700-10
Pipet, serological, 1 mL .....			each	9190-02
Pipet, serological, 5 mL .....			each	532-37
Pipet, TenSette, 0.1 to 1.0 mL.....			each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....		50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....		1000/pkg		21856-28
Pipet, volumetric, Class A, 5.0 mL.....			each	14515-37
Pipet, volumetric, Class A, 10.0 mL.....			each	14515-38
Pipet Filler, safety bulb .....			each	14651-00
Thermometer, -20 to 110 °C, non-mercury .....			each	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**NITRATE, High Range (0 to 30.0 mg/L NO<sub>3</sub><sup>-</sup>-N) For water, wastewater, and seawater\*****Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)  
Using Powder Pillows**

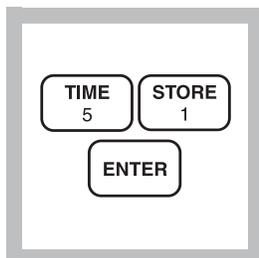
**1.** Enter the stored program number for high range nitrate nitrogen (NO<sub>3</sub><sup>-</sup>-N) powder pillows.

Press: **PRGM**

The display will show:

**PRGM ?**

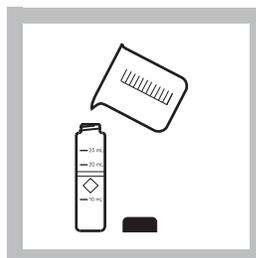
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **51 ENTER**

The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

*Note: For alternate forms (NO<sub>3</sub>), press the **CONC** key.*



**3.** Fill a sample cell with 10 mL of sample.

*Note: Adjust the pH of stored samples before analysis.*

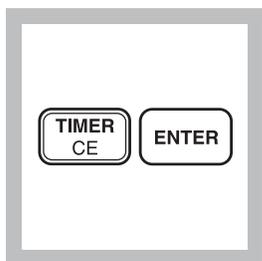


**4.** Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the sample cell.

*Note: It is important to remove all of the powder from the foil pillow. Tap the pillow until no more powder pours out.*

\* Seawater requires a manual calibration; see Interferences.

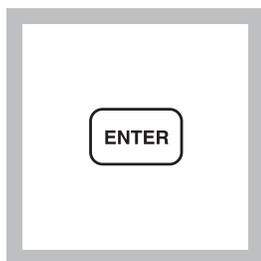
## NITRATE, High Range, continued



5. Press:  
**TIMER ENTER**

A one-minute reaction period will begin. Shake the sample cell vigorously until the timer beeps.

*Note: It is important to shake the cell vigorously. Shaking time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the shaking time to obtain the correct result.*



6. After the timer beeps, the display will show:

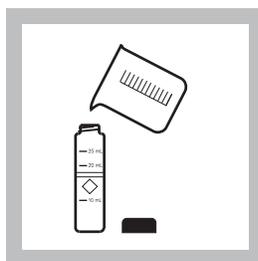
**5:00 TIMER 2**

Press: **ENTER**

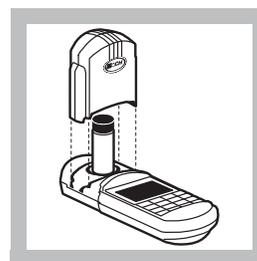
A five-minute reaction period will begin.

*Note: A deposit will remain after the reagent dissolves and will not affect test results.*

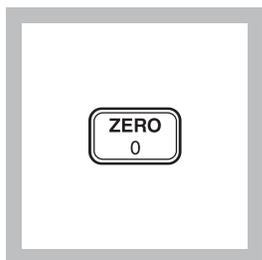
*Note: An amber color will develop if nitrate nitrogen is present.*



7. Fill another cell with 10 mL of sample (the blank). Wipe off any fingerprints or liquid.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

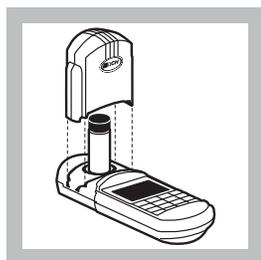


9. When the timer beeps, press **ZERO**.

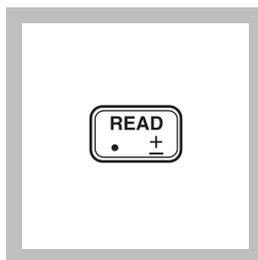
The cursor will move to the right, then the display will show:

**0.0 mg/L NO<sub>3</sub>-N**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

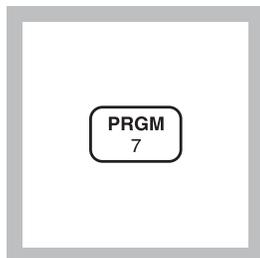
The cursor will move to the right, then the result in mg/L NO<sub>3</sub>-N (or alternate form) will be displayed.

*Note: Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See Accuracy Check.*

*Note: Rinse the sample cell immediately after use to remove all cadmium particles. Save the spent sample for proper hazardous waste disposal for cadmium.*

## NITRATE, High Range, continued

### Using AccuVac Ampuls



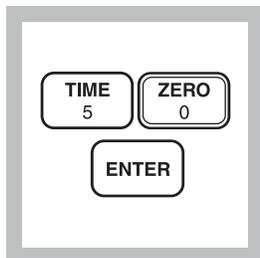
1. Enter the stored program number for high range nitrate nitrogen ( $\text{NO}_3^- - \text{N}$ ) AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

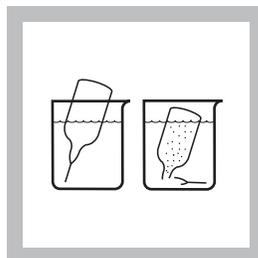
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **50 ENTER**

The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

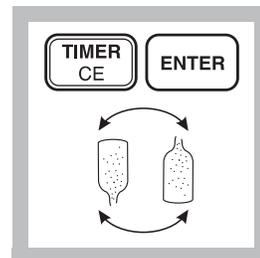
*Note: For alternate forms ( $\text{NO}_3$ ), press the **CONC** key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

*Note: Keep the tip immersed while the ampul fills. The ampul will not fill completely.*

*Note: Adjust the pH of stored samples before analysis.*

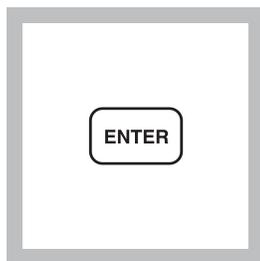


4. Press:

**TIMER ENTER**

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints.

*Note: Mixing time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the mixing time to obtain the correct result.*



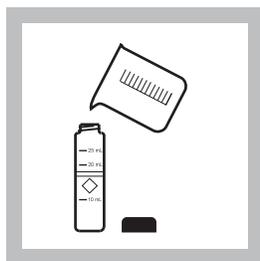
5. The display will show: **5:00 TIMER 2**

Press: **ENTER**

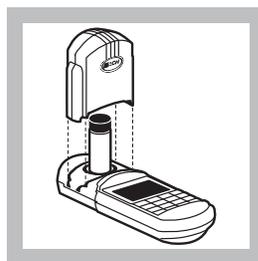
A five-minute reaction period will begin.

*Note: A deposit will remain after the reagent dissolves and will not affect results.*

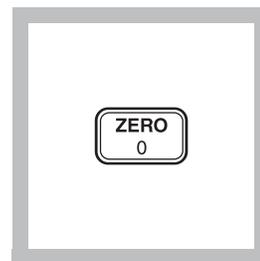
*Note: An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

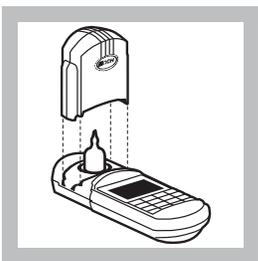
The cursor will move to the right, then the display will show:

**0.0 mg/L NO<sub>3</sub>-N**

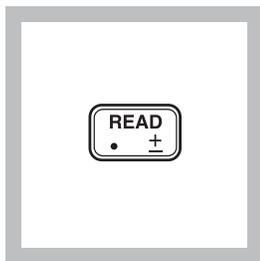
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

## NITRATE, High Range, continued

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**9.** Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L NO<sub>3</sub>-N (or alternate form) will be displayed.

*Note: Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See Accuracy Check.*

*Note: See Pollution Prevention and Waste Management for proper disposal of cadmium.*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions (Section 1)* for more information.

## NITRATE, High Range, continued

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### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard, 500 mg/L nitrate nitrogen.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen Standard Solution to the three samples. Stopper and mix thoroughly.
- d) For AccuVac analysis, transfer the solutions to clean, dry 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to clean, dry sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen ( $\text{NO}_3^-$ -N) concentration should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

#### Standard Solution Method

Use a Hach Nitrate-Nitrogen Standard Solution, 10.0 mg/L  $\text{NO}_3^-$ -N, listed under Optional Reagents as the sample and perform the procedure as described above.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. See *Section 1, Standard Curve Adjustment* for more information. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust value is entered.

### Method Performance

#### Precision

In a single laboratory using standard solutions of 25.0 mg/L nitrate nitrogen ( $\text{NO}_3^-$ -N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.3$  mg/L nitrate nitrogen for program #50 and  $\pm 1.7$  mg/L nitrate nitrogen for program # 51.

## NITRATE, High Range, continued

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### Estimated Detection Limit

The estimated detection limit for program 50 is 0.5 mg/L NO<sub>3</sub><sup>-</sup>-N and 0.8 mg/L NO<sub>3</sub><sup>-</sup>-N for program 51. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels Compensate for nitrite interference as follows: Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Add one drop of 30-g/L Phenol Solution to destroy the color. Proceed with Step 4. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

### Summary Of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

### Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

## NITRATE, High Range, continued

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
NitraVer 5 Nitrate Reagent Powder Pillows.....	1 pillow	.....	100/pkg	.....21061-69
Sample Cell, 10-20-25 mL, w/cap .....	2	.....	6/pkg	.....24019-06

### REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul .....	1 ampul	.....	25/pkg	.....25110-25
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### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....	1	.....	each	.....500-41H
Stopper .....	1	.....	6/pkg	.....1731-06

### OPTIONAL REAGENTS

Bromine Water 30 g/L .....	29 mL	.....	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as (NO <sub>3</sub> <sup>-</sup> -N) .....	500 mL	.....	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L as (NO <sub>3</sub> <sup>-</sup> -N) .....	500 mL	.....	12792-49
Nitrate Nitrogen Standard Solution, PourRite ampule, 500 mg/L as NO <sub>3</sub> <sup>-</sup> -N, 2 mL .....	20/pkg	.....	14260-20
Phenol Solution .....	29 mL	.....	2112-20
Sodium Hydroxide Standard Solution, 5.0 N.....	50 mL*	.....	2450-26
Sulfuric Acid, ACS .....	500 mL*	.....	979-49
Water, deionized .....	4 L	.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each	.....	24052-00
Cylinder, graduated, mixing, 25 mL .....	each	.....	1896-40
Dropper, for 29-mL bottle .....	each	.....	2258-00
pH Indicator Paper, 1 to 11 pH.....	5 rolls/pkg	.....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable, with electrode.....	each	.....	51700-10
Pipet Filler, safety bulb .....	each	.....	14651-00
Pipet, serological, 2 mL.....	each	.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL .....	each	.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg	.....	21856-28
PourRite Ampule Breaker .....	each	.....	24846-00
Thermometer, -20 to 110 °C, non-mercury .....	each	.....	26357-02

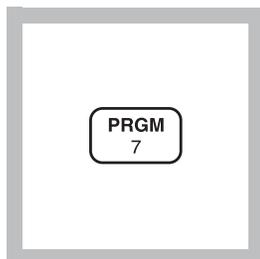
### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

\* Contact Hach for larger sizes.



**NITRATE, Mid Range (0 to 5.0 mg/L NO<sub>3</sub><sup>-</sup>-N) For water, wastewater and seawater\*****Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)****Using Powder Pillows**

**1.** Enter the stored program number for medium range nitrate nitrogen using powder pillows.

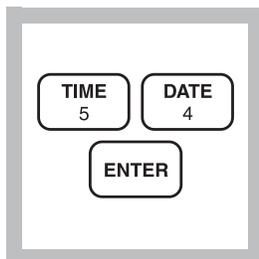
Press: **PRGM**

The display will show:

**PRGM ?**

*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

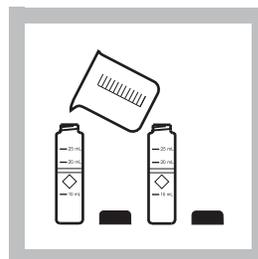
*Note:* Adjust the pH of stored samples before analysis.



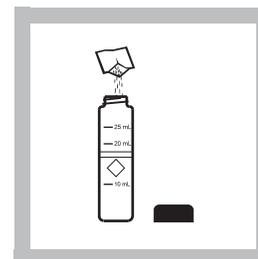
**2.** Press: **54 ENTER**

The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

*Note:* For alternate form (NO<sub>3</sub>), press the **CONC** key.



**3.** Fill two sample cells with 10 mL of sample each. One cell will be the prepared sample, the other is the blank. Set the blank aside.



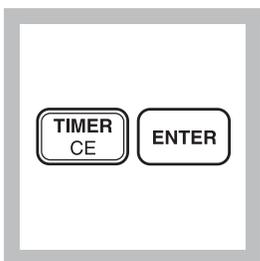
**4.** Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to one cell (the prepared sample). Cap the cell.

*Note:* It is necessary to remove all the powder from the foil pouch by tapping repeatedly until no more powder comes out.

\* Seawater requires a manual calibration; see *Interferences*.

## NITRATE, Mid Range, continued

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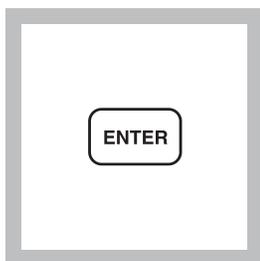


5. Press:

**TIMER ENTER**

A one-minute reaction period will begin. Shake the sample vigorously until the timer beeps.

*Note: Shaking time and technique influence color development. Low results usually occur if shaking is not vigorous enough. For most accurate results, do successive tests on a standard solution and adjust the shaking time by  $\pm 1$  minute to obtain the correct result. See the Accuracy Check section for more information.*



6. After the timer beeps, the display will show:

**5:00 TIMER 2**

Press: **ENTER**

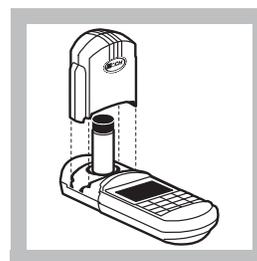
A five-minute reaction period will begin.

*Note: A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect test results.*

*Note: An amber color will develop if nitrate nitrogen is present.*



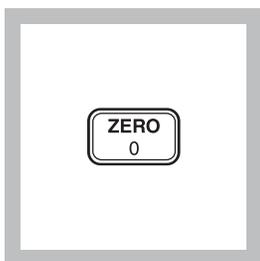
7. After the timer beeps, wipe off any liquid or fingerprints.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## NITRATE, Mid Range, continued

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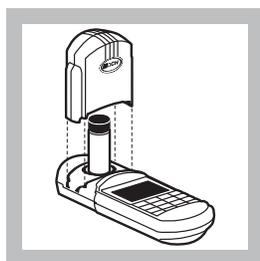


**9. Press: ZERO**

The cursor will move to the right, then the display will show:

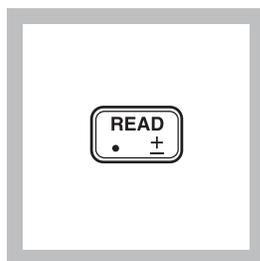
**0.0 mg/L NO<sub>3</sub>-N**

*Note: If Reagent Blank Correction is on, the display may flash "limit".*



**10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**

*Note: Read the sample within two minutes after the timer beeps.*



**11. Press: READ**

The cursor will move to the right, then the result in mg/L NO<sub>3</sub>-N (or NO<sub>3</sub>) will be displayed.

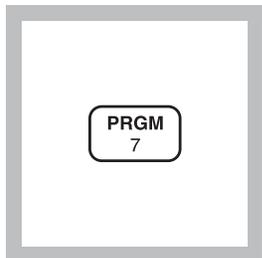
*Note: Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

*Note: Rinse the sample cell immediately after use to remove all the cadmium particles. See Pollution Prevention and Waste Management following these steps for disposal of cadmium particles.*

## NITRATE, Mid Range, continued

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### Using AccuVac Ampuls



**1.** Enter the stored program number for medium range nitrate nitrogen using AccuVac Ampuls.

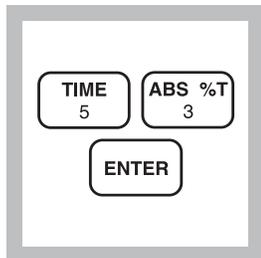
Press: **PRGM**

The display will show:

**PRGM ?**

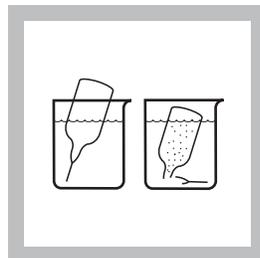
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

*Note:* Adjust the pH of stored samples before analysis.



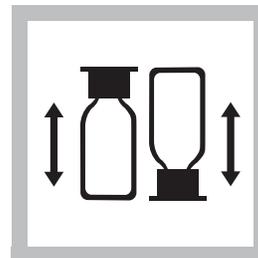
**2.** Press: **53 ENTER**  
The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

*Note:* For alternate form ( $\text{NO}_3$ ), press the **CONC** key.



**3.** Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

*Note:* Keep the tip immersed while the ampul fills. The ampul will not fill completely to allow room for mixing.



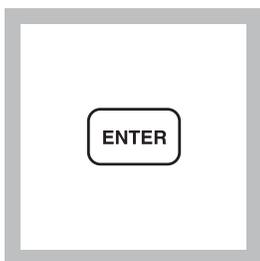
**4.** Press:

**TIMER ENTER**

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints after mixing.

*Note:* Mixing speed and technique influence color development. For most accurate results, do successive tests on a standard solution and increase or decrease the mixing time to obtain the correct result. See Accuracy Check for more information.

## NITRATE, Mid Range, continued



**5.** After the timer beeps, the display will show:

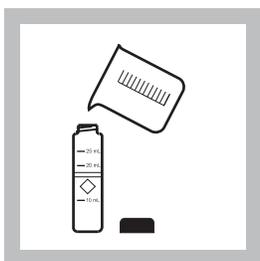
**05:00 Timer 2**

Press: **ENTER**

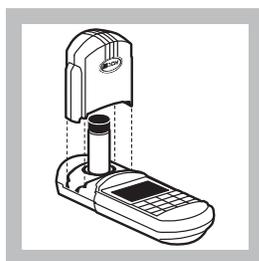
A five-minute reaction period will begin.

*Note: A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect results.*

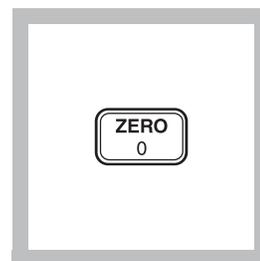
*Note: An amber color will develop if nitrate nitrogen is present.*



**6.** Fill a sample cell with at least 10 mL of sample (the blank).



**7.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

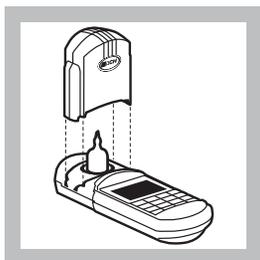


**8.** Press: **ZERO**

The cursor will move to the right, then the display will show:

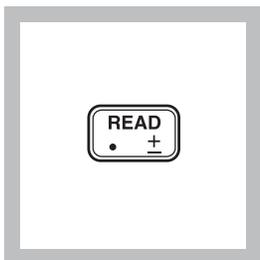
**0.0 mg/L NO<sub>3</sub>-N**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**9.** Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: Read the sample within two minutes after the timer beeps.*



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L NO<sub>3</sub>-N (or NO<sub>3</sub>) will be displayed.

*Note: Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

## NITRATE, Mid Range, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions*, (Section 1) for more information.

### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 100 mg/L  $\text{NO}_3^-$ -N.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50 mL beakers. For analysis with powder pillows, transfer only 10 mL of the solution to dry, clean sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen ( $\text{NO}_3^-$ -N) concentration should increase 0.4 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

#### Standard Solution Method

A 1.0 mg/L Nitrate Nitrogen Standard Solution is available from Hach. Use this standard in place of sample in the above procedure.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.0** to edit the

## NITRATE, Mid Range, continued

standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 3.0 mg/L nitrate nitrogen ( $\text{NO}_3^-$ -N) and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of  $\pm 0.2$  mg/L nitrate nitrogen.

In a single laboratory using a standard solution of 3.0 mg/L  $\text{NO}_3^-$ -N and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.1$  mg/L nitrate nitrogen.

#### Estimated Detection Limit

The estimated detection limit for programs 53 and 54 is 0.2 mg/L  $\text{NO}_3^-$ -N. For more information on the estimated detection limit, see *Section 1*.

### Interferences

#### Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels interfere. Compensate for nitrite interference as follows: <b>1.</b> Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. <b>2.</b> Add one drop of 30-g/L Phenol Solution to destroy the color. <b>3.</b> Proceed with Step 3. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

### Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

## NITRATE, Mid Range, continued

### Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

### REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Qty/ Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows .....	1 pillow .....	100/pkg.....	21061-69
Sample Cell, 10-20-25 mL, w/ caps .....	2 .....	6/pkg.....	24019-06

### REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul.....	1 ampul .....	25/pkg.....	25110-25
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### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL.....	1 .....	each.....	500-41
Stopper .....	1 .....	6/pkg.....	1731-06

### OPTIONAL REAGENTS

Bromine Water 30 g/L .....	29 mL*.....	2211-20
Drinking Water Standard, Inorganics, (Fe <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , PO <sub>4</sub> <sup>3-</sup> ) .....	500 mL.....	28330-49
Nitrate Nitrogen Standard Solution, 1.0 mg/L as NO <sub>3</sub> <sup>-</sup> -N.....	500 mL.....	2046-49
Nitrate Nitrogen Standard Solution, 100 mg/L as NO <sub>3</sub> <sup>-</sup> -N.....	500 mL.....	1947-49
Nitrate Nitrogen Standard Solution, PourRite Ampule, 100 mg/L as NO <sub>3</sub> <sup>-</sup> -N, 2 mL .....	20/pkg.....	1947-20
Phenol Solution, 30 g/L .....	29 mL.....	2112-20
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL SCDB*.....	2450-26
Sulfuric Acid, ACS .....	500 mL* .....	979-49
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

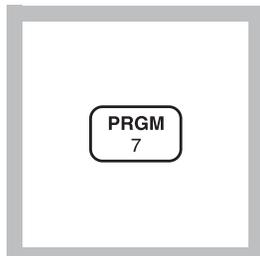
AccuVac Snapper Kit.....	each.....	24052-00
Cylinder, graduated, mixing, 25 mL.....	each.....	20886-40
Dropper, for 1-oz bottle .....	each.....	2258-00
pH Paper, 1 to 11 pH units.....	5 rolls/pkg.....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> <b>I</b> , portable, with electrode .....	each.....	51700-10
Pipet Filler, safety bulb .....	each.....	14651-00
Pipet, serological, 2 mL .....	each.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
PourRite Ampule Breaker.....	each.....	24846-00

\* Contact Hach for larger sizes.

## NITRATE, Low Range (0 to 0.50 mg/L NO<sub>3</sub><sup>-</sup>-N)

For water, wastewater and seawater\*

### Cadmium Reduction Method



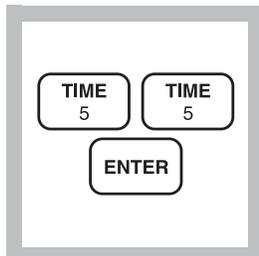
**1.** Enter the stored program number for low range nitrate nitrogen (NO<sub>3</sub><sup>-</sup>-N).

Press: **PRGM**

The display will show:

**PRGM ?**

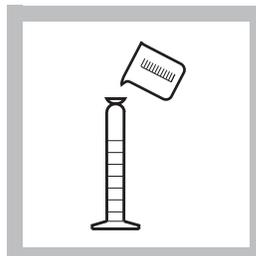
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



**2.** Press: **55 ENTER**

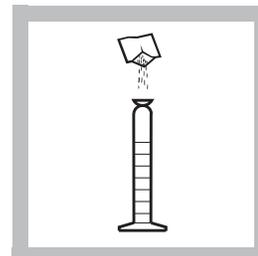
The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

*Note:* For alternate forms (NO<sub>3</sub>), press the **CONC** key.



**3.** Fill a 25-mL graduated mixing cylinder to the 15-mL mark with sample.

*Note:* Adjust the pH of stored samples before analysis.

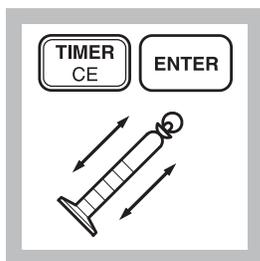


**4.** Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.

*Note:* It is necessary to remove **all** the powder from the foil pillow. Tap the pillow until no more powder pours out. Be sure to remove powder from the corners of the pillow.

\* Seawater requires a manual calibration; see Interferences.

## NITRATE, Low Range, continued

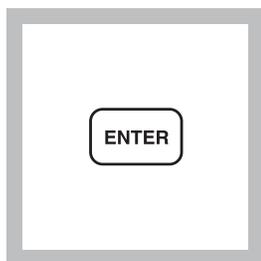


5. Press:

**TIMER ENTER**

A 3-minute reaction period will begin. Shake the cylinder vigorously throughout this three minute period.

*Note: Shaking time and technique influence color development. For most accurate results, analyze a standard solution several times and adjust the shaking time to obtain the correct result.*

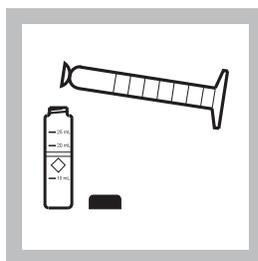


6. When the timer beeps, the display will show: **2:00 TIMER 2**

Press: **ENTER**

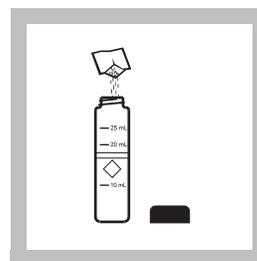
A 2-minute reaction period will begin.

*Note: A deposit will remain after the powder dissolves and will not affect results.*



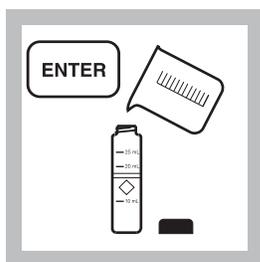
7. When the timer beeps, pour 10 mL of the sample into a sample cell.

*Note: Do not transfer any cadmium particles.*



8. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and shake gently for 30 seconds.

*Note: A pink color will form if nitrate is present.*

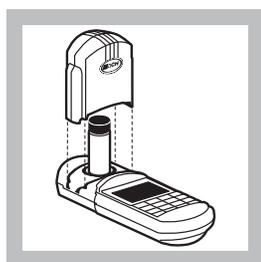


9. The display will show: **15:00 TIMER 3**

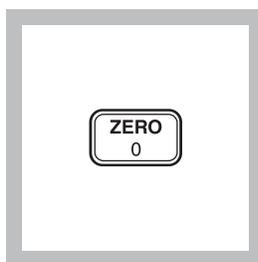
Press: **ENTER**

A 15-minute reaction period will begin.

Fill another sample cell (the blank) with 10 mL of sample.



10. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

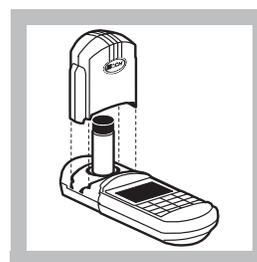


11. Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L NO3-N**

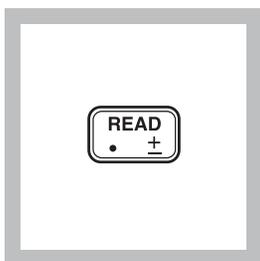
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



12. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

## NITRATE, Low Range, continued

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### 13. Press: **READ**

The cursor will move to the right, then the result in mg/L NO<sub>3</sub><sup>-</sup>-N (or alternate form) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

*Note:* Rinse the sample cell and cylinder immediately after use to remove all cadmium particles.

*Note:* See Pollution Prevention and Waste Management for proper disposal of cadmium.

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct the test result for volume additions; see *Correction for Volume Additions* (Section 1) for more information.

## NITRATE, Low Range, continued

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### Accuracy Check

#### Standard additions Method

- a) Fill three 25-mL graduated mixing cylinders with 15 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 12.0 mg/L  $\text{NO}_3^-$ -N.
- c) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- d) Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.08 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

#### Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of a 10.0 mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water. Use this standard in place of sample in Step 3.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 0.20-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **0.20** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust feature is entered. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.25 mg/L nitrate nitrogen ( $\text{NO}_3^-$ -N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.03$  mg/L nitrate nitrogen.

## NITRATE, Low Range, continued

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### Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO<sub>3</sub><sup>-</sup>-N. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Calcium	100 mg/L
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels: This method measures both the nitrate and nitrite in the sample. If nitrite is present, the nitrite nitrogen test Program 60 should be done on the sample. Pretreat the nitrate nitrogen sample with the following pretreatment. Then subtract the amount of nitrite found from the results of the LR nitrate nitrogen test using the pretreated sample. <b>1.</b> Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Mix after each drop. <b>2.</b> Add one drop of 30-g/L Phenol Solution to destroy the yellow color. <b>3.</b> Proceed with the LR Nitrate procedure.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

### Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to chromotropic acid to form a pink-colored product.

### Pollution Prevention and Waste Management

NitaVer 6 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

## NITRATE, Low Range, continued

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### REQUIRED REAGENTS

Low Range Nitrate Reagent Set (100 tests)..... 24298-00  
Includes: (1) 21071-69, (1) 21072-49

Description	Quantity Required		Unit	Cat. No.
	Per Test			
NitriVer 3 Nitrite Reagent Powder Pillows.....	1 pillow		100/pkg.....	21071-69
NitraVer 6 Nitrate Reagent Powder Pillows .....	1 pillow		100/pkg.....	21072-49

### REQUIRED APPARATUS

Cylinder, graduated, mixing, 25 mL..... 1..... each..... 1896-40  
Sample Cell, 10-20-25 mL, w/ cap..... 2..... 6/pkg..... 24019-06

### OPTIONAL REAGENTS

Description		Unit	Cat. No.
Bromine Water, 30 g/L.....		29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as NO <sub>3</sub> <sup>-</sup> -N.....		500 mL.....	307-49
Nitrate Nitrogen Standard Solution, Voluette ampule, 12 mg/L as NO <sub>3</sub> <sup>-</sup> -N, 10 mL .....		16/pkg.....	14333-10
Phenol Solution, 30 g/L .....		29 mL.....	2112-20
Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20.....		each.....	2268-00
Sodium Hydroxide Standard Solution, 5.0 N .....		50 mL* SCDB.....	2450-26
Sulfuric Acid, ACS .....		500 mL*.....	979-49
Water, deionized.....		4 L.....	272-56

### OPTIONAL APPARATUS

Ampule Breaker..... each..... 21968-00  
Dropper, for 29-mL bottle..... each..... 2258-00  
Flask, volumetric, Class A, 100 mL .....  | each..... | 14574-42 |  || pH Indicator Paper, 1 to 11 pH ..... |  | 5-roll/pkg..... | 391-33 |  |
pH Meter, *sensio*<sup>TM</sup>*1*, portable, with electrode .....		each.....	51700-10	
Pipet, serological, 2 mL .....		each.....	532-36	
Pipet, TenSette, 0.1 to 1.0 mL.....		each.....	19700-01	
Pipet Tips, for 19700-01 TenSette Pipet .....		50/pkg.....	21856-96	
Pipet Tips, for 19700-01 TenSette Pipet .....		1000/pkg.....	21856-28	
Pipet, volumetric, Class A, 2.00 mL.....		each.....	14515-36	
Pipet Filler, safety bulb .....		each.....	14651-00	
Thermometer, -20 to 110 °C.....		each.....	26357-02	

Nitrate at these levels can also be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 23488-00).

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

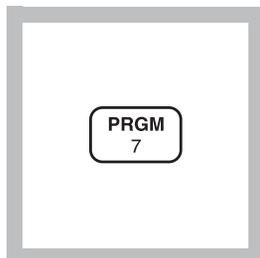
Outside the U.S.A.—Contact the Hach office or distributor serving you.

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\* Contact Hach for larger sizes

**NITRATE, High Range, Test 'N Tube (0 to 30.0 mg/L NO<sub>3</sub><sup>-</sup>-N)****Chromotropic Acid Method**

For water and wastewater



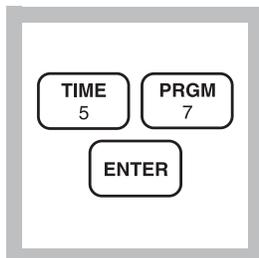
**1.** Enter the stored program number for Test 'N Tube nitrate nitrogen (NO<sub>3</sub><sup>-</sup>-N).

Press: **PRGM**

The display will show:

**PRGM ?**

*Note: If samples cannot be analyzed immediately, see Sampling and Storage on page 331.*



**2.** Press: **57 ENTER**

The display will show **mg/L, NO<sub>3</sub>-N** and the **ZERO** icon.

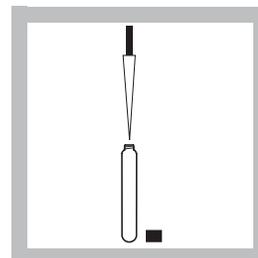
*Note: For alternate forms (NO<sub>3</sub>) press the **CONC** key.*



**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: For proof of accuracy, use a 20 mg/L NO<sub>3</sub><sup>-</sup>-N standard in place of the sample.*

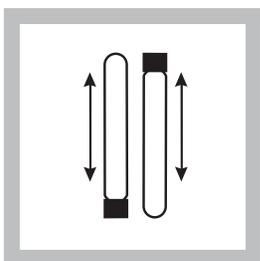
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**4.** Remove the cap from a Nitrate Pretreatment Solution Vial and add 1 mL of sample (the blank).

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

## NITRATE, High Range, Test 'N Tube, continued



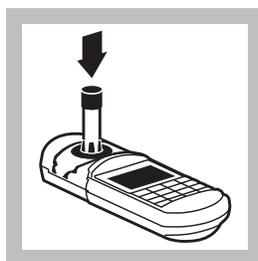
**5.** Cap the tube and invert 10 times to mix.

*Note:* This test is technique-sensitive. Low results may occur if these instructions are not followed. Hold the vial vertical with the cap up. Invert the vial so the cap points down. Wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position. Wait for all the solution to flow to the vial bottom. This process equals 1 inversion. Do this 10 times.



**6.** Clean the outside of the vial with a towel.

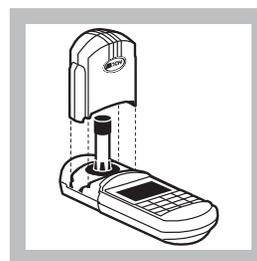
*Note:* Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.



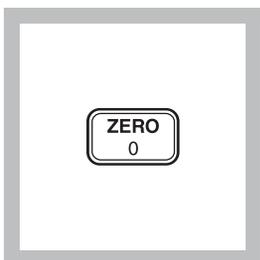
**7.** Place the blank in the vial adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**8.** Cover the vial tightly with the instrument cap.

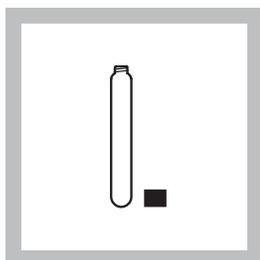


**9.** Press: **ZERO**

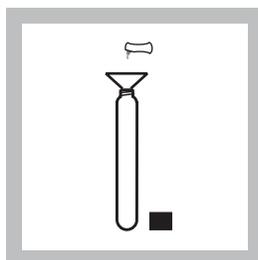
The cursor will move to the right, then the display will show:

**0.0 mg/L NO<sub>3</sub>-N**

*Note:* If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



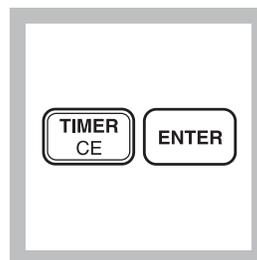
**10.** Remove the vial from the instrument. Remove the cap from the vial.



**11.** Using a funnel, add the contents of one NitraVer X Reagent B Powder Pillow to the vial. Cap. Invert 10 times to mix (this will be the prepared sample).

*Note:* See Step 5 for inversion instructions

*Note:* Some solid matter will not dissolve.



**12.** Press:

**TIMER ENTER**

A five-minute reaction period will begin. Do not invert the vial again.

*Note:* A yellow color will develop if nitrate nitrogen is present.

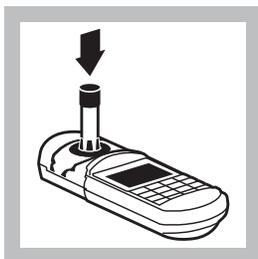
*Note:* Complete Steps 13-16 within five minutes after the timer beeps.

## NITRATE, High Range, Test 'N Tube, continued

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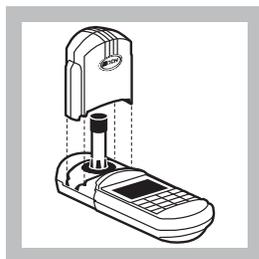


**13.** After the timer beeps, clean the outside of the vial with a damp towel and follow with a dry one to remove fingerprints and other marks.

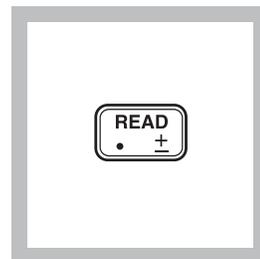


**14.** Place the prepared sample in the adapter with the Hach logo facing the front of the instrument. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**15.** Cover the vial tightly with the instrument cap.



**16.** Press: **READ**  
The cursor will move to the right, then the result in mg/L nitrate nitrogen ( $\text{NO}_3\text{-N}$ ) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1 for more information.

## NITRATE, High Range, Test 'N Tube, continued

---

### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a fresh High Range Nitrate Nitrogen Voluette Ampule Standard, 500 mg/L  $\text{NO}_3\text{-N}$ .
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to the three mixing cylinders, respectively. Mix each thoroughly.
- d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the spiked sample in each test. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

#### Standard Solution Method

To test accuracy, prepare a 20.0 mg/L nitrate nitrogen standard solution by pipetting 2.00 mL of a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L  $\text{NO}_3\text{-N}$ , into a 50 mL Class A volumetric flask. Dilute to the line with deionized water. Substitute this standard for the sample and perform the test as described in the procedure.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 25.0 mg/L nitrate nitrogen ( $\text{NO}_3\text{-N}$ ) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L  $\text{NO}_3\text{-N}$ .

#### Estimated Detection Limit

The estimated detection limit for program 57 is 0.3 mg/L  $\text{NO}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

## NITRATE, High Range, Test 'N Tube, continued

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### Interferences

Interfering Substance	Interference Level
Barium	A negative interference at concentrations greater than 1 mg/L.
Chloride	Does not interfere below 1000 mg/L.
Hardness	Does not interfere.
Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400 mg of urea (one full 0.5 g Hach measuring spoon) to 10 mL of sample. Swirl to dissolve. Proceed with the nitrate test as usual.

### Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

## NITRATE, High Range, Test 'N Tube, continued

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### REQUIRED REAGENTS

	<b>Cat. No.</b>
NitraVer X Nitrate, High Range Test 'N Tube Reagent Set (50 tests).....	26053-45
Includes: (1) 26055-46, (1) 272-42, *(50) Nitrate Pretreatment Solution Vials	

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Nitrate Pretreatment Solution Vials .....	1 .....		50/pkg.....	*
NitraVer X Reagent B Powder Pillows .....	1 .....		50/pkg.....	26055-46

### REQUIRED APPARATUS

COD Vial Adapter .....	1 .....	each.....	48464-00
Funnel, micro .....	1 .....	each.....	25843-35
Pipet, TenSette, 0.1 to 1.0 mL.....	1 .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	varies .....	50/pkg.....	21856-96
Test Tube Rack .....	1-3 .....	each.....	18641-00

### OPTIONAL REAGENTS

Nitrate-Nitrogen Standard Solution, Voluette				
Ampules, 500 mg/L N .....	16/pkg.....		14260-10	
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL .....		2450-26	
Sulfuric Acid, ACS, concentrated.....	500 mL.....		979-49	
Urea, ACS .....	100 g.....		11237-26	
Water, deionized.....	4 L.....		272-56	

### OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....	21968-00
Cylinder, graduated, mixing, 25-mL (3 required).....	each.....	26363-40
Flask, volumetric, Class A, 50 mL .....	each.....	14574-41
pH Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
Pipet, volumetric, Class A, 2 mL.....	each.....	14515-36
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
Spoon, measuring, 0.5 g.....	each.....	907-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

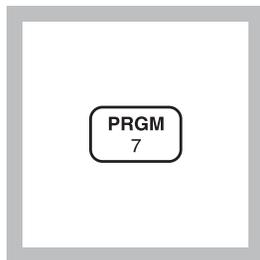
Outside the U.S.A.—Contact the Hach office or distributor serving you.

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\* Not available separately.

**NITRITE, High Range (0 to 150 mg/L NO<sub>2</sub><sup>-</sup>)**

For water and wastewater

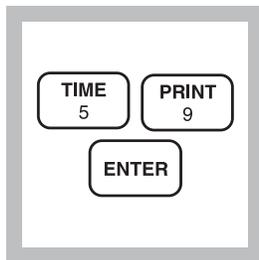
**Ferrous Sulfate Method\***

1. Enter the stored program number for high range nitrite (NO<sub>2</sub><sup>-</sup>).

Press: **PRGM**

The display will show:  
**PRGM ?**

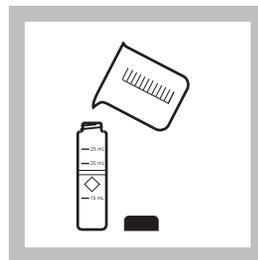
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



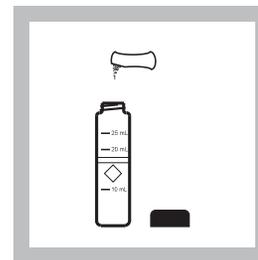
2. Press: **59 ENTER**

The display will show **mg/L, NO<sub>2</sub>** and the **ZERO** icon.

*Note: For alternate forms (NO<sub>2</sub>-N, NaNO<sub>2</sub>), press the CONC key.*



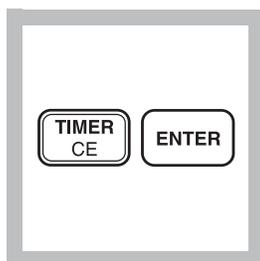
3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one NitriVer 2 Nitrite Reagent Powder Pillow. Cap the cell and invert 5-7 times to mix (the prepared sample).

*Note: A greenish-brown color will develop if nitrite is present.*

*Note: Avoid excessive mixing or low results may occur. Accuracy is not affected by undissolved powder.*

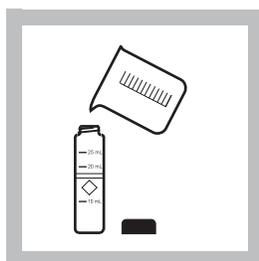


5. Press:

**TIMER ENTER**

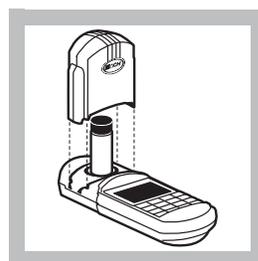
A ten-minute reaction period will begin.

Do not move or disturb the sample cell during this reaction period.

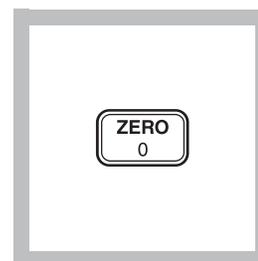


6. Fill another sample cell with 10 mL of sample (the blank). Clean the outside of the cells with a towel.

*Note: Wiping with a damp towel, followed by a dry one, removes fingerprints and other marks.*



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

The cursor will move to the right, then the display will show:

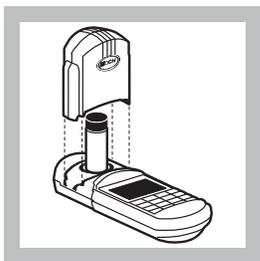
**0 mg/L NO<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

\* Adapted from McAlpine, R. and Soule, B., *Qualitative Chemical Analysis*, New York, 476,575 (1933)

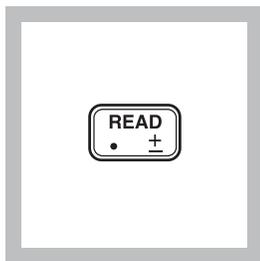
## NITRITE, High Range, continued

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**9.** After the timer beeps, gently invert the prepared sample twice. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: Avoid excessive mixing or low results may occur.*



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. If prompt analysis is impossible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 48 hours. Warm to room temperature before running the test. Do not use acid preservatives. Remove suspended solids by filtration.

### Accuracy Check

#### Standard Solution Method

Dissolve 0.150 grams of fresh sodium nitrite and dilute to 1000 mL with deionized water to prepare a 100 mg/L nitrite standard solution. Prepare this solution daily.

Alternatively, make a dilution of a fresh Hach Nitrite Standard Solution, 821 mg/L NO<sub>2</sub><sup>-</sup> (250 mg/L NO<sub>2</sub><sup>-</sup>-N) using Class A glassware. Dilute 10 mL of this standard to 100 mL with deionized water to give an 82 mg/L nitrite standard. Prepare this solution just before use. Using this solution as the sample, perform the nitrite procedure as described above.

## NITRITE, High Range, continued

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### Method Performance

#### Precision

In a single laboratory using a standard solution of 123 mg/L nitrite and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 1$  mg/L nitrite.

#### Estimated Detection Limit

The estimated detection limit for program 59 is 2 mg/L  $\text{NO}_2^-$ . For more information on the estimated detection limit, see *Section 1*.

### Interferences

This test does not measure nitrates nor is it applicable to glycol based samples. Dilute glycol based samples and follow the Low Range Nitrite Procedure.

### Summary of Method

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present.

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### REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
NitriVer 2 Nitrite Reagent Powder Pillows .....	1	pillow.....	100/pkg .....	21075-69
Sample cell, 10-20-25, w/ cap.....	2	.....	6/pkg .....	24019-06

### OPTIONAL REAGENTS

Nitrite Standard Solution, 821 mg/L $\text{NO}_2^-$ (250 mg/L $\text{NO}_2^-$ -N).....	500	mL .....	23402-49
Sodium Nitrite, ACS .....	454	g .....	2452-01
Water, deionized .....	4	L .....	272-56

### OPTIONAL APPARATUS

Balance, analytical, 110 V, Acculab UI Series, 120 g.....	each .....	26947-00
Flask, volumetric, 1000 mL .....	each .....	14547-53
Flask, volumetric, 100 mL, Class A.....	each .....	14574-42
Pipet, volumetric, 10.00 mL, Class A .....	each .....	14515-38
Pipet Filler, safety bulb .....	each .....	14651-00

### *For Technical Assistance, Price and Ordering*

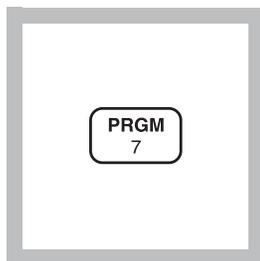
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**NITRITE, Low Range (0 to 0.350 mg/L NO<sub>2</sub><sup>-</sup>-N)** For water, wastewater, seawater

**Diazotization Method\*** (Powder Pillows or AccuVac Ampuls);  
USEPA approved for reporting wastewater and drinking water analyses.



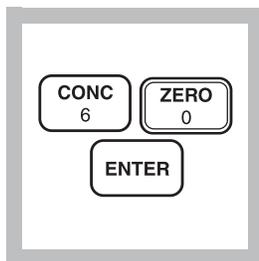
**1.** Enter the stored program number for nitrite nitrogen (NO<sub>2</sub><sup>-</sup>-N), powder pillows.

Press: **PRGM**

The display will show:

**PRGM ?**

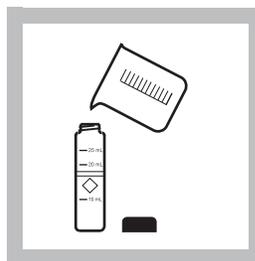
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



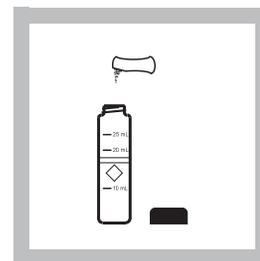
**2.** Press: **60 ENTER**

The display will show **mg/L, NO<sub>2</sub>-N** and the **ZERO** icon.

*Note: For alternate forms (NO<sub>2</sub><sup>-</sup>, NaNO<sub>2</sub>), press the **CONC** key.*



**3.** Fill a sample cell with 10 mL of sample.

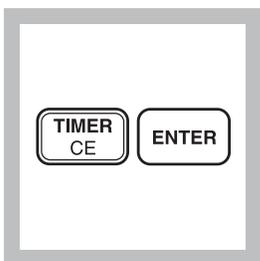


**4.** Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell. Cap the cell and shake to dissolve.

*Note: Accuracy is not affected by undissolved powder.*

\* Federal Register, 44(85) 25505 (May 1, 1979)

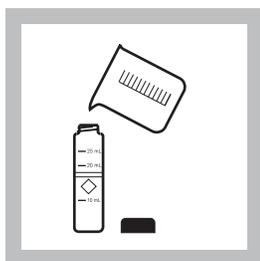
## NITRITE, Low Range, continued



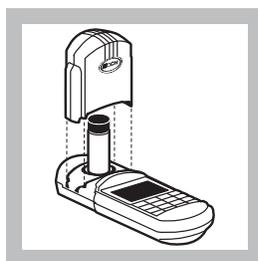
5. Press: **TIMER ENTER**

A 15-minute reaction period will begin.

*Note: A pink color will develop if nitrite is present.*

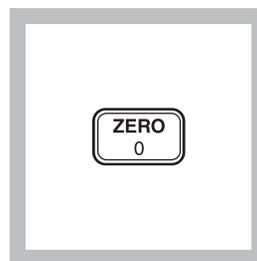


6. When the timer beeps, fill an empty sample cell with 10 mL of sample (the blank).



7. Wipe the outside of the sample cell with a towel. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note: Wiping with a damp cloth, followed by a dry pme, removes fingerprints and other marks.*

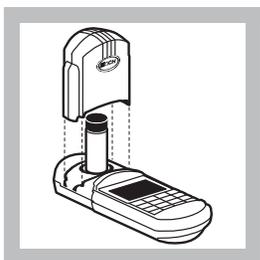


8. Press: **ZERO**

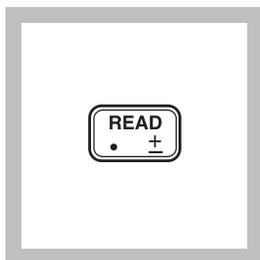
The cursor will move to the right, then the display will show:

**0.000 mg/L NO<sub>2</sub>-N**

*Note: If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*



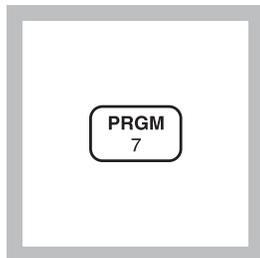
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**
- The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

## NITRITE, Low Range, continued

### Using AccuVac Ampuls



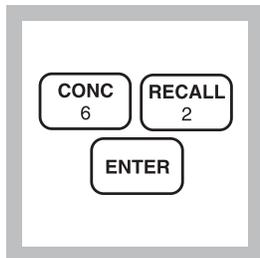
1. Enter the stored program number for nitrite nitrogen ( $\text{NO}_2^-$ -N), AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

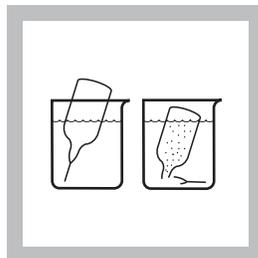
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **62 ENTER**

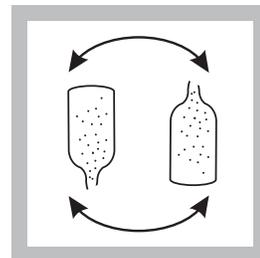
The display will show **mg/L, NO<sub>2</sub>-N** and the **ZERO** icon.

*Note: For alternate forms ( $\text{NO}_2^-$ ,  $\text{NaNO}_2$ ), press the **CONC** key.*



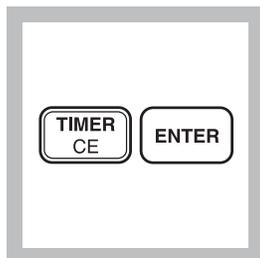
3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitriVer 3 Nitrite AccuVac Ampul with the sample.

*Note: Keep the tip immersed while the ampul fills completely.*



4. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

*Note: Accuracy is not affected by undissolved powder.*



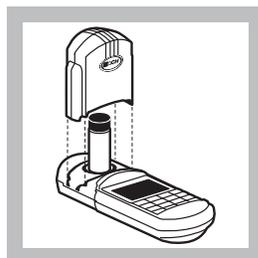
5. Press: **TIMER ENTER**

A 15-minute reaction period will begin.

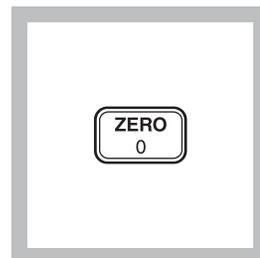
*Note: A pink color will develop if nitrite is present.*



6. When the timer beeps, fill a sample cell with at least 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

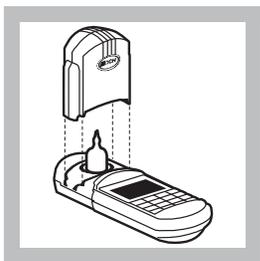
The cursor will move to the right, then the display will show:

**0.000 mg/L NO<sub>2</sub>-N**

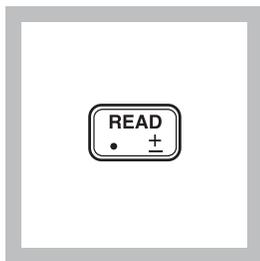
*Note: If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*

## NITRITE, Low Range, continued

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**9.** Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L nitrite nitrogen will be displayed.

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove the suspended solids by filtration.

### Accuracy Check

#### Standard Solution Method

Pipet 5.00 mL of a fresh 250 mg/L  $\text{NO}_2^-$ -N standard into a 250.0 mL volumetric flask. Dilute to the mark with deionized water. This makes a 5.00-mg/L intermediate standard. To prepare a 0.100-mg/L  $\text{NO}_2^-$ -N standard solution, dilute 10.00 mL of the 5.00-mg/L intermediate standard to 500 mL in a volumetric flask. Prepare this solution immediately before use.

Run the test using the 0.100 mg/L  $\text{NO}_2^-$ -N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L  $\text{NO}_2^-$ -N.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.001$  mg/L  $\text{NO}_2^-$ -N for the powder pillow method and  $\pm 0.003$  mg/L  $\text{NO}_2^-$ -N for the AccuVac method.

## NITRITE, Low Range, continued

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### Estimated Detection Limit

The estimated detection limit for programs 60 and 62 is 0.005 mg/L NO<sub>2</sub><sup>-</sup>-N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels
Antimonous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

### Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

## NITRITE, Low Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
NitriVer 3 Nitrite Reagent Powder Pillows.....	1 pillow.....	100/pkg.....	21071-69
or			
NitriVer 3 Nitrite Reagent AccuVac Ampuls.....	1 ampul.....	25/pkg.....	25120-25

### REQUIRED APPARATUS

Beaker, 50 mL (for AccuVac procedure).....	1.....	each.....	500-41H
or			
Sample Cells, 10-20-25 mL (powder pillow procedure).....	2.....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO <sub>2</sub> <sup>-</sup> -N.....	500 mL.....	23402-49
Water, deionized.....	4 L.....	272-56

### OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit.....	each.....	24052-00
Flask, volumetric, 250 mL.....	each.....	14574-46
Flask, volumetric, 500 mL.....	each.....	14574-49
Pipet, serological, 10 mL.....	each.....	532-38
Pipet, TenSette, 1 to 10 mL.....	each.....	19700-01
Pipet Tips for 19700-01 TenSette Pipet.....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet.....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 5.00 mL.....	each.....	14515-37
Pipet, volumetric, Class A, 10.00 mL.....	each.....	14515-38
Pipet Filler, safety bulb.....	each.....	14651-00
Thermometer, -20 to 110 °C.....	each.....	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

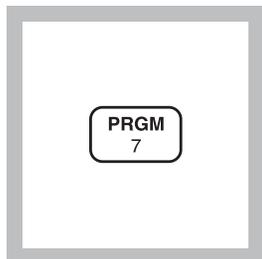
Outside the U.S.A.—Contact the Hach office or distributor serving you.

## NITRITE, Low Range, Test 'N Tube (0–0.500 mg/L NO<sub>2</sub><sup>-</sup>-N)

### Diazotization Method

USEPA approved for wastewater analysis\*

For water, wastewater, and seawater



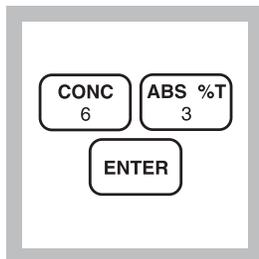
**1.** Enter the stored program number for nitrite nitrogen (NO<sub>2</sub><sup>-</sup>-N), Test 'N Tube.

Press: **PRGM**

The display will show:

**PRGM ?**

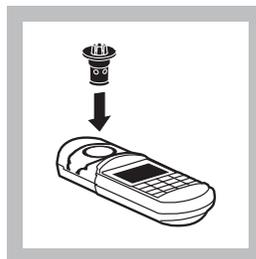
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **63 ENTER**

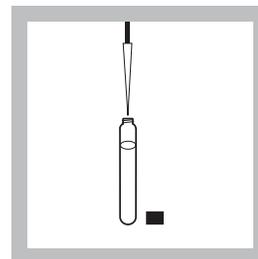
The display will show **mg/L, NO<sub>2</sub>-N** and the **ZERO** icon.

*Note: For alternate forms (NO<sub>2</sub><sup>-</sup>, NaNO<sub>2</sub>), press the **CONC** key.*

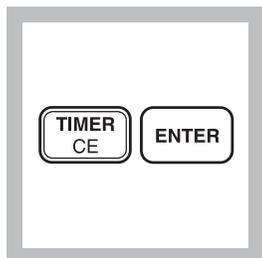


**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



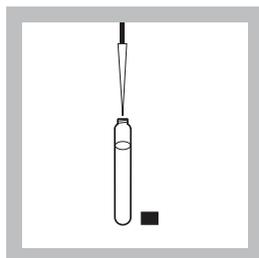
**4.** Fill a Test 'N Tube NitriVer® 3 Nitrite vial with 5 mL of sample. Cap and shake to dissolve powder. This is the prepared sample.



**5.** Press: **TIMER ENTER**

A 20-minute reaction period will begin.

*Note: A pink color will develop if nitrite is present.*

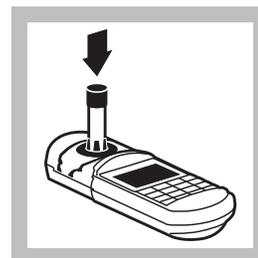


**6.** When the timer beeps, fill an empty Test 'N Tube vial with 5 mL of sample (the blank).



**7.** Clean the outside of the vials with a towel.

*Note: Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.*



**8.** Place the blank in the vial adapter.

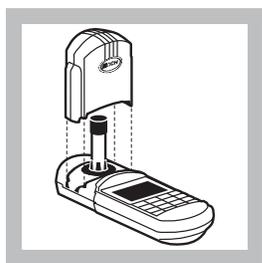
Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*

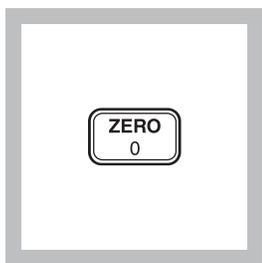
\* Federal Register, 44(85) 25505 (May 1, 1979).

## NITRITE, Test 'N Tube, continued

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**9.** Cover the sample cell tightly with the instrument cap.

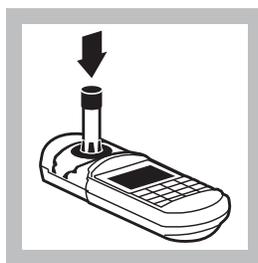


**10.** Press: **ZERO**

The cursor will move to the right, then the display will show:

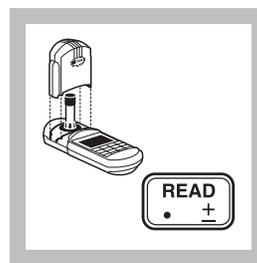
**0.000 mg/L NO<sub>2</sub>-N**

*Note:* If the reagent blank correction is on, the display may flash "limit." See Section 1.



**11.** Place the prepared sample in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**12.** Tightly cover the sample cell with the instrument cap. Press: **READ**

Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove suspended solids by filtration.

### Accuracy Check

#### Standard Solution Method

Pipet 5.00 mL of a fresh Hach standard, 250 mg/L as NO<sub>2</sub><sup>-</sup>-N into a Class A 250-mL volumetric flask. Dilute to the line with deionized water to make a 5.00-mg/L intermediate standard.

Pipet 10.00 mL of the 5.0-mg/L intermediate standard into a Class A 500-mL volumetric flask. Dilute to the line with deionized water to make a 0.100 mg/L NO<sub>2</sub><sup>-</sup>-N standard solution. Prepare immediately before use.

Run the test using the 0.100 mg/L NO<sub>2</sub><sup>-</sup>-N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO<sub>2</sub><sup>-</sup>-N.

## NITRITE, Test 'N Tube, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.004$  mg/L  $\text{NO}_2^-$ -N.

#### Estimated Detection Limit

The estimated detection limit for program 63 is 0.006 mg/L  $\text{NO}_2^-$ -N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels
Antimonous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

### Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink-colored complex directly proportional to the amount of nitrite present.

## NITRITE, Test 'N Tube, continued

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### REQUIRED REAGENTS

Description	Cat. No.
NitriVer® 3 Nitrite, Low Range Test 'N Tube Reagent Set (50 tests) .....	26083-45
Includes:	
(50) NitriVer® 3 Nitrite Test 'N Tube Vials .....	*
Vials, 6 x 100 mm, 6/pkg .....	22758-06
Caps, for 22758-06 vials, 6/pkg .....	22411-06
Deionized water, 100-mL .....	272-42

### REQUIRED APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
COD/TNT Adapter .....	1	each	48464-00
Test Tube Rack .....	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL .....	1	each	19700-10
Pipet Tips for 19700-10 TenSette Pipet .....	1	50/pkg	21997-96

### OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO <sub>2</sub> -N .....	500 mL	.....	23402-49
Water, deionized .....	4 L	.....	272-56

### OPTIONAL APPARATUS

Flask, volumetric, 250 mL .....	each	.....	14574-46
Flask, volumetric, 500 mL .....	each	.....	14574-49
Pipet, volumetric, Class A, 10.00 mL .....	each	.....	14515-38

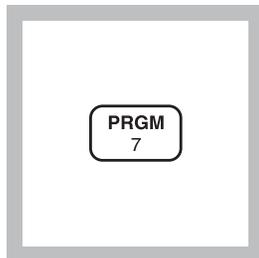
### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

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\* Not available separately.

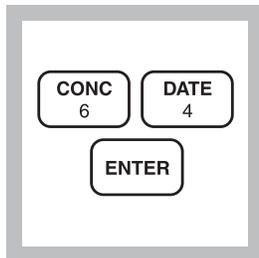
**NITROGEN, AMMONIA (0 to 0.50 mg/L NH<sub>3</sub>-N) For water, wastewater, seawater****Salicylate Method\***

**1.** Enter the stored program number for ammonia nitrogen (NH<sub>3</sub>-N).

Press: **PRGM**

The display will show:

**PRGM ?**



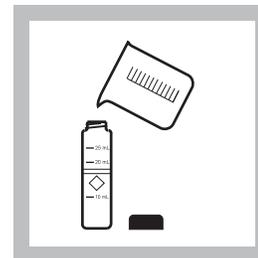
**2. Press: 64 ENTER**

The display will show **mg/L, NH<sub>3</sub>-N** and the **ZERO** icon.

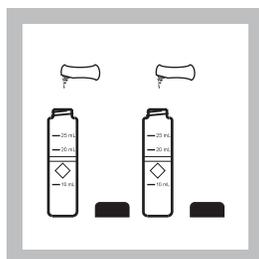
*Note: For alternate forms (NH<sub>3</sub>, NH<sub>4</sub>), press the **CONC** key.*



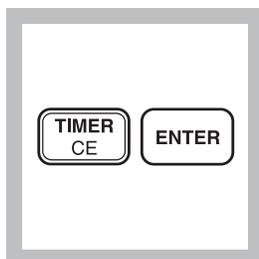
**3.** Fill a sample cell with 10 mL of deionized water (the blank).



**4.** Fill a second sample cell with 10 mL of the sample.

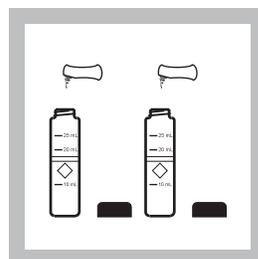


**5.** Add the contents of one Ammonia Salicylate Reagent Powder Pillow to each sample cell. Cap both cells and shake to dissolve.



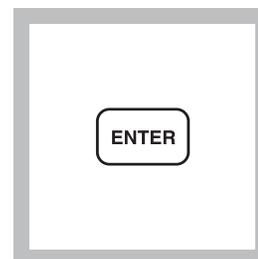
**6. Press: TIMER ENTER**

A three-minute reaction period will begin.



**7.** After the timer beeps add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each sample cell. Cap the cells and shake to dissolve the reagent.

*Note: A green color will develop if ammonia nitrogen is present.*



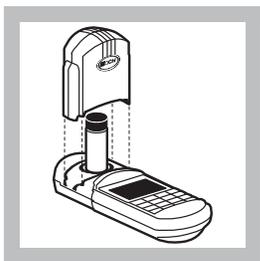
**8.** The display will show: **15:00 TIMER 2**  
Press: **ENTER**

A 15-minute reaction period will begin.

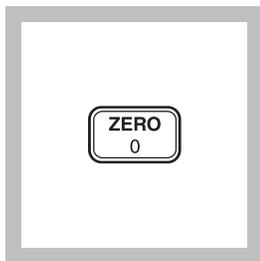
\* Adapted from Clin. Chim. Acta., 14 403 (1966)

## NITROGEN, AMMONIA, continued

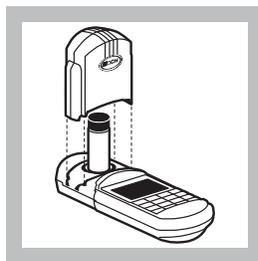
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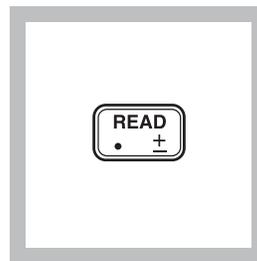
**9.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L NH<sub>3</sub>-N**



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

If chlorine is known to be present, the sample must be treated immediately with sodium thiosulfate. Add one drop of Sodium Thiosulfate Standard Solution, 0.1 N, for each 0.3 mg of chlorine present in a one liter sample.

To preserve the sample, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see *Correction for Volume Additions*, in Section 1 for more detailed information.

## NITROGEN, AMMONIA, continued

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### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL mixing cylinders with 20 mL of sample.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Ammonium Nitrogen Standard, 10 mg/L as  $\text{NH}_3\text{-N}$  to the three samples. Stopper the cylinders and mix well.
- c) Analyze a 10-mL portion of sample as described above. The ammonia nitrogen concentration should increase 0.05 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Prepare a 0.40 mg/L ammonia nitrogen standard by diluting 4.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with deionized water. Or, using the TenSette Pipet, prepare a 0.40 mg/L ammonia nitrogen standard by diluting 0.8 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as  $\text{NH}_3\text{-N}$ , to 100 mL with deionized water.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 0.40 mg/L ammonia nitrogen ( $\text{NH}_3\text{-N}$ ) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L ammonia nitrogen.

#### Estimated Detection Limit

The estimated detection limit for program 64 is 0.02 mg/L  $\text{NH}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

## NITROGEN, AMMONIA, continued

### Interferences

#### Interfering Substances and Suggested Treatments.

Interfering Substance	Interference Level and Treatments
Calcium	Greater than 1000 mg/L as CaCO <sub>3</sub>
Glycine, hydrazine	Less common. Will cause intensified colors in the prepared sample.
Iron	All levels. Correct for iron interference as follows: <ol style="list-style-type: none"><li>1. Determine the amount of iron present in the sample using one of the Total Iron procedures.</li><li>2. Prepare a deionized water sample containing the same iron concentration as the original sample. Run the procedure on this solution to determine the interference due to iron. Subtract this value from the result in Step 12 obtained on the original sample.</li></ol>
Magnesium	Greater than 6000 mg/L as CaCO <sub>3</sub>
Nitrate	Greater than 100 mg/L as NO <sub>3</sub> <sup>-</sup> -N
Nitrite	Greater than 12 mg/L as NO <sub>2</sub> <sup>-</sup> -N
Phosphate	Greater than 100 mg/L as PO <sub>4</sub> <sup>3-</sup> -P
Sulfate	Greater than 300 mg/L as SO <sub>4</sub> <sup>2-</sup>
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: <ol style="list-style-type: none"><li>1. Measure about 350 mL of sample in a 500-mL erlenmeyer flask.</li><li>2. Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.</li><li>3. Filter the sample through a folded filter paper.</li><li>4. Use the filtered solution in Step 3.</li></ol>
Turbidity, sample color	Turbidity and sample color will give erroneous high values. Samples with severe interferences require distillation. Albuminoid nitrogen samples also require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See the Optional Apparatus list.

### Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

## NITROGEN, AMMONIA, continued

### REQUIRED REAGENTS AND APPARATUS

	Cat. No.
Ammonia Nitrogen Reagent Set for 10-mL samples (100 tests) .....	26680-00
Includes: (2) 26531-99, (2) 26532-99	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Ammonia Cyanurate Reagent Powder Pillows .....	2 pillows .....	100/pkg .....	26531-99
Ammonia Salicylate Reagent Powder Pillows .....	2 pillows .....	100/pkg .....	26532-99
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06

### OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 10 mg/L as NH <sub>3</sub> -N .....	500 mL .....	153-49
Ammonia Nitrogen, PourRite Ampules, 50 mg/L as NH <sub>3</sub> -N, 2 mL .....	20/pkg .....	14791-20
Cylinder, graduated, mixing, 25 mL .....	each .....	20886-40
Sodium Hydroxide Standard Solution, 1.0 N .....	100 mL MDB .....	1045-32
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL SCDB .....	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N .....	100 mL MDB .....	323-32
Sulfide Inhibitor Reagent Powder Pillows .....	100/pkg .....	2418-99
Sulfuric Acid, concentrated, ACS .....	500 mL .....	979-49
Sulfuric Acid Standard Solution, 1.0 N .....	100 mL MDB .....	1270-32
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

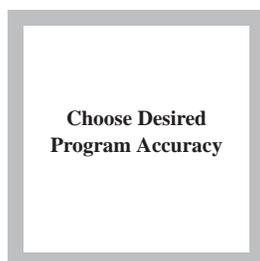
Cylinder, graduated, polypropylene, 500 mL .....	each .....	1081-49
Distillation Heater and Support Apparatus, 115 V .....	each .....	22744-00
Distillation Heater and Support Apparatus, 230 V .....	each .....	22744-02
Distillation Set, General Purpose .....	each .....	22653-00
Filter Paper, folded, 12.5 cm .....	100 .....	1894-57
Flask, Erlenmeyer, polypropylene, 500 mL .....	each .....	1082-49
Flask, volumetric, Class A, 100 mL .....	each .....	14574-42
Funnel, poly, 65 mm .....	each .....	1083-67
pH Meter, <i>sensio</i> <sup>TM</sup> <i>I</i> , portable, with electrode .....	each .....	51700-10
Pipet Filler, safety bulb .....	each .....	14651-00
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, volumetric, Class A, 2.0 mL .....	each .....	14515-36
PourRite Ampule Breaker Kit .....	each .....	24846-00
Thermometer, -20 to 110 °C .....	each .....	26357-02

### *For Technical Assistance, Price and Ordering*

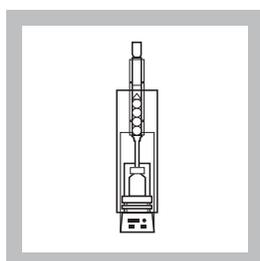
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

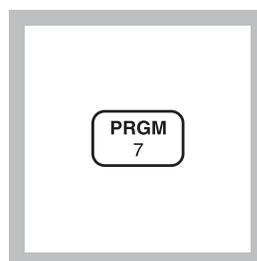


**NITROGEN, TOTAL KJELDAHL (0 to 150 mg/L)****Nessler Method\* (digestion required)****For water, wastewater and sludge**

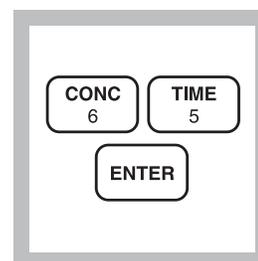
**1.** A User-Entered Calibration is necessary to obtain the most accurate results. See the User Calibration section following these steps.



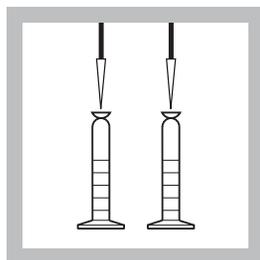
**2.** Digest the sample as described in the Digesdahl Apparatus Instruction manual. Digest an equal amount of deionized water as the blank.



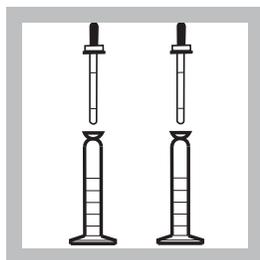
**3.** Enter the stored program number for total Kjeldahl nitrogen. Press: **PRGM**  
The display will show:  
**PRGM ?**



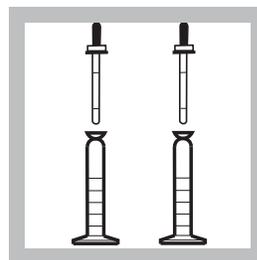
**4.** Press: **65 ENTER**  
The display will show **mg/L, TKN** and the **ZERO** icon.



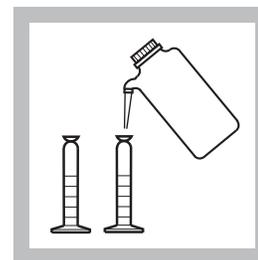
**5.** Select the appropriate analysis volume of the digested sample given in *Table 1* on page 357. Pipet the analysis volume from the sample and the digested blank into separate 25-mL mixing graduated cylinders.



**6.** Add one drop of TKN Indicator to each cylinder. Add 8.0 N KOH dropwise to each cylinder, mixing after each addition. Continue until the first apparent blue color is visible.



**7.** Add 1.0 N KOH to each cylinder, one drop at a time, mixing after each addition. Continue until the first permanent blue color appears.

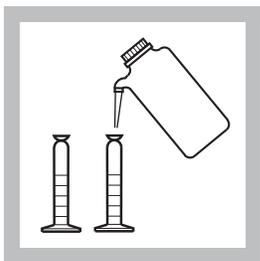


**8.** Fill both mixing cylinders to the 20-mL mark with deionized water. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Invert several times to mix.

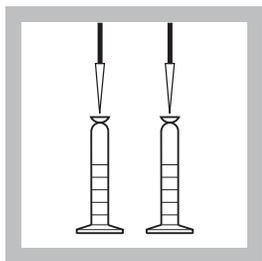
*Note: Hold the dropping bottles upright while dispensing.*

\* Adapted from: Hach et al., *Journal of Association of Official Analytical Chemists*, 70 (5) 783-787 (1987); Hach et al., *Journal of Agricultural and Food Chemistry*, 33 (6) 1117-1123 (1985); *Standard Methods for the Examination of Water and Wastewater*.

## NITROGEN, TOTAL KJELDAHL, continued

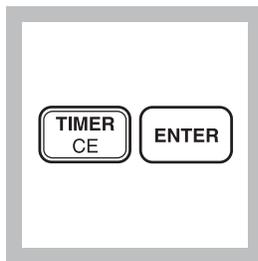


**9.** Fill both cylinders to the 25-mL mark with deionized water.

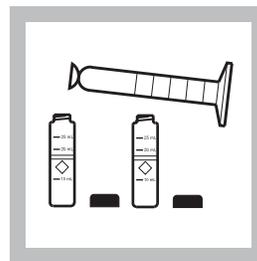


**10.** Pipet 1 mL of Nessler's Reagent to each cylinder. Stopper, invert repeatedly. The solution should not be hazy.

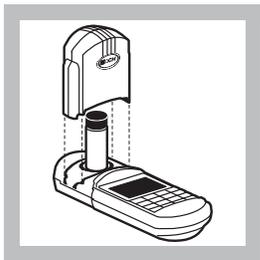
*Note:* Any haze (turbidity) will cause incorrect results.



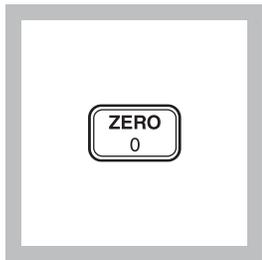
**11.** Press:  
**TIMER ENTER**  
A two-minute reaction period will begin.



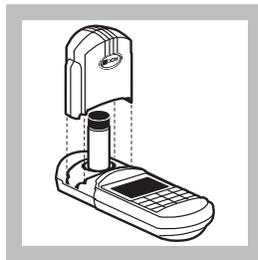
**12.** When the timer beeps, pour the contents of each cylinder into a separate labeled sample cell.



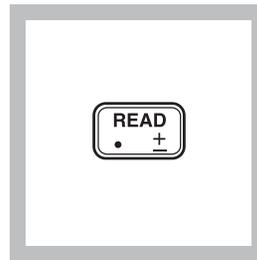
**13.** Place the blank into a cell holder. Tightly cover the sample cell with the instrument cap.



**14.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0. mg/L TKN**



**15.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**16.** Press: **READ**  
The cursor will move to the right, then the result in mg/L total Kjeldahl nitrogen will be displayed.

*Note:* Standard Adjust may be performed using a prepared ammonia standard (see Standard Adjust in Section 1).

## NITROGEN, TOTAL KJELDAHL, continued

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

**17.** Use the formula shown to calculate the final TKN value.

**Where:**

A = mg/L displayed

B = g (or mL of water) sample taken for digest

C = mL analysis volume of digested sample (step 5).

*Note: For water samples ppm TKN = mg/L TKN.*

*Note: For maximum accuracy, the reagent blank value may be determined by repeating procedure using reagents only. Subtract the reagent blank value from the reading on the display.*

**Table 1 Analysis Volumes Based on Concentration**

<b>AQUEOUS SAMPLES</b> (Solutions of suspensions in water- less than 1% solids)	
<b>Expected Nitrogen Concentration (mg/L)</b>	<b>Analysis Volume (mL)</b>
0.5-28	10.00
2-112	5.00
11-560	2.00
45-2250	1.00
425-22500	0.50
<b>DRY SAMPLES</b>	
<b>Expected Nitrogen Concentration (mg/L)</b>	<b>Analysis Volume (mL)</b>
42-2200	10.0
106-5600	5.00
350-18000	2.00
1000-56000	1.00
4200-220000	0.50
<b>OILS AND FATS</b>	
<b>Expected Nitrogen Concentration (mg/L)</b>	<b>Analysis Volume (mL)</b>
85-4500	10.0
210-11000	5.00
2100-11000	1.00

## NITROGEN, TOTAL KJELDAHL, continued

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### Sampling and Storage

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter) and cool to 4 °C. Preserved samples can be stored up to 28 days.

### Accuracy Check

#### **Kjeldahl Nitrogen Standard Method**

This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the Accuracy Check section following the procedures in the Digesdahl Digestion Apparatus Instruction Manual. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within about  $\pm 3\%$  of the value of the prepared Kjeldahl standard.

#### **Standard Solution Method (to check calibration accuracy only)**

Add one drop of TKN Indicator to each of two 25-mL graduated mixing cylinders. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0 mg/L Ammonia Nitrogen Solution. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Perform the TKN procedure as described in Steps 9 to 16. This display should show 26-27 mg/L TKN.

### User Calibration

For most accurate results, use a user-calibrated program. The Standard Adjust feature should not be used with a user-entered calibration; it will hinder performance.

A one-time setup of a program for TKN is recommended for each new lot of reagents. A new calibration may be performed for each lot of Nessler Reagent by following these instructions:

#### **Standard Preparation**

Use the following standards to make a calibration curve. See *Preparing a User-Entered Calibration Curve* on page 49, for more information and instructions. Prepare standards representing concentrations of 20, 60, 80, 100, 140 and 160 mg/L  $\text{NH}_3\text{-N}$  as follows:

## NITROGEN, TOTAL KJELDAHL, continued

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- a) Using volumetric pipets, transfer 5.0, 15.0, 20.0, 25.0, 35.0, and 40.0 mL of 100 mg/L  $\text{NH}_3\text{-N}$  standard solution into six separate 100-mL volumetric flasks. Dilute to volume with deionized water, stopper, and invert to mix.
- b) Begin at step 4 of the procedure using a 3-mL aliquot for the sample volume. Also prepare a blank solution by substituting a 3 mL aliquot of deionized water for sample in Step 4.

*Note:* Standard solutions are prepared as if a 25-mL volume was used for the digestion. Actual concentrations prepared in Step 1 are 5, 15, 20, 25, 35, and 40 mg/L  $\text{NH}_3\text{-N}$ . These represent original concentrations of 20, 60, 80, 100, 140, and 160 mg/L  $\text{NH}_3\text{-N}$ , based on the 25 to 100 mL dilution in the digestion.

### User Entered Calibration Settings For TKN

Program # = 101 to 105  
Wavelength = 420 nm  
Resolution = 0 mg/L

### Method Performance

#### Precision

In a single laboratory using a standard solution of 64 mg/L TKN and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 1.0$  mg/L TKN.

#### Estimated Detection Limit

The estimated detection limit for program 65 is 2 mg/L TKN. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

“Total Kjeldahl Nitrogen” (also called crude protein) refers to the combination of ammonia and organic nitrogen. Organically-bound in the trinegative state, it is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified nessler method test. The Mineral Stabilizer complexes calcium and magnesium. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color forms, proportional to the ammonia concentration.

## NITROGEN, TOTAL KJELDAHL, continued

### Pollution Prevention And Waste Management

Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section 3 for more information on proper disposal of these materials.

### REQUIRED REAGENTS

Total Kjeldahl Nitrogen Reagent Set ..... 24953-00

Includes: (1) 21196-49, (1) 23766-26, (1) 21194-49, (1) 23765-26, (1) 282-32H,  
(1) 23144-26, (1) 979-49, (1) 22519-26

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Hydrogen Peroxide, 50% .....	20 mL.....	490 mL.....		21196-49
Mineral Stabilizer .....	6 drops.....	50 mL SCDB.....		23766-26
Nesslers Reagent.....	2 mL.....	500 mL.....		21194-49
Polyvinyl Alcohol Dispersing Agent.....	6 drops.....	50 mL SCDB.....		23765-26
Potassium Hydroxide Standard Solution, 8.0 N .....	varies .....	100 mL MDB.....		282-32H
Potassium Hydroxide Standard Solution, 1.0 N .....	varies .....	50 mL SCDB.....		23144-26
Sulfuric Acid, ACS .....	6 mL.....	500 mL.....		979-49
TKN Indicator Solution .....	2 drops.....	50 mL SCDB.....		22519-26
Water, deionized.....	varies .....	4 L.....		272-56

### REQUIRED APPARATUS

Boiling Chips, silicon carbide.....	2-3 .....	500 g.....		20557-34
Cylinder, graduated, mixing, tall-form, 25 mL.....	2 .....	each.....		20886-40
Pipet, TenSette, 0.1 to 1.0 mL.....	1 .....	each.....		19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	2 .....	50/pkg.....		21856-96
Safety Shield, for Digesdahl .....	1 .....	each.....		50040-00
Sample Cell, 10-20-25 mL, w/ cap.....	2 .....	6/pkg.....		24019-06

#### Select one based on available voltage:

Digesdahl Digestion Apparatus, 115 V .....	1 .....	each.....		23130-20
Digesdahl Digestion Apparatus, 230 V .....	1 .....	each.....		23130-21

### OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 1 mg/L NH <sub>3</sub> -N.....	500 mL.....			1891-49
Ammonia Nitrogen Standard Solution, Voluette Ampule, 150 mg/L NH <sub>3</sub> -N, 10 mL .....	16/pkg.....			21284-10
Ammonia Nitrogen Standard Solution, 100 mg/L NH <sub>3</sub> -N.....	500 mL.....			24065-49
Nitrogen Standard, Primary .....	3/set.....			22778-00

## NITROGEN, TOTAL KJELDAHL, continued

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### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit .....	each	21968-00
Balance, AccuLab Pocket Pro 250B .....	each	27969-00
Bottle, glass dispenser, 118 mL.....	each	591-00
Bottle, plastic wash, 1000 mL.....	each	620-16
Cylinder, graduated, 50 mL.....	each	508-41
Flask, volumetric, 100 mL, Class A.....	each	14574-42
Mini Grinder, 120 V .....	each	20991-00
pH Paper, 1 to 11 pH units .....	5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg	21856-28
Pipet, volumetric, Class A, 0.50 mL .....	each	14515-34
Pipet, volumetric, Class A, 1.00 mL .....	each	14515-35
Pipet, volumetric, Class A, 2.00 mL .....	each	14515-36
Pipet, volumetric, Class A, 5.00 mL .....	each	14515-37
Pipet, volumetric, Class A, 10.00 mL .....	each	14515-38
Pipet, volumetric, Class A, 15.00 mL .....	each	14515-39
Pipet, volumetric, Class A, 20.00 mL .....	each	14515-20
Pipet, volumetric, Class A, 25.00 mL .....	each	14515-40
Safety Glasses .....	each	18421-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

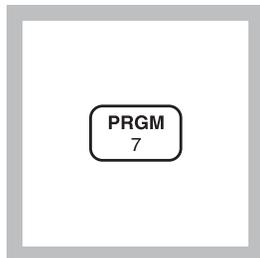
Outside the U.S.A.—Contact the Hach office or distributor serving you.



# NITROGEN, AMMONIA, Low Range, Test 'N Tube (0 to 2.50 mg/L NH<sub>3</sub>-N)

## Salicylate Method\*

For water, wastewater, and seawater

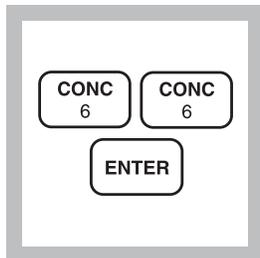


**1.** Enter the stored program number for low range nitrogen, ammonia Test 'N Tube.

Press: **PRGM**

The display will show:

**PRGM ?**



**2.** Press: **66 ENTER**

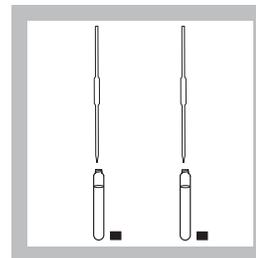
The display will show **mg/L, NH<sub>3</sub>-N** and the **ZERO** icon.

*Note: For alternate forms (NH<sub>3</sub>), press the **CONC** key.*



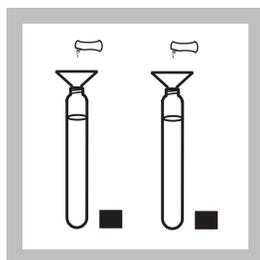
**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

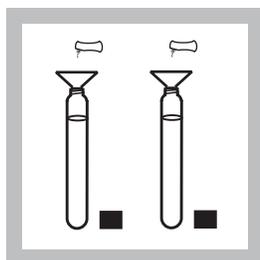


**4.** Remove the caps from 2 AmVer Diluent Reagent vials. Add 2 mL of sample to one vial (the sample). Add 2 mL of deionized water to the other vial (the blank).

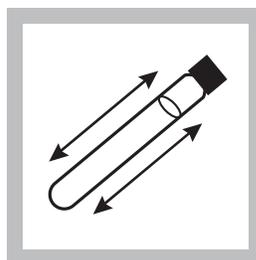
*Note: Adjust the pH of stored samples before analysis. See Interferences on page 365.*



**5.** Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow for 5 mL sample to each vial.

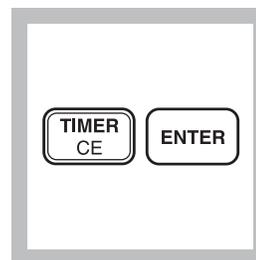


**6.** Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow for 5 mL sample to each vial.



**7.** Cap the vials tightly and shake thoroughly to dissolve the powder.

*Note: A green color will develop if ammonia is present.*



**8.** Press: **TIMER ENTER**  
A 20-minute reaction period will begin.

\* Adapted from *Clin. Chim. Acta*, 14 403 (1966).

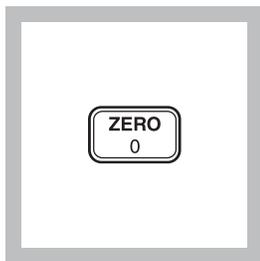
## NITROGEN, AMMONIA, Low Range, Test 'N Tube, continued

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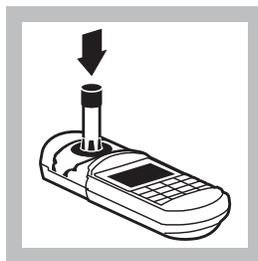


**9.** Wipe the outside of the vials with a towel. After the timer beeps, place the blank into the adapter. Tightly cover the vial with the instrument cap.

*Note: Wipe with a damp cloth followed by a dry one to remove fingerprints and other marks.*

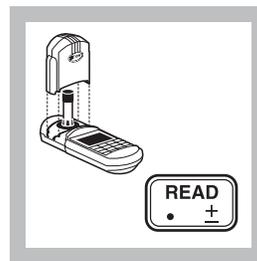


**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L NH<sub>3</sub>-N**



**11.** Place the prepared sample in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**12.** Tightly cover the sample cell with the instrument cap.  
Press: **READ**  
The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust (Adjusting the Standard Curve) on page 47).*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl<sub>2</sub> in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions. See *Correcting for Volume Additions* on page 22 for more information.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Nitrogen, Ammonia Ampule Standard Solution, 50 mg/L NH<sub>3</sub>-N.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25 mL samples. Mix thoroughly.

## NITROGEN, AMMONIA, Low Range, Test 'N Tube, continued

- c) Analyze each sample as described above. The nitrogen concentration should increase 0.20 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions, Section 1*, for more information.

### Standard Solution Method

To check accuracy, use a 1.0 mg/L Nitrogen, Ammonia Standard Solution listed under Optional Reagents. Or, dilute 1 mL of solution from a 50 mg/L Ampule Standard for Nitrogen, Ammonia to 50 mL with deionized water using a 50-mL volumetric flask.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.0 mg/L ammonia nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L  $\text{NH}_3\text{-N}$ .

#### Estimated Detection Limit

The estimated detection limit for program 66 is 0.08 mg/L  $\text{NH}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Level and Treatment
Calcium	2500 mg/L as $\text{CaCO}_3$
Iron	<ol style="list-style-type: none"><li>1. Determine the amount of iron present in the sample following one of the total iron procedures.</li><li>2. Add the same iron concentration to the deionized water in step 4. The interference will then be successfully blanked out.</li></ol>
Magnesium	5000 mg/L as $\text{CaCO}_3$
Nitrite	30 mg/L as $\text{NO}_2^- \text{-N}$
Nitrate	250 mg/L as $\text{NO}_3^- \text{-N}$
Orthophosphate	250 mg/L as $\text{PO}_4^{3-} \text{-P}$
pH	Acidic or basic samples should be adjusted to about pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Sulfate	300 mg/L as $\text{SO}_4^{2-}$

## NITROGEN, AMMONIA, Low Range, Test 'N Tube, continued

Interfering Substance	Interference Level and Treatment
Sulfide	<ol style="list-style-type: none"> <li>1. Measure about 350 mL of sample in a 500 mL erlenmeyer flask.</li> <li>2. Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.</li> <li>3. Filter the sample through a folded filter paper.</li> <li>4. Use the filtered solution in step 4.</li> </ol>
Other	Less common interferences such as <b>hydrazine</b> and <b>glycine</b> will cause intensified colors in the prepared sample. <b>Turbidity</b> and <b>color</b> will give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See Optional Apparatus at the end of this procedure.

### Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

### Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

### REQUIRED REAGENTS

**Cat. No.**

AmVer Reagent Set for Nitrogen, Ammonia, Low Range TNT (25 tests)..... 26045-45  
 Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, \* (50) AmVer Low Range Vials

Description	Quantity Required		Unit	Cat. No.
	Per Test			
AmVer Diluent Reagent, Low Range Test 'N Tube ...	2 vials	.....	50/pkg	..... *
Salicylate Reagent Powder Pillows, 5 mL sample ....	2 pillows	.....	50/pkg	..... 23952-66
Cyanurate Reagent Powder Pillows, 5 mL sample....	2 pillows	.....	50/pkg	..... 23954-66

\* Not available separately.

## NITROGEN, AMMONIA, Low Range, Test 'N Tube, continued

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### REQUIRED APPARATUS

Vial Adapter, COD .....	1 .....	each .....	48464-00
Test Tube Rack .....	1-3 .....	each .....	18641-00
Pipet, TenSette, 0-10 mL.....	1 .....	each .....	19700-10
Pipet Tips for 19700-10.....	2 .....	50/pkg .....	21997-96
Funnel, micro (for reagent addition) .....	1 .....	each .....	25843-35

### OPTIONAL REAGENTS

Nitrogen, Ammonia Standard Solution, 1.0 mg/L NH <sub>3</sub> -N .....	500 mL .....	1891-49
Nitrogen, Ammonia Standard Solution, 10 mL Voluette ampules, 50 mg/L NH <sub>3</sub> -N.....	16/pkg .....	14791-10
Nitrogen, Ammonia Standard Solution, 2 mL PourRite ampules, 50 mg/L NH <sub>3</sub> -N.....	20/pkg .....	14791-20
Hydrochloric Acid, ACS .....	500 mL .....	134-49
Sodium Hydroxide Standard Solution, 5.0 N.....	50 mL SCDB .....	2450-26
Sodium Hydroxide, 1.000 N .....	100 mL MDB .....	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N.....	100 mL MDB .....	323-32
Sulfide Inhibitor Reagent Powder Pillows .....	100/pkg .....	2418-99
Sulfuric Acid, 1.00 N .....	100 mL MDB .....	1270-32
Wastewater Effluent Standard, Inorganics (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit .....	each .....	21968-00
Cylinder, graduated, mixing, 25 mL, Class A.....	each .....	508-40
Distillation Apparatus Set .....	each .....	22653-00
Heater and Support Apparatus (for distillation), 115 Vac .....	each .....	22744-00
Heater and Support Apparatus (for distillation), 230 Vac .....	each .....	22744-02
Filter Paper, folded .....	100/box .....	1894-57
Flask, Erlenmeyer, 500 mL .....	each .....	505-49
Flask, volumetric, 50 mL, Class A.....	each .....	14547-41
Funnel, analytical (for filtering).....	each .....	1083-68
Jack, laboratory (use with distillation apparatus).....	each .....	22743-00
pH Indicator Paper, 1 to 11 pH.....	5 rolls/pkg .....	391-33
Ampule Breaker Kit, PourRite .....	each .....	24846-00
Thermometer, -20 to 110 °C, non-mercury .....	each .....	26357-02
Thermometer, -10 to 260 °C, non-mercury .....	each .....	26357-01

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

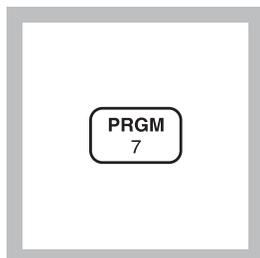


## NITROGEN, AMMONIA, High Range, Test 'N Tube

(0 to 50 mg/L NH<sub>3</sub>-N)

For water, wastewater, and seawater

### Salicylate Method\*

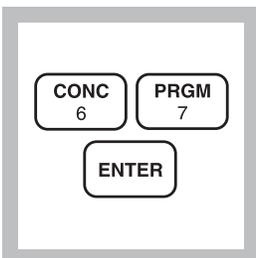


**1.** Enter the stored program number for nitrogen, ammonia, high range Test 'N Tube (NH<sub>3</sub>-N) method.

Press: **PRGM**

The display will show:

**PRGM ?**

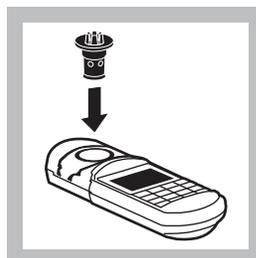


**2.** Press: **67 ENTER**

The display will show **mg/L, NH<sub>3</sub>-N** and the **ZERO** icon.

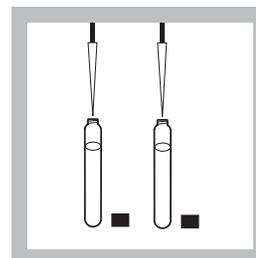
*Note: For alternate forms (NH<sub>3</sub>), press the **CONC** key.*

*Note: For proof of accuracy, use a 10-mg/L nitrogen, ammonia standard in place of the sample.*

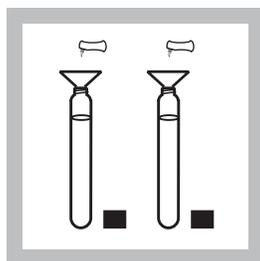


**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

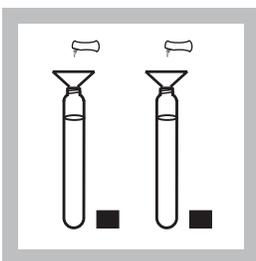
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



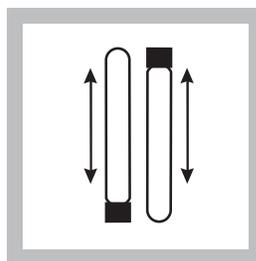
**4.** Remove the caps from 2 AmVer Diluent Reagent High Range Vials. Add 0.1 mL of sample to one vial (the sample). Add 0.1 mL of deionized water to the other (the blank).



**5.** Add the contents of 1 Ammonia Salicylate Reagent Powder Pillow for 5 mL Sample to each vial.

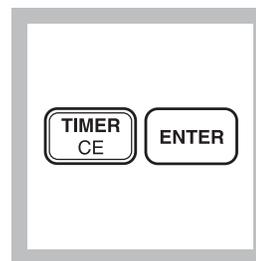


**6.** Add the contents of 1 Ammonia Cyanurate Reagent Powder Pillow for 5 mL Sample to each vial.



**7.** Cap the vials tightly and shake thoroughly to dissolve the powder.

*Note: A green color will develop if ammonia is present.*



**8.** Press:

**TIMER ENTER**

A 20-minute reaction period will begin.

\* Adapted from *Clin. Chim. Acta*, **14** 403 (1966).

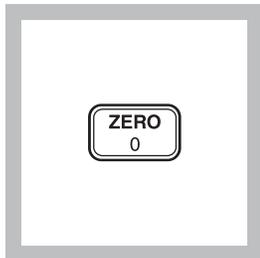
## NITROGEN, AMMONIA, High Range, Test 'N Tube, continued

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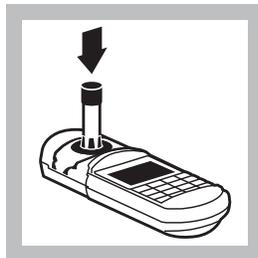
**9.** Clean the outside of the vial with a towel. After the timer beeps, place the blank into the vial adapter. Tightly cover the vial with the instrument cap.

*Note: Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



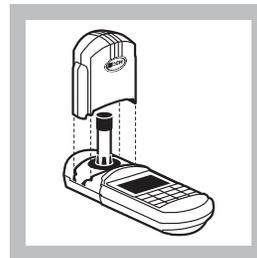
**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 mg/L NH<sub>3</sub>-N**

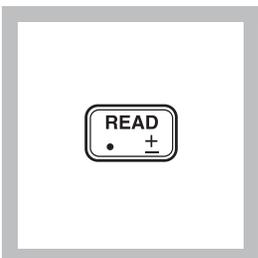


**11.** Place the prepared sample in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**12.** Tightly cover the vial with the instrument cap.



**13.** Press: **READ**

The cursor will move to the right, then the result in mg/L NH<sub>3</sub>-N will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

## NITROGEN, AMMONIA, High Range, Test 'N Tube, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L  $\text{Cl}_2$  in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions.

### Accuracy Check

#### Standard Additions Method

- a) Snap the top off an Ammonia PourRite Ampule Standard, 150 mg/L  $\text{NH}_3\text{-N}$ .
- b) Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 25-mL samples. Swirl to mix.
- c) Analyze each sample as described above. The ammonia concentration should increase approximately 1.2 mg/L  $\text{NH}_3\text{-N}$  for each 0.2 mL of standard added.
- d) If these increases do not occur, see *Standard Additions in Section 1* for more information.

#### Standard Solution Method

To check accuracy, use a 10 or 50 mg/L Nitrogen, Ammonia Standard Solution or use a Nitrogen, Ammonia Voluette Ampule Standard, 50 mg/L.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 50 mg/L ammonia nitrogen ( $\text{NH}_3\text{-N}$ ) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 5$  mg/L  $\text{NH}_3\text{-N}$ .

#### Estimated Detection Limit

The estimated detection limit for program 67 is 1 mg/L  $\text{NH}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

## NITROGEN, AMMONIA, High Range, Test 'N Tube, continued

### Interferences

The following ions may interfere when present in concentrations exceeding those listed below.

In some lab environments, airborne cross contamination of the blank is possible. Complete preparation of the blank before opening or handling any samples or standards to avoid transfer of ammonia. If sample or standard containers have already been open, move to a separate area of the lab to prepare the blank.

Substance	Concentration and Suggested Treatments
Acidic or basic samples	Adjust to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Calcium	50,000 mg/L as CaCO <sub>3</sub>
Glycine, hydrazine	Will cause intensified colors in the prepared sample.
Magnesium	300,000 mg/L as CaCO <sub>3</sub>
Iron	Eliminate iron interference as follows: <ol style="list-style-type: none"> <li>1. Determine the amount of iron present in the sample using one of the total iron procedures.</li> <li>2. Add the same iron concentration to the deionized water in step 4.</li> <li>3. The interference will then be successfully blanked out.</li> </ol>
Nitrite	600 mg/L as NO <sub>2</sub> <sup>-</sup> -N
Nitrate	5,000 mg/L as NO <sub>3</sub> <sup>-</sup> -N
Orthophosphate	5,000 mg/L as PO <sub>4</sub> <sup>3-</sup> -P
Sulfate	6,000 mg/L as SO <sub>4</sub> <sup>2-</sup>
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: <ol style="list-style-type: none"> <li>1. Measure about 350 mL of sample in a 500 mL Erlenmeyer flask.</li> <li>2. Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.</li> <li>3. Filter the sample through folded filter paper. Use the filtered solution in step 4.</li> </ol>
Turbidity and color	Give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set.

## NITROGEN, AMMONIA, High Range, Test 'N Tube, continued

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### Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a green-colored solution.

### Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheets* for information specific to the reagents used. For additional information, refer to *Section 3*.

### Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

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### REQUIRED REAGENTS

AmVer™ Reagent Set for Nitrogen, Ammonia, High Range, TNT (25 tests) .....26069-45  
Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, \* (50) AmVer HR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
AmVer™ HR Reagent Test 'N Tube™ Vials .....	2 vials .....	50/pkg .....	* .....
Ammonia Salicylate Reagent Powder Pillows .....	2 pillows .....	50/pkg .....	23952-66 .....
Ammonia Cyanurate Reagent Powder Pillows .....	2 pillows .....	50/pkg .....	23954-66 .....

### REQUIRED APPARATUS

COD/TNT Adapter .....	1 .....	each .....	48464-00 .....
Pipet, TenSette®, 0-1 mL .....	1 .....	each .....	19700-01 .....
Pipet Tips for 19700-01 .....	varies .....	50/pkg .....	21856-96 .....
Test Tube Rack .....	1-3 .....	each .....	18641-00 .....
Funnel, micro (for reagent addition) .....	1 .....	each .....	25843-35 .....

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\* Not available separately.

## NITROGEN, AMMONIA, High Range, Test 'N Tube, continued

### OPTIONAL REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrogen, Ammonia Standard Solution, 50 mg/L NH <sub>3</sub> -N.....		500 mL.....	14791-50
Nitrogen, Ammonia Standard Solution, 10 mg/L NH <sub>3</sub> -N.....		500 mL.....	153-49
Ammonia Standard Solution, PourRite™ ampules, 150 mg/L NH <sub>3</sub> -N, 2 mL.....		20/pkg.....	21284-20
Hydrochloric Acid, ACS.....		500 mL.....	134-49
Sodium Hydroxide Standard Solution, 5.0 N .....		50 mL.....	2450-26
Sodium Hydroxide Standard Solution, 1.0 N .....		100 mL.....	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N .....		100 mL.....	323-32
Sulfide Inhibitor Powder Pillows.....		100/pkg.....	2418-99
Sulfuric Acid, 1.00 N.....		100 mL MDB.....	1270-32
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> , PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....		500 mL.....	28331-49
Water, deionized.....		4 L .....	272-56

### OPTIONAL APPARATUS

Cylinder, 25 mL, graduated, mixing .....	each.....	20886-40
Distillation Apparatus Set, general purpose .....	each.....	22653-00
Heater and Support Apparatus (for distillation), 115 VAC.....	each.....	22744-00
Heater and Support Apparatus (for distillation), 230 VAC .....	each.....	22744-02
Filter Paper, folded.....	100/pkg.....	1894-57
Flask, Erlenmeyer, 500 mL.....	each.....	505-49
Funnel, analytical (for filtering).....	each.....	1083-68
Jack, laboratory (use with distillation apparatus) .....	each.....	22743-00
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg.....	391-33
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
PourRite™ Ampule Breaker .....	each.....	24846-00
Sample Cell, 10-20-25 mL, w/cap .....	6/pkg.....	24019-06

### *For Technical Assistance, Price and Ordering*

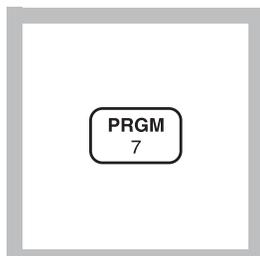
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# NITROGEN, Total Inorganic, Test 'N Tube™ (0 to 25.0 mg/L N)

## Titanium Trichloride Reduction Method Requires Centrifuge

For water, wastewater, and seawater

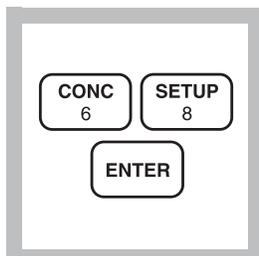


1. Enter the stored program number for Test 'N Tube Total Inorganic Nitrogen.

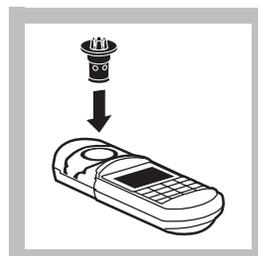
Press: **PRGM**

The display will show:

**PRGM ?**

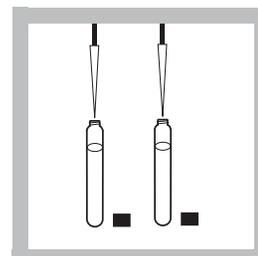


2. Press: **68 ENTER**  
The display will show **mg/L, N** and the **ZERO** icon.

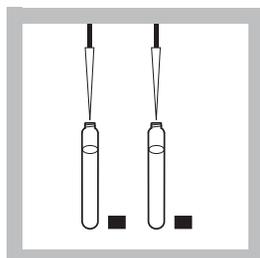


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

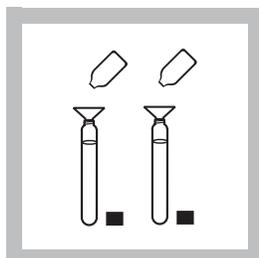
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



4. Pipet 1 mL of Total Inorganic Nitrogen Pretreatment Base Concentrate into each of 2 Total Inorganic Nitrogen Pretreatment Diluent Vials.



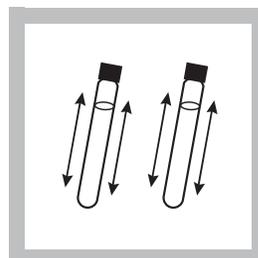
5. Pipet 1 mL of sample into 1 TIN Diluent Vial (the sample). Pipet 1 mL of deionized water into the other vial (the blank). Cap the vials and shake for 30 seconds to mix.



6. Snap the necks off two Total Inorganic Nitrogen Reductant ampules and pour the contents of one into the TIN Diluent Vial containing sample. Repeat for the second vial, the blank.

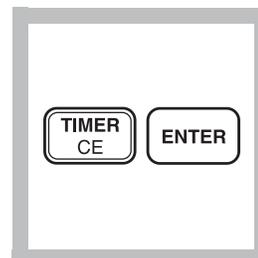
*Note: For safety, wear gloves while breaking the ampules.*

*Note: A black precipitate will form immediately.*



7. Cap the vials. Shake gently for 30 seconds to mix the reagents. Allow the vials to sit for at least one minute.

*Note: The precipitate should remain black after shaking. Excessive shaking will cause a white precipitate and low results.*

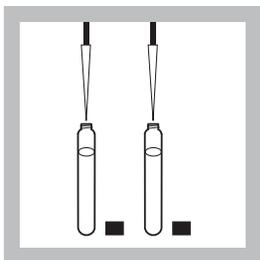


8. Centrifuge the vials for 3 minutes or until the solids settle to the bottom of the vial.

Press: **TIMER ENTER** immediately after starting the centrifuge.

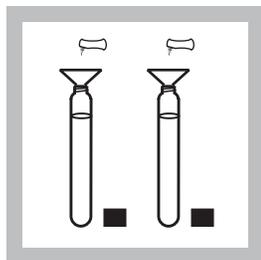
*Note: The precipitate will settle without using a centrifuge, but it may take up to 30 minutes.*

## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

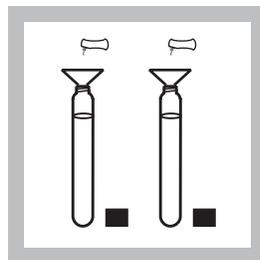


**9.** Remove the caps from 2 AmVer Diluent Reagent Test 'N Tubes for Low Range Ammonia Nitrogen. Using a pipet, add 2 mL of centrifuged sample into 1 vial. Add 2 mL of centrifuged blank to the other vial. Label the vials appropriately.

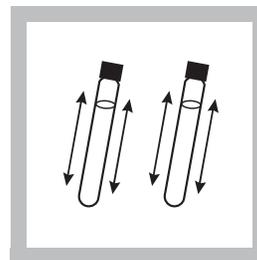
*Note: Pipet carefully to avoid disturbing the sediment.*



**10.** Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow to each vial.

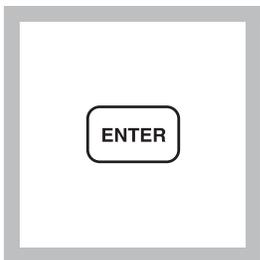


**11.** Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.

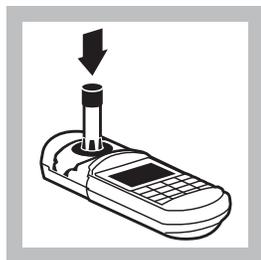


**12.** Cap the vials tightly and shake thoroughly to dissolve the powder.

*Note: A green color will develop if inorganic nitrogen is present.*

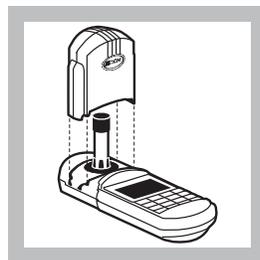


**13.** The display will show: **15:00 TIMER 2**  
Press: **ENTER**  
A 15-minute reaction period will begin.

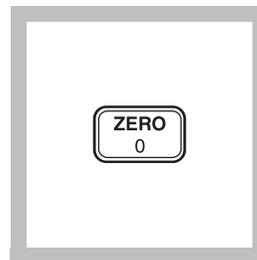


**14.** After the timer beeps, clean the outside of the vials with a towel. Place the blank in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter. Do not move the vial from side to side as this can cause errors.

*Note: Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



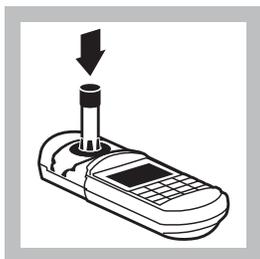
**15.** Tightly cover the sample cell with the instrument cap.



**16.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.0 mg/L N**

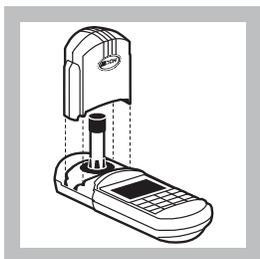
## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

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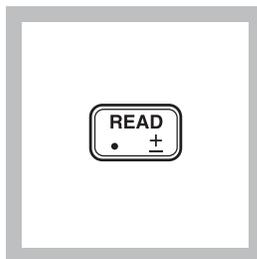


**17.** Place the prepared sample in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**18.** Tightly cover the sample cell with the instrument cap.



**19.** Press: **READ**  
The cursor will move to the right, then the result in mg/L total inorganic nitrogen will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

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### Sampling And Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

If chlorine is known to be present, add 1 drop of 0.1 N sodium thiosulfate for each 0.3 mg/L  $\text{Cl}_2$  in a 1 liter sample.

Preserve the sample by reducing the pH to 2 or less with concentrated hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

### Accuracy Check

#### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off a fresh High Range Nitrate Nitrogen PourRite Ampule Standard, 500 mg/L  $\text{NO}_3^-$ -N.
- Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to 3 25-mL mixing cylinders. Mix thoroughly.

## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

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- d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the prepared sample in Step 5. The nitrogen concentration should increase about 1.8 to 1.9 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To check accuracy, use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional Reagents. Alternatively, a 20.0 mg/L nitrate nitrogen standard can be prepared by diluting 2 mL of solution from a PourRite Ampule Standard for High Range Nitrate Nitrogen, 500 mg/L NO<sub>3</sub><sup>-</sup>-N, to 50 mL with deionized water. Substitute this standard for the sample and perform the test as described. The recovery of the standards should be about 90-95%.

## Method Performance

### Precision/Accuracy

The total inorganic nitrogen test provides an estimate of the total nitrite, nitrate, and ammonia nitrogen load in water or wastewater samples. This test is most applicable for monitoring an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test exhibits different recoveries of each of the three nitrogen species, as summarized below. This test is not recommended for quantifying only one of the three species. In that case, use a specific procedure for each particular analyte.

#### Ammonia Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NH<sub>3</sub><sup>-</sup>N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 21.3 mg/L with a standard deviation of ± 0.77 mg/L N (replicate number = 7 per reagent lot).

#### Nitrate Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO<sub>3</sub><sup>-</sup>N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 18.9 mg/L with a standard deviation of ± 0.55 mg/L N (replicate number = 7 per reagent lot).

#### Nitrite Nitrogen

## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

In a single laboratory, using a standard solution of 20.0 mg/L  $\text{NO}_2^- \text{N}$  and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 14.6 mg/L with a standard deviation of  $\pm 0.77$  mg/L N (replicate number = 7 per reagent lot).

### Estimated Detection Limit

The estimated detection limit for program 68 is 0.7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

### Interferences

The following ions may interfere when present in concentrations exceeding those listed below:

Species	Level	Effect
Calcium	1000 mg/L as $\text{CaCO}_3$	Positive
Manganese (IV)	3 mg/L	Negative
Magnesium	1000 mg/L as $\text{CaCO}_3$	Positive
Sulfide	3 mg/L	Negative
Sulfate	250 mg/L	Negative

The following do not interfere below the levels listed:

Species	Level
$\text{Al}^{3+}$	8 mg/L
$\text{Ba}^{2+}$	40 mg/L
$\text{Cu}^{2+}$	40 mg/L
$\text{Fe}^{3+}$	8 mg/L
$\text{Zn}^{2+}$	80 mg/L
$\text{F}^-$	40 mg/L
$\text{PO}_4^{3-}\text{-P}$	8 mg/L
$\text{SiO}_2$	80 mg/L
EDTA	80 mg/L

### Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

### REQUIRED REAGENTS

Total Inorganic Nitrogen Pretreatment Reagent Set (TiCl<sub>3</sub> Reduction) (25 tests)..... 26049-45  
Includes: (1) 26051-50, (1) 2040-59, \*(50) TIN Pretreatment Diluent Vials

AmVer™ Reagent Set for Nitrogen, Ammonia, Low Range (25 tests) ..... 26045-45  
Includes: (1) 23952-66, (1) 23954-66 , (1) 272-42, \*(50) AmVer™ Diluent LR Vials

Description	Quantity Required			Cat. No.
	Per Test	Unit		
Total Inorganic Nitrogen Pretreatment Diluent Vials .....	2 vials.....	50/pkg.....		*
Total Inorganic Nitrogen Reductant Ampules .....	2 ampules .....	50/pkg.....		26051-50
Total Inorganic Nitrogen Pretreatment Base Concentrate ...	2 mL.....	50 mL.....		2040-59
AmVer™ Diluent Reagent, Low Range Vials.....	2 vials.....	50/pkg.....		*
Ammonia Salicylate Reagent Powder Pillows				
for 5-mL sample .....	2 pillows.....	50/pkg.....		23952-66
Ammonia Cyanurate Reagent Powder Pillows				
for 5-mL sample .....	2 pillows.....	50/pkg.....		23954-66

### REQUIRED APPARATUS

Centrifuge, 115V.....	1 .....	each.....		26765-00
Centrifuge, 230V .....	1 .....	each.....		26765-02
COD/TNT Vial Adapter.....	1 .....	each.....		48464-00
Funnel, micro .....	1 .....	each.....		25843-35
Pipet, TenSette® , 0.1 to 1.0.....	1 .....	each.....		19700-01
Pipet Tips for 19700-01 .....	2 .....	50/pkg.....		21856-96
Test Tube Rack .....	1 .....	each.....		18641-00

### OPTIONAL REAGENTS

Hydrochloric Acid, ACS.....	500 mL.....			134-49
Nitrate Nitrogen Standard Solution, 10 mg/L NO <sub>3</sub> <sup>-</sup> -N.....	500 mL.....			307-49
Nitrate Nitrogen Standard Solution, PourRite Ampules,				
500 mg/L NO <sub>3</sub> <sup>-</sup> -N, 2 mL.....	20/pkg.....			14260-20
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL SCDB.....			2450-26
Sodium Thiosulfate Standard Solution, 0.1 N .....	100 mL MDB.....			323-32
Wastewater Effluent Standard, Inorganics				
(NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL.....			28332-49
Water, deionized.....	4 L.....			272-56

\* These items are not sold separately. Please order the complete set (cat. no. 26049-45 or 26045-45).

## NITROGEN, TOTAL INORGANIC, Test 'N Tube, continued

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### OPTIONAL APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Cylinder, graduated, mixing, 25 mL .....		each .....	20886-40
Flask, volumetric, Class A, 50.0 mL.....		each .....	14574-41
pH Indicator Paper, 1 to 11 pH.....	5	rolls/pkg .....	391-33
Pipet, volumetric, Class A, 2.0 mL .....		each .....	14515-36
Pipet Tips, for 19700-01 TenSette Pipet .....	1000	/pkg .....	21856-28
PourRite Ampule Breaker .....		each .....	24846-00

### *For Technical Assistance, Price and Ordering*

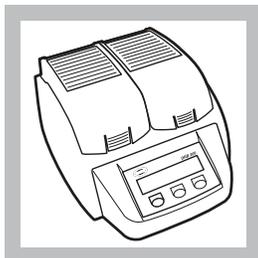
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



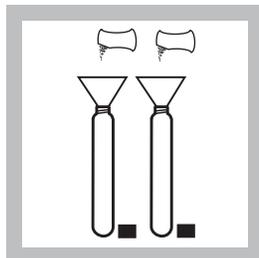
**NITROGEN, TOTAL, Test 'N Tube (0.0 to 25.0 mg/L N)****TNT Persulfate Digestion Method**

For water and wastewater



**1.** Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

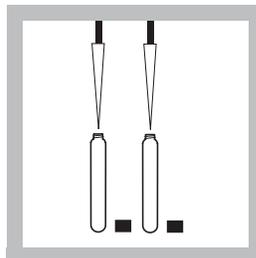
*Note:* For proof of accuracy, run a 20 mg/L  $\text{NH}_3\text{-N}$  standard through digestion and analysis.



**2.** Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Reagent vials.

*Note:* Wipe off any reagent that may get on the lid or the tube threads.

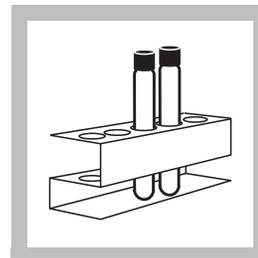
*Note:* One reagent blank is sufficient for each set of samples.



**3.** Add 2 mL of sample to one vial. Add 2 mL of organic-free water to another vial (the reagent blank). Cap both vials and shake vigorously (about 30 seconds). Place the vials in the Reactor. Heat for 30 minutes.

*Note:* The reagent may not dissolve completely after shaking.

*Note:* Alternate water must be free of all nitrogen-containing species.

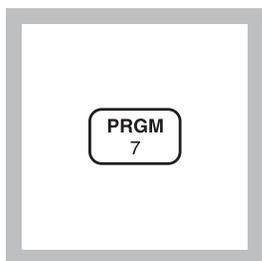


**4.** Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

*Note:* It is very important to remove the vials from the Reactor after exactly 30 minutes.

## NITROGEN, TOTAL, Test 'N Tube, continued

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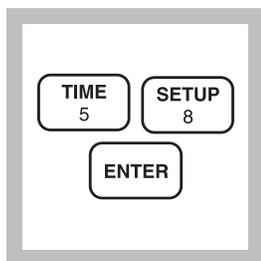


5. Enter the stored program number for Test 'N Tube Total Nitrogen.

Press: **PRGM**

The display will show:

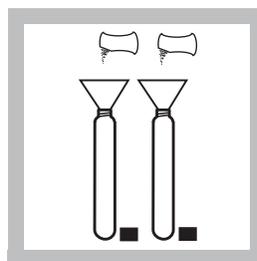
**PRGM ?**



6. Press: **58 ENTER**

The display will show **mg/L, N** and the **ZERO** icon.

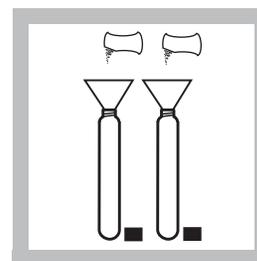
*Note: For alternate forms ( $NH_3$ ,  $NO_3$ ), press the **CONC** key.*



7. Remove the caps from the digested vials and add the contents of one TN Reagent A Powder Pillow to each vial. Cap the vials and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



8. After the timer beeps, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial. Cap the vials and shake for 15 seconds.

The display will show:

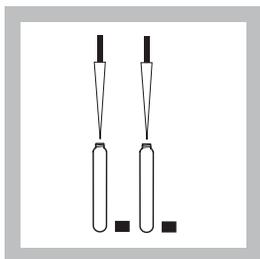
**02:00 Timer 2**

Press **ENTER** after shaking.

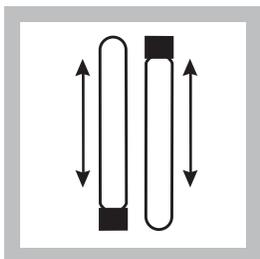
A two-minute reaction period will begin.

*Note: The reagent will not completely dissolve. The solution will begin to turn yellow.*

## NITROGEN, TOTAL, Test 'N Tube, continued

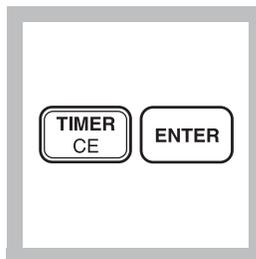


**9.** After the timer beeps, remove the caps from two TN Reagent C Vials. Add 2 mL of digested, treated sample to one vial. Add 2 mL of the digested, treated reagent blank to the second TN Reagent C Vial.



**10.** Cap and invert 10 times to mix. Use slow, deliberate inversions for complete recovery. The vials will be warm.

*Note: Follow these instructions for inversion or low results may occur. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*



**11.** The display will show: **05:00 Timer 3**

Press: **ENTER**

A five-minute reaction period will begin.

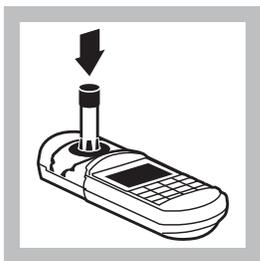
*Note: The yellow color will intensify.*



**12.** During the reaction period, insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

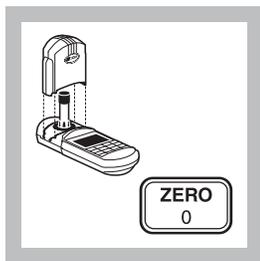
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

## NITROGEN, TOTAL, Test 'N Tube, continued



**13.** After the timer beeps, wipe the TN Reagent C vial containing the reagent blank. Place the vial in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**14.** Tightly cover the vial with the instrument cap.

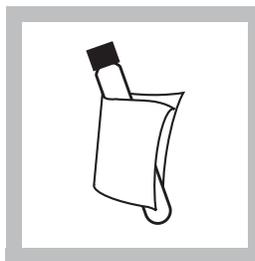
Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.0 mg/L N**

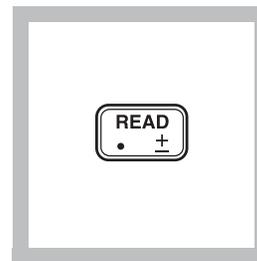
*Note:* Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.

*Note:* The reagent blank is stable when stored in the dark; see Blanks For Colorimetric Measurement following these steps.



**15.** Wipe the TN Reagent C vial containing the sample and place it into the adapter. Tightly cover the vial with the instrument cap.

*Note:* Multiple samples may be read after zeroing on one reagent blank.



**16.** Press: **READ**

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

*Note:* If the display flashes "limit", dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see Section 1.

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

### Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

## NITROGEN, TOTAL, Test 'N Tube, continued

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1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.
  - a) Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
  - b) Weigh 0.4416 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
  - c) Weigh 0.5274 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Each analyst has found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

### Standard Solution Method

Substitute 2 mL of a 20 mg/L ammonia nitrogen standard solution for the sample. To prepare a 20-mg/L standard, use a 20-mL Class A pipet to transfer 20 mL of a 100-mg/L Ammonia Nitrogen Standard (see *Optional Reagents*) to a 100-mL Class A volumetric flask. Dilute to the line with organic-free water. A single analyst should obtain less than 5% variation on replicates. Comparison of the user-obtained value with the standard concentration is an indication of test performance for this user.

## NITROGEN, TOTAL, Test 'N Tube, continued

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### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off an Ammonia Nitrogen Voluette Ampule Standard Solution, 160 mg/L as  $\text{NH}_3\text{-N}$ .
- c) Use the TenSette Pipet to add 0.3 mL, 0.6 mL, and 0.9 mL of standard, respectively, to the three mixing cylinders.
- d) Stopper each cylinder and mix thoroughly.
- e) Add 2 mL of each prepared solution, respectively, to three TN Hydroxide Reagent Sample Digestion Vials.
- f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 2 mg/L for each 0.3 mL of standard added.
- g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

### Blanks for Colorimetric Measurement

The reagent blank may be used up to 7 days for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18-25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

### Method Performance

#### Precision

A Hach chemist analyzed two independent nutrient standards. The lowest average percent recovery was 95% with a standard deviation of  $\pm 2\%$ .

In a single laboratory, using a standard solution of 15.0 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than  $\pm 0.5$  mg/L N. For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program 58 is 2 mg/L N. For more information on the estimated detection limit, see *Section 1*.

## NITROGEN, TOTAL, Test 'N Tube, continued

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### Interferences

Interfering substances that resulted in a concentration change of  $\pm 10\%$ :

Substance	Level and Effect
Bromide	>60 ppm; positive interference
Chloride	>1000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels (in mg/L):

Substance	Maximum Level Tested (mg/L)
Barium	2.6
Calcium	300
Chromium (3+)	0.5
Iron	2
Lead	6.6 ppb
Magnesium	500
Organic Carbon	150
pH	13 pH units
Phosphorus	100
Silica	150
Silver	0.9
Tin	1.5

Hach chemists tested this chemistry on standard nitrogen solutions prepared from the following compounds and obtained  $\geq 95\%$  recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in  $\geq 95\%$  recovery.

## NITROGEN, TOTAL, Test 'N Tube, continued

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Large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

### Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

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### REQUIRED REAGENTS

Description	Cat. No.
Test 'N Tube Total Nitrogen Reagent Set (50 vials).....	26722-45
Includes:	
TN Reagent C Vials, Acid Solution*, 50/pkg .....	26721-45
TN Hydroxide Reagent Sample Digestion Vials*, 50/pkg.....	26717-45

Description	Quantity Required		Cat. No.
	Per Test	Unit	
TN Persulfate Reagent Powder Pillows.....	2 pillows .....	100/pkg.....	26718-49
TN Reagent A, Bisulfite Powder Pillows .....	2 pillows .....	100/pkg.....	26719-49
TN Reagent B, Indicator Powder Pillows.....	2 pillows .....	100/pkg.....	26720-49

### REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....	LTV082.53.40001		
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....	LTV082.52.40001		
COD/TNT Adapter .....	1 .....	each.....	48464-00
Funnel, micro .....	1 .....	each.....	25843-35
Pipet, TenSette, 1.0-10.0 mL .....	1 .....	each.....	19700-10
Pipet Tips for 19700-10 .....	1 .....	50/pkg.....	21997-96
Pipet, TenSette, 0.1 to 1.0 mL.....	1 .....	each.....	19700-01
Pipet Tips for 19700-01 .....	2.....	50/pkg.....	21856-96
Test Tube Cooling Rack.....	1-3 .....	each.....	18641-00

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\* Not available separately.

## NITROGEN, TOTAL, Test 'N Tube, continued

### OPTIONAL REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Nitrogen, Ammonia, 100 mg/L NH <sub>3</sub> -N .....	500 mL .....	24065-49	
Nitrogen, Ammonia, Voluette Ampule, 160 mg/L NH <sub>3</sub> -N, 10 mL .....	16/pkg .....	21091-10	
Sulfuric Acid, ACS .....	500 mL .....	979-49	
Primary Standards for Kjeldahl Nitrogen.....	set of 3 .....	22778-00	
Ammonium p-Toluenesulfonate.....	25 g .....	22779-24	
Glycine p-Toluenesulfonate .....	25 g .....	22780-24	
Nicotinic Acid p-Toluenesulfonate .....	25 g .....	22781-24	
Sodium Hydroxide Standard Solution, 5.0 N.....	50 mL MDB .....	2450-26	
Wastewater Effluent Standard, Inorganics (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49	
Water, organic-free .....	500 mL .....	26415-49	

### OPTIONAL APPARATUS

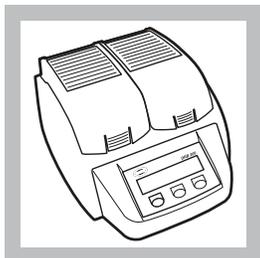
Ampule Breaker Kit .....	each .....	21968-00
Balance, analytical, 115 VAC.....	each .....	28014-01
Balance, analytical, 230 VAC .....	each .....	28014-02
Cots, finger .....	2/pkg .....	14647-02
Cylinder, graduated, mixing, 25 mL (3 required) .....	each .....	26363-40
Flask, volumetric, Class A, 1000 mL (3 required).....	each .....	14574-53
Flask, volumetric, Class A, 100 mL.....	each .....	14574-42
Pipet, volumetric, Class A, 20 mL .....	each .....	14515-20
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
pH Paper, 1 to 11 pH units .....	5 rolls/pkg .....	391-33
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.52.30001	



# NITROGEN, TOTAL, HR, Test 'N Tube™ (10.0 to 150.0 mg/L N)

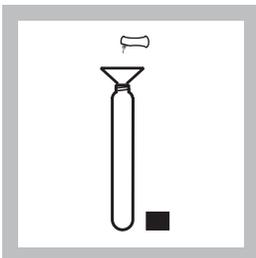
## TNT Persulfate Digestion Method

For water and wastewater



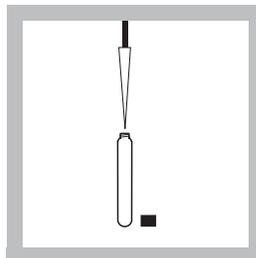
**1.** Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

*Note:* For proof of accuracy, run a 125 mg/L  $\text{NH}_3\text{-N}$  standard through digestion and analysis.



**2. Prepare a reagent blank:** Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

*Note:* Wipe off any reagent that gets on the lid or the tube threads.



**3.** Add 0.5 mL of organic-free water to the vial. Cap the vial and shake vigorously for about 30 seconds.

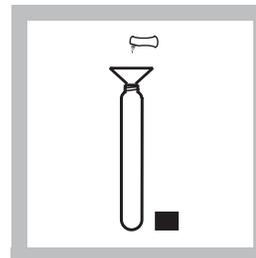
Process this reagent blank exactly the same as the sample, including digestion and color finish. Proceed to step 6.

*Note:* Alternate water must be free of all nitrogen-containing species.

*Note:* The persulfate reagent may not dissolve completely after shaking.

*Note:* One reagent blank is sufficient for each set of samples using the same lots of reagents.

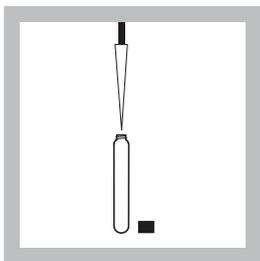
*Note:* The reagent blank is stable for as long as seven days when stored in the dark; see Blanks for Colorimetric Measurement following this procedure.



**4. Prepare a sample:** Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

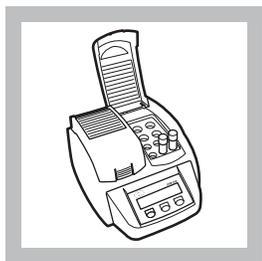
*Note:* Wipe off any reagent that gets on the lid or the tube threads.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

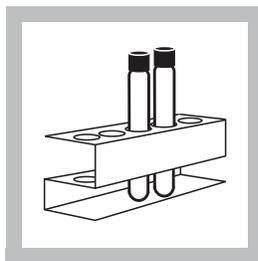


**5.** Add 0.5 mL of sample to the vial. Cap the vial and shake vigorously for about 30 seconds.

*Note: The persulfate reagent may not dissolve completely after shaking.*

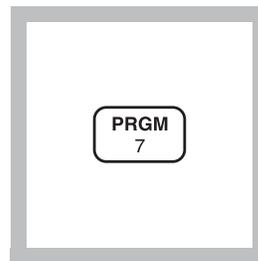


**6.** Place the vials in the Reactor. Heat for 30 minutes.



**7.** Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

*Note: It is very important to remove the vials from the Reactor after exactly 30 minutes.*

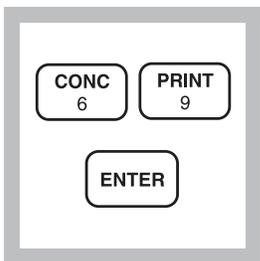


**8.** Enter the stored program number for Test 'N Tube HR Total Nitrogen.

Press: **PRGM**

The display will show:

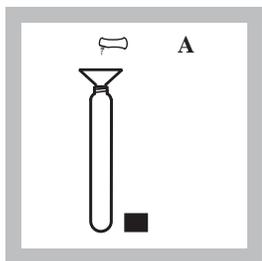
**PRGM ?**



**9. Press: 69 ENTER**

The display will show **mg/L, N** and the **ZERO** icon.

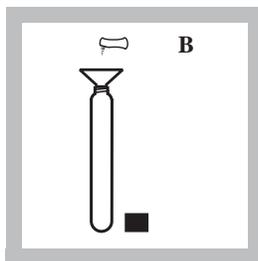
*Note: For alternate forms ( $NH_3$ ,  $NO_3$ ), press the **CONC** key.*



**10.** Add the contents of one Total Nitrogen Reagent A Powder Pillow to the vial containing the digested blank or sample. Cap the vial and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



**11.** After the timer beeps, add one Total Nitrogen Reagent B Powder Pillow to the vial. Cap the vial and shake for 15 seconds.

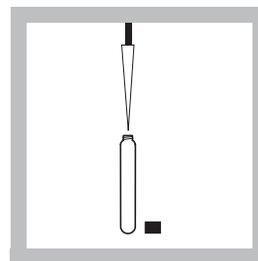
The display will show:

**02:00 Timer 2**

Press **ENTER** after shaking.

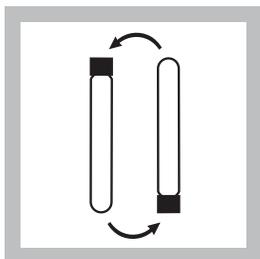
A two-minute reaction period will begin.

*Note: The reagent will not completely dissolve. The solution will begin to turn yellow.*



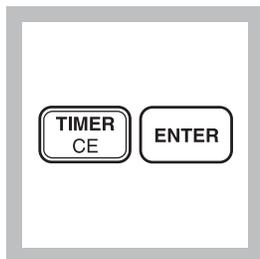
**12.** After the timer beeps, remove the cap from one Total Nitrogen Reagent C Vial. Add 2 mL of digested, treated sample (or reagent blank) to the vial. The vial will be warm.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued



**13.** Cap and invert slowly 10 times to mix. The vial will be warm.

*Note: Proper mixing is important for complete recovery. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*

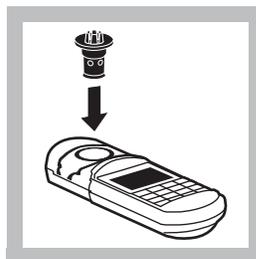


**14.** The display will show: **05:00 Timer 3**

Press: **ENTER**

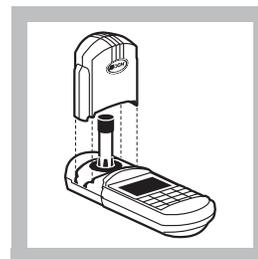
A five-minute reaction period will begin. Do not invert the vial again.

*Note: The yellow color will intensify.*



**15.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**16.** When the timer beeps, wipe the outside of the Total Nitrogen Reagent C vial containing the reagent blank.

Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

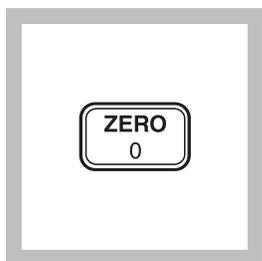
Tightly cover the vial with the instrument cap.

*Note: Do not move the vial from side to side during insertion, as this can cause errors.*

*Note: Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

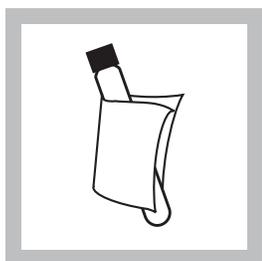
## NITROGEN, TOTAL, HR, Test 'N Tube, continued

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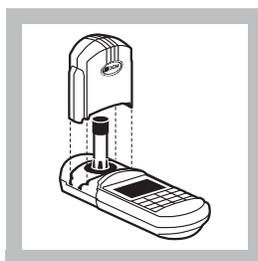
**17. Press: ZERO**  
The cursor will move to the right, then the display will show:

**0 mg/L N**



**18. Wipe the Total Nitrogen Reagent C vial containing the sample.**

*Note: Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

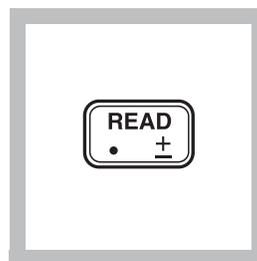


**19. Place the vial into the adapter with the Hach logo facing the front of the instrument. Push straight down on the top of the vial until it seats solidly into the adapter.**

Tightly cover the vial with the instrument cap.

*Note: Do not move the vial from side to side during insertion, as this can cause errors.*

*Note: Multiple samples may be read after zeroing on one reagent blank.*



**20. Press: READ**  
The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1 of the Procedures Manual).*

*Note: If the display flashes **Limit**, dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see SECTION 1.*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL/L). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

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### Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 120 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.
  - a) Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
  - b) Weigh 2.1179 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
  - c) Weigh 2.5295 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{120} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

### Standard Solution Method

For proof of accuracy, substitute 0.5 mL of a 125 mg/L ammonia nitrogen standard solution for the sample in the procedure. To prepare a 125-mg/L standard, use a 25-mL Class A pipet to transfer 25.00 mL of a 1000-mg/L Ammonia Nitrogen Standard

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

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(see *OPTIONAL REAGENTS* on page 400) to a 200-mL Class A volumetric flask. Dilute to the line with organic-free water.

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off an Ammonia Nitrogen Voluette™ Ampule Standard Solution, 1000 mg/L as NH<sub>3</sub>-N.
- c) Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders.
- d) Stopper each cylinder and mix thoroughly.
- e) Add 0.5 mL of each prepared solution, respectively, to three HR Total Nitrogen Hydroxide Digestion Vials.
- f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 4 mg/L N for each 0.1 mL of standard added.
- g) If these increases do not occur, see *Standard Additions in Section 1* for troubleshooting information.

### Blanks for Colorimetric Measurement

The reagent blank may be used repeatedly for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18–25 °C) for a maximum of seven days. If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 125 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 3 mg/L N. For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program 69 is 7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

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### Interferences

Interfering substances that resulted in a concentration change of  $\pm 10\%$ :

Substance	Level and Effect
Bromide	> 240 ppm; positive interference
Chloride	$\geq 3000$ ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels:

Substance	Maximum Level Tested (mg/L)
Barium	10.4
Calcium	1200
Chromium (3+)	2
Iron	8
Lead	26.4 ppb
Magnesium	2000
Organic Carbon	600
pH	13 pH units
Phosphorus	400
Silica	600
Silver	3.6
Tin	6.0

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

### Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

### REQUIRED REAGENTS

Test 'N Tube HR Total Nitrogen Reagent Set (50 vials) ..... 27141-00  
 Includes: (1) 26718-46, (1) 26719-46, (1) 26720-46, \*(50) Hydroxide Digestion Vials,  
 \*(50) Acid Solution Vials

Description	Quantity Required		Unit	Cat. No.
	Per Test			
HR Total Nitrogen Hydroxide Digestion Vials.....	1 vial	50/pkg		*
Total Nitrogen Persulfate Reagent Powder Pillows....	1 pillow	50/pkg		26718-46
Total Nitrogen Reagent A, Bisulfite Powder Pillows .1	pillow	50/pkg		26719-46
Total Nitrogen Reagent B, Indicator Powder Pillows.1	pillow	50/pkg		26720-46
Total Nitrogen Reagent C Vials, Acid Solution.....	1 vial	50/pkg		*

### REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....				LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....				LTV082.52.40001
COD/TNT Adapter .....	1	each		48464-00
Funnel, micro .....	1	each		25843-35
Pipet, TenSette, 0.1 to 1.0 mL.....	1	each		19700-01
Pipet Tips for 19700-01 .....	2	50/pkg		21856-96
Test Tube Rack, for cooling vials .....	1-3	each		18641-00

### OPTIONAL REAGENTS

Nitrogen, Ammonia, 1000 mg/L NH <sub>3</sub> -N .....	1 L			23541-53
Nitrogen, Ammonia, Voluette Ampule, 1000 mg/L NH <sub>3</sub> -N, 10 mL .....	16/pkg			23541-10
Sulfuric Acid, ACS .....	500 mL			979-49
Primary Standards for Kjeldahl Nitrogen .....	set of 3			22778-00
Ammonium p-Toluenesulfonate .....	25 g			22779-24
Glycine p-Toluenesulfonate .....	25 g			22780-24
Nicotinic Acid p-Toluenesulfonate .....	25 g			22781-24
Sodium Hydroxide Standard Solution, 5.0 N .....	50 mL			2450-26
Wastewater Influent Standard, Inorganics (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL			28331-49
Water, organic-free.....	500 mL			26415-49

\* These items are not sold separately. Please order the complete set (Cat. No. 27141-00) as a replacement.

## NITROGEN, TOTAL, HR, Test 'N Tube, continued

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### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit .....	each.....	21968-00
Balance, analytical, 115 Vac.....	each.....	28014-01
Balance, analytical, 230 Vac .....	each.....	28014-02
Cots, finger .....	2/pkg.....	14647-02
Cylinder, graduated, mixing, 25 mL .....	3..... each.....	26363-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm.....		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....		LTV082.52.30001
Flask, volumetric, Class A, 1000 mL .....	3..... each.....	14574-53
Flask, volumetric, Class A, 200 mL.....	each.....	14574-45
Pipet, volumetric, Class A, 25 mL .....	2..... each.....	14515-40
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
pH Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224. Out side the U.S.A— Contact the Hach office or distributor serving you.

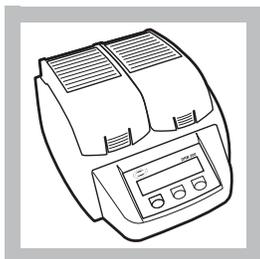
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**ORGANIC CARBON, TOTAL, Low Range (0.0–20.0 mg/L C)**

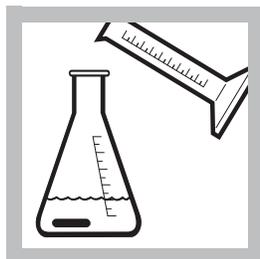
Direct Method\*

For water, drinking water, and wastewater

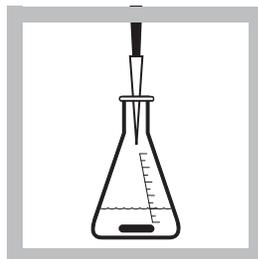


**1.** Turn on the DRB 200 reactor. Heat to 103-105 °C.

*Note:* See DRB 200 user manual for selecting pre-programmed temperature applications.

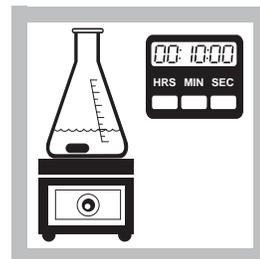


**2.** Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

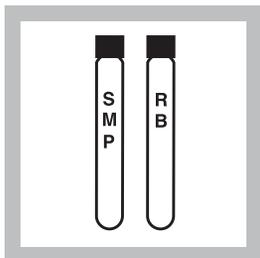


**3.** Add 0.4 mL of Buffer Solution, pH 2.0.

*Note:* Use pH paper to make sure the sample pH is 2.

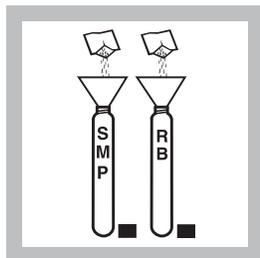


**4.** Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

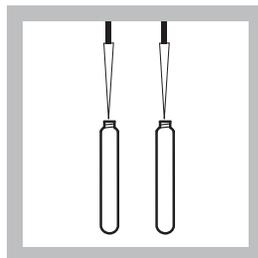


**5.** Label two Low Range Acid Digestion vials: **sample** and **reagent blank**.

*Note:* A reagent blank is required for each series of samples.



**6.** Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



**7.** Use a TenSette® Pipet to add 3.0 mL of **organic-free water** to the **reagent blank** vial and 3.0 mL of **prepared sample** to the **sample** vial. Swirl to mix.

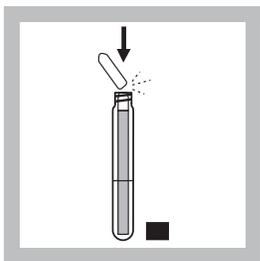


**8.** Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

*Note:* Do not touch the ampules on the sides after wiping. Pick them up by the top.

\* Patent pending

## ORGANIC CARBON, TOTAL, Low Range, continued

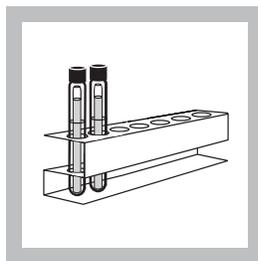


**9.** Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

*Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.*

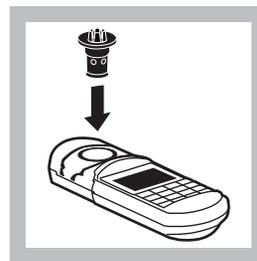


**10.** Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103-105 °C.



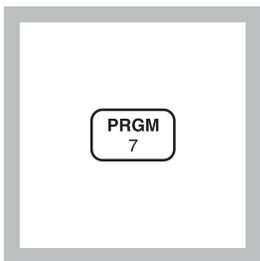
**11.** Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



**12.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

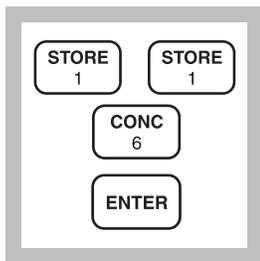
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**13.** Enter the stored program number for Low Range TOC.

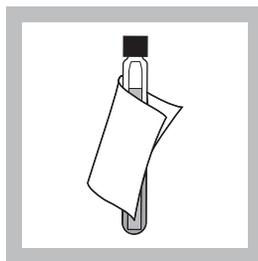
Press: **PRGM**

The display will show:  
**PRGM?**



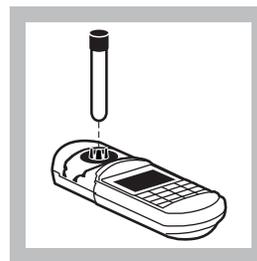
**14.** Press: **116 ENTER**

The display will show **mg/L** and the **ZERO** icon.



**15.** Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

*Note: The liquid in the reagent blank vial should be dark blue.*

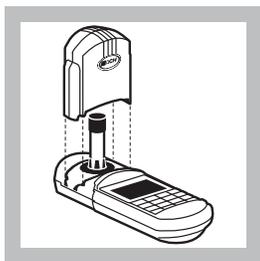


**16.** Place the **reagent blank** vial assembly in the adapter.

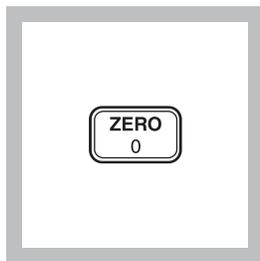
Push straight down on the top of the vial until it seats solidly in the adapter.

## ORGANIC CARBON, TOTAL, Low Range, continued

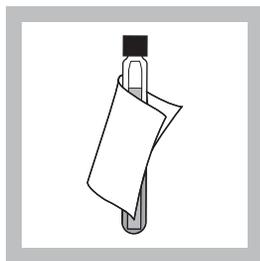
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**17.** Tightly cover the vial assembly with the instrument cap.



**18.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.0 mg/L C**

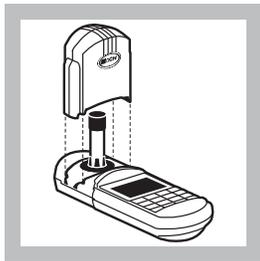


**19.** Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

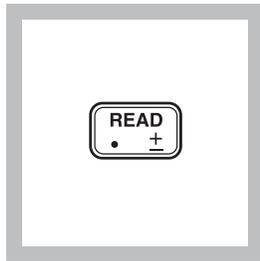


**20.** Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



**21.** Tightly cover the vial assembly with the instrument cap.



**22.** Press: **READ**  
The cursor will move to the right, then the result in mg/L C will be displayed.

---

### Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

## ORGANIC CARBON, TOTAL, Low Range, continued

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### Accuracy Check

#### Standard Solutions Method

- a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

- b. Prepare a 10.0 mg/L C standard by transferring 1.00 mL of the stock standard to a 100-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

#### Standard Additions Method

- a. Prepare a 150 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with organic-free water. Mix.
- b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 150 mg/L C standard to each of three Acid Digestion vials.
- c. Add the contents of one TOC Persulfate powder pillow to each vial.
- d. Add 3.0 mL of sample to each vial. Swirl to mix.
- e. Proceed with the procedure starting at *step 8*.
- f. The mg/L C concentration should increase by 5.0 mg/L for each 0.1 mL increment.

## ORGANIC CARBON, TOTAL, Low Range, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 9.0 mg/L C and one lot of reagents, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L C.

#### Estimated Detection Limit

The estimated detection limit for Method 10129 is 0.3 mg/L C.

#### Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 0.2 mg/L C.

### Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br <sup>-</sup>
Bromine	25 mg/L Br <sub>2</sub>
Calcium	2000 mg/L as CaCO <sub>3</sub>
Chloride	500 mg/L
Chlorine	10 mg/L Cl <sub>2</sub>
Chlorine Dioxide	6 mg/L ClO <sub>2</sub>
Copper	10 mg/L
Cyanide	10 mg/L CN <sup>-</sup>
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as CaCO <sub>3</sub>
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH <sub>2</sub> Cl as Cl <sub>2</sub>
Nitrite	500 mg/L NO <sub>2</sub> <sup>-</sup>
Ozone	2 mg/L O <sub>3</sub>
Phosphate	3390 mg/L PO <sub>4</sub> <sup>2-</sup>

## ORGANIC CARBON, TOTAL, Low Range, continued

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO <sub>2</sub>
Sulfate	5000 mg/L SO <sub>4</sub> <sup>2-</sup>
Sulfide	20 mg/L S <sup>2-</sup>
Sulfite	50 mg/L SO <sub>3</sub> <sup>2-</sup>
Zinc	5 mg/L

If the sample contains greater than 600 mg/L CaCO<sub>3</sub> alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

### Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

### REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Total Organic Carbon Direct Method Low Range			
Test 'N Tube Reagent Set.....	50 vials.....		27603-45
<b>Includes:</b>			
Acid Digestion Solution Vials, Low Range TOC.....	1 .....	50/pkg .....	*
Buffer Solution, Sulfate .....	0.4 mL .....	25 mL.....	452-33
Funnel, micro .....	1 .....	each.....	25843-35
Indicator Ampules, Low Range TOC .....	1 .....	10/pkg.....	*
TOC Persulfate Powder Pillows .....	1 .....	50/pkg.....	*
Water, organic-free** .....	3.0 mL .....	500 mL.....	26415-49

\* These items are not sold separately.

\*\* This item must be purchased separately.

## ORGANIC CARBON, TOTAL, Low Range, continued

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### REQUIRED APPARATUS

Description	Qty/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL .....	1.....	each .....	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....		LTV082.52.40001	
Flask, Erlenmeyer, 50-mL.....	1.....	each .....	505-41
Magnetic Stirrer, 115 V, 4" x 4" .....	1.....	each .....	28812-00
Test Tube Rack .....	1-3.....	each .....	18641-00
Pipet, TenSette <sup>®</sup> , 0.1 to 1.0 mL.....	1.....	each .....	19700-01
Pipet, TenSette <sup>®</sup> , 1.0 to 10.0 mL.....	1.....	each .....	19700-10
Pipet Tips, for 19700-01 TenSette <sup>®</sup> Pipet .....	2.....	50/pkg .....	21856-96
Pipet Tips, for 19700-10 TenSette <sup>®</sup> Pipet .....	2.....	50/pkg .....	21997-96
Stir Bar, Magnetic .....	1.....	each .....	45315-00
Wipes, Disposable, Kimwipes.....	1.....	280/pkg .....	20970-00

### OPTIONAL REAGENTS

Potassium Acid Phthalate.....	500 g .....	315-34
Sulfuric Acid Reagent Solution, 5.25 N.....	100 mL MDB .....	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C).....	5/pkg .....	27915-05
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49

### OPTIONAL APPARATUS

Analytical Balance .....	each .....	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....		LTV082.52.30001
Flask, volumetric, 100-mL .....	each .....	14574-42
Pipet, Class A, 200-mL .....	each .....	14515-35
Pipet, Class A, 15.00-mL .....	each .....	14515-39



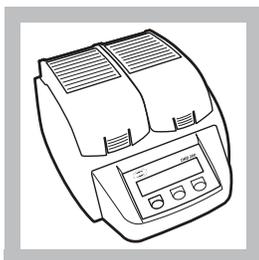
# ORGANIC CARBON, TOTAL, Mid Range

Method 10173

(15–150 mg/L C)

Direct Method\*

For wastewater and industrial waters

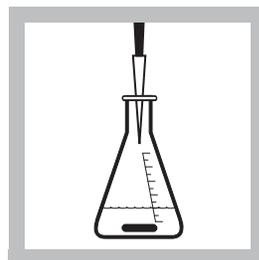


**1.** Turn on the DRB 200 reactor. Heat to 103–105 °C.

*Note: See DRB 200 user manual for selecting pre-programmed temperature applications.*

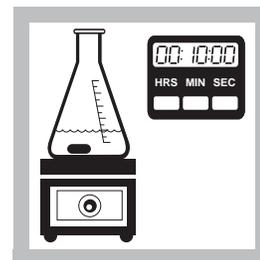


**2.** Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

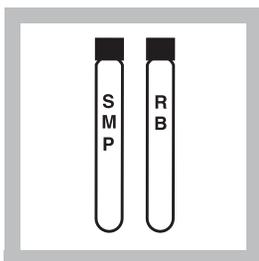


**3.** Add 0.4 mL of Buffer Solution, pH 2.0.

*Note: Use pH paper to make sure the sample pH is 2.*

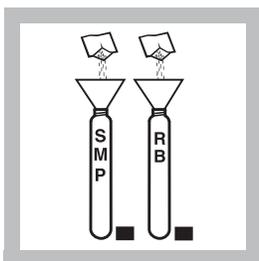


**4.** Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

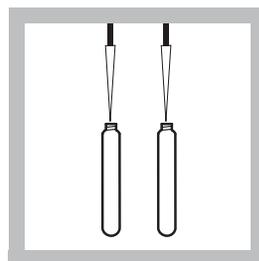


**5.** Label two Mid Range Acid Digestion vials: **sample** and **reagent blank**.

*Note: A reagent blank is required for each series of samples.*



**6.** Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



**7.** Use a TenSette® Pipet to add 1.0 mL of **organic-free water** to the **reagent blank** vial and 1.0 mL of **prepared sample** to the **sample** vial. Do not cap the vial; swirl gently to mix.

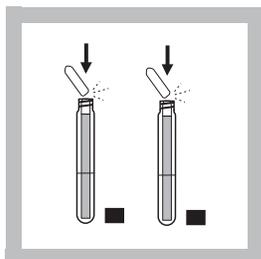


**8.** Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

*Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.*

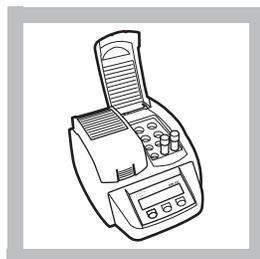
\* Patent pending

## ORGANIC CARBON, TOTAL, Mid Range, continued

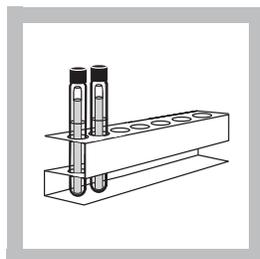


**9.** Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

*Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.*

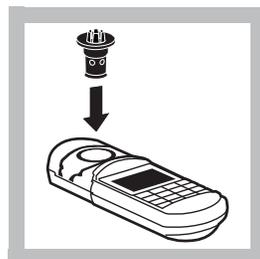


**10.** Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



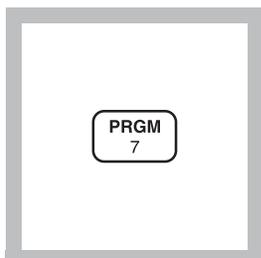
**11.** Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



**12.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

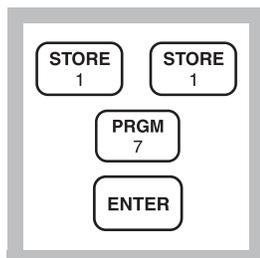
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



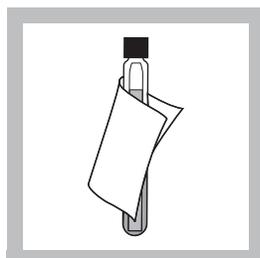
**13.** Enter the stored program number for Mid Range TOC.

Press: **PRGM**

The display will show:  
**PRGM?**



**14.** Press: **117 ENTER**  
The display will show **mg/L** and the **ZERO** icon.



**15.** Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

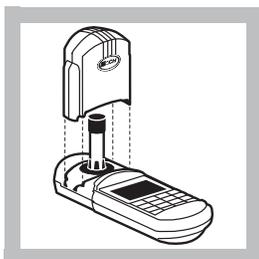
*Note: The liquid in the reagent blank vial should be dark blue.*



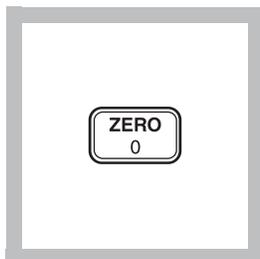
**16.** Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.

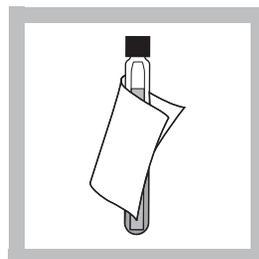
## ORGANIC CARBON, TOTAL, Mid Range, continued



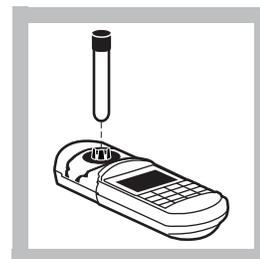
**17.** Tightly cover the vial assembly with the instrument cap.



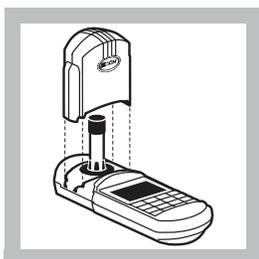
**18.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0 mg/L C**



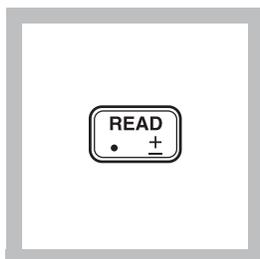
**19.** Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.



**20.** Place the **sample** vial assembly in the adapter.  
Push straight down on the top of the vial assembly until it seats solidly in the adapter.



**21.** Tightly cover the vial assembly with the instrument cap.



**22.** Press: **READ**  
The cursor will move to the right, then the result in mg/L C will be displayed.

## ORGANIC CARBON, TOTAL, Mid Range, continued

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### Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

### Accuracy Check

#### Standard Solutions Method

- a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature. Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).
- b. Prepare a 100 mg/L C standard by transferring 5.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

#### Standard Additions Method

- a. Prepare a 300 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.
- b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.
- c. Add the contents of one TOC Persulfate powder pillow to each vial.
- d. Add 1.0 mL of sample to each vial. Swirl to mix.
- e. Proceed with the procedure starting at *step 8*.
- f. The mg/L C concentration should increase by 30 mg/L for each 0.1 mL increment.

## ORGANIC CARBON, TOTAL, Mid Range, continued

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### Method Performance

#### Precision

mg/L C	95% Confidence Limits
15	± 5 mg/L C
50	± 6 mg/L
75	± 7 mg/L
115	± 4 mg/L
150	± 6 mg/L

#### Estimated Detection Limit

Use Method Number 10173 to test TOC levels below 15 mg/L C.

#### Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 1.9 mg/L C.

#### Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

**Table 1 Non-interfering Substances**

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br <sub>2</sub>
Calcium	2000 mg/L as CaCO <sub>3</sub>
Chloride	1500 mg/L
Chlorine	10 mg/L Cl <sub>2</sub>
Chlorine Dioxide	6 mg/L ClO <sub>2</sub>
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as CaCO <sub>3</sub>
Manganese (VII)	1 mg/L

## ORGANIC CARBON, TOTAL, Mid Range, continued

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Monochloramine	14 mg/L $\text{NH}_2\text{Cl}$ as $\text{Cl}_2$
Nitrite	500 mg/L $\text{NO}_2^-$
Ozone	2 mg/L $\text{O}_3$
Phosphate	3390 mg/L $\text{PO}_4^{3-}$
Silica	100 mg/L $\text{SiO}_2$
Sulfate	5000 mg/L $\text{SO}_4^{2-}$
Sulfide	20 mg/L $\text{S}^{2-}$
Sulfite	50 mg/L $\text{SO}_3^{2-}$
Zinc	5 mg/L

*Note: If the sample contains greater than 1000 mg/L  $\text{CaCO}_3$  alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.*

*Note: Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.*

### Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

## ORGANIC CARBON, TOTAL, Mid Range, continued

### Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change a number already entered.

Line Number	Entry	Line Number	Entry
1	117	29	0
2	42	30	0
3	72	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	66	36	0
9	36	37	0
10	92	38	0
11	40	39	0
12	195	40	0
13	89	41	0
14	74	42	0
15	61	43	0
16	0	44	165
17	0	45	128
18	0	46	0
19	0	47	10
20	67	48	0
21	0	49	0
22	0	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	25
27	0	55	0
28	0	56	255

## ORGANIC CARBON, TOTAL, Mid Range, continued

### REQUIRED REAGENTS

Total Organic Carbon Direct Method Mid Range

Test 'N Tube Reagent Set ..... 50 vials..... 28159-45

#### Includes:

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Acid Digestion Solution Vials, Mid Range TOC .....	1	50/pkg	*
Buffer Solution, Sulfate .....	0.4 mL	25 mL	452-33
Funnel, micro .....	1	each	25843-35
Indicator Ampules, Mid/High Range TOC.....	1	50/pkg	*
TOC Persulfate Powder Pillows .....	1	50/pkg	*
Water, organic-free** .....	1.0 mL	500 mL	26415-49

### REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....	LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....	LTV082.52.40001
Cylinder, graduated, 10-mL.....	1 each..... 508-38
Flask, Erlenmeyer, 50-mL .....	1 each..... 505-41
Magnetic Stirrer, 115 V, 4" x 4" .....	1 each..... 28812-00
Test Tube Rack .....	1-3 each..... 18641-00
Pipet, TenSette <sup>®</sup> , 0.1 to 1.0 mL .....	1 each..... 19700-01
Pipet Tips, for 19700-01 TenSette <sup>®</sup> Pipet.....	2 50/pkg..... 21856-96
Stir Bar, Magnetic .....	1 each..... 45315-00
Wipes, Disposable, Kimwipes .....	1 280/pkg..... 20970-00

### OPTIONAL REAGENTS

Description	Per Test	Unit	Cat. No.
TOC Standard Solution (KHP Standard, 1000 mg/L C).....	5	5/pkg	27915-05
Potassium Acid Phthalate .....	500	g	315-34
Sulfuric Acid Reagent Solution, 5.25 N .....	100	mL MDB	2449-32

### OPTIONAL APPARATUS

Analytical Balance .....	each..... 28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm .....	LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm .....	LTV082.52.30001
Flask, volumetric, 100-mL.....	each..... 14574-42
Pipet, Class A, 10.00-mL.....	each..... 14515-38
Pipet, Class A, 15.00-mL.....	each..... 14515-39
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg..... 21856-28

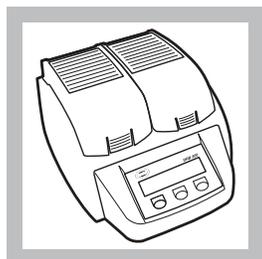
\* These items are not sold separately.

\*\* This item must be purchased separately.

**ORGANIC CARBON, TOTAL, High Range (20–700 mg/L C)**

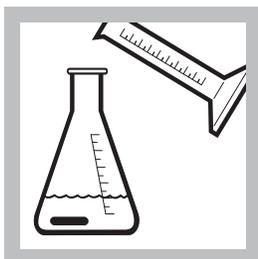
Direct Method\*

For wastewater and industrial waters

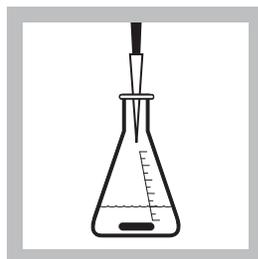


**1.** Turn on the DRB 200 reactor. Heat to 103-105 °C.

*Note:* See DRB 200 user manual for selecting pre-programmed temperature applications.

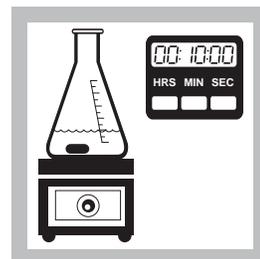


**2.** Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

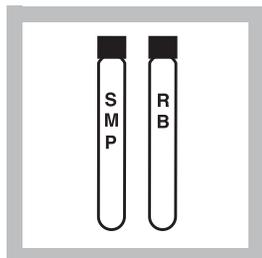


**3.** Add 0.4 mL of Buffer Solution, pH 2.0.

*Note:* Use pH paper to make sure the sample pH is 2.

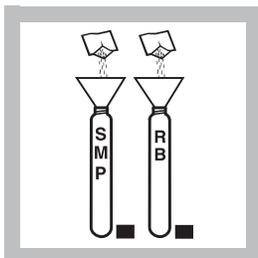


**4.** Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

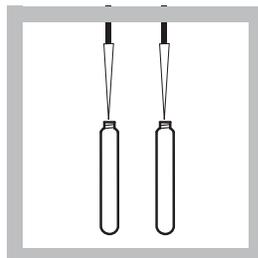


**5.** Label two High Range Acid Digestion vials: **sample** and **reagent blank**.

*Note:* A reagent blank is required for each series of samples.



**6.** Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



**7.** Use a TenSette® Pipet to add 0.3 mL of **organic-free water** to the **reagent blank** vial and 0.3 mL of **prepared sample** to the **sample** vial. Swirl to mix.

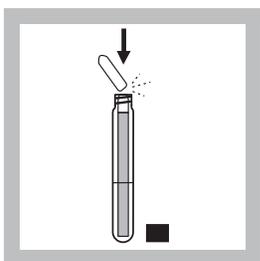


**8.** Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

*Note:* Do not touch the ampules on the sides after wiping. Pick them up by the top.

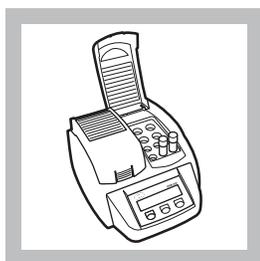
\* Patent pending

## ORGANIC CARBON, TOTAL, High Range, continued

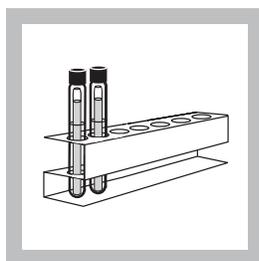


**9.** Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

*Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.*

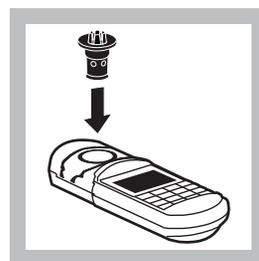


**10.** Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



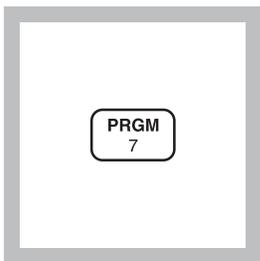
**11.** Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



**12.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

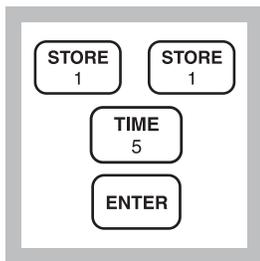
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



**13.** Enter the stored program number for High Range TOC.

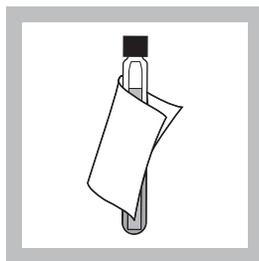
Press: **PRGM**

The display will show:  
**PRGM?**



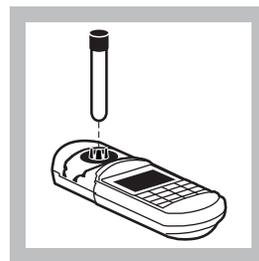
**14.** Press: **115 ENTER**

The display will show **mg/L** and the **ZERO** icon.



**15.** Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

*Note: The liquid in the reagent blank vial should be dark blue.*

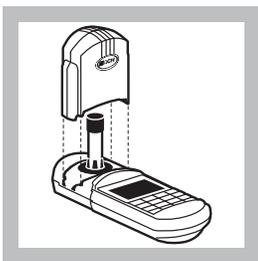


**16.** Place the **reagent blank** vial assembly in the adapter.

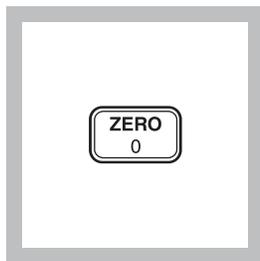
Push straight down on the top of the vial until it seats solidly in the adapter.

## ORGANIC CARBON, TOTAL, High Range, continued

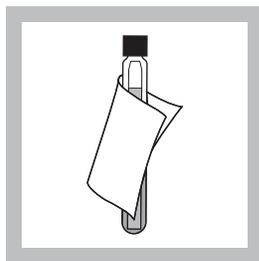
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**17.** Tightly cover the vial assembly with the instrument cap.



**18.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0 mg/L C**

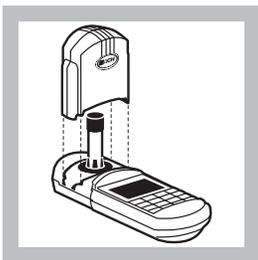


**19.** Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

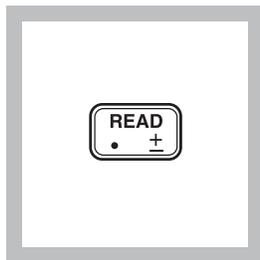


**20.** Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



**21.** Tightly cover the vial assembly with the instrument cap.



**22.** Press: **READ**  
The cursor will move to the right, then the result in mg/L C will be displayed.

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### Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

## ORGANIC CARBON, TOTAL, High Range, continued

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### Accuracy Check

#### Standard Solutions Method

- a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.  
Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).
- b. Prepare a 300 mg/L C standard by transferring 15.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

#### Standard Additions Method

- a. Prepare a 300 mg/L C standard by transferring 18.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.
- b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.
- c. Add the contents of one TOC Persulfate powder pillow to each vial.
- d. Add 0.3 mL of sample to each vial. Swirl to mix.
- e. Proceed with the procedure starting at *step 8*.
- f. The mg/L C concentration should increase by 100 mg/L for each 0.1 mL increment.

## ORGANIC CARBON, TOTAL, High Range, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 360 mg/L C and one lot of reagents, a single operator obtained a standard deviation of  $\pm 8$  mg/L C.

#### Estimated Detection Limit

Use Method Number 10129 to test TOC levels below 20 mg/L C.

#### Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 6 mg/L C.

### Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br <sub>2</sub>
Calcium	2000 mg/L as CaCO <sub>3</sub>
Chloride	5000 mg/L
Chlorine	10 mg/L Cl <sub>2</sub>
Chlorine Dioxide	6 mg/L ClO <sub>2</sub>
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as CaCO <sub>3</sub>
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH <sub>2</sub> Cl as Cl <sub>2</sub>
Nitrite	500 mg/L NO <sub>2</sub> <sup>-</sup>
Ozone	2 mg/L O <sub>3</sub>
Phosphate	3390 mg/L PO <sub>4</sub> <sup>3-</sup>

## ORGANIC CARBON, TOTAL, High Range, continued

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO <sub>2</sub>
Sulfate	5000 mg/L SO <sub>4</sub> <sup>2-</sup>
Sulfide	20 mg/L S <sup>2-</sup>
Sulfite	50 mg/L SO <sub>3</sub> <sup>2-</sup>
Zinc	5 mg/L

If the sample contains greater than 600 mg/L CaCO<sub>3</sub> alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 900 NTU have been tested without interference.

### Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

### REQUIRED REAGENTS

Total Organic Carbon Direct Method High Range

Test 'N Tube Reagent Set..... 50 vials..... 27604-45

#### Includes:

Description	Qty/Test	Unit	Cat. No.
Acid Digestion Solution Vials, High Range TOC .....	1 .....	50/pkg .....	*
Buffer Solution, Sulfate .....	0.4 mL .....	25 mL.....	452-33
Funnel, micro .....	1 .....	each.....	25843-35
Indicator Ampules, High Range TOC .....	1 .....	10/pkg.....	*
TOC Persulfate Powder Pillows .....	1 .....	50/pkg.....	*
Water, organic-free** .....	0.3 mL .....	500 mL.....	26415-49

\* These items are not sold separately.

## ORGANIC CARBON, TOTAL, High Range, continued

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### REQUIRED APPARATUS

Cylinder, graduated, 10-mL .....	1.....each .....	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....	LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....	LTV082.52.40001	
Flask, Erlenmeyer, 50-mL.....	1.....each .....	505-41
Magnetic Stirrer, 115 V, 4" x 4" .....	1.....each .....	28812-00
Safety Shield, laboratory bench .....	1.....each .....	50030-00
Test Tube Rack.....	1-3.....each .....	18641-00
Pipet, TenSette <sup>®</sup> , 0.1 to 1.0 mL.....	1.....each .....	19700-01
Pipet, TenSette <sup>®</sup> , 1.0 to 10.0 mL.....	1.....each .....	19700-10
Pipet Tips, for 19700-01 TenSette <sup>®</sup> Pipet.....	2.....50/pkg .....	21856-96
Pipet Tips, for 19700-10 TenSette <sup>®</sup> Pipet.....	2.....50/pkg .....	21997-96
Stir Bar, Magnetic .....	1.....each .....	45315-00
Wipes, Disposable, Kimwipes.....	1.....280/pkg .....	20970-00

### OPTIONAL REAGENTS

Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules.....	16/pkg .....	28335-10
Potassium Acid Phthalate.....	500 g .....	315-34
Sulfuric Acid Reagent Solution, 5.25 N.....	100 mL MDB .....	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C).....	5/pkg .....	27915-05
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28331-49

### OPTIONAL APPARATUS

Analytical Balance .....	each .....	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.52.30001	
Flask, volumetric, 1000-mL .....	each .....	14574-53
Flask, volumetric, 100-mL .....	each .....	14574-42
Pipet, Class A, 10.00-mL .....	each .....	14515-38
Pipet, Class A, 15.00-mL .....	each .....	14515-39

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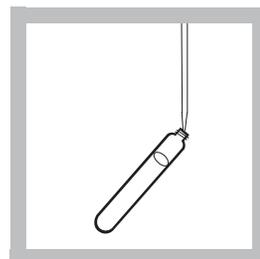
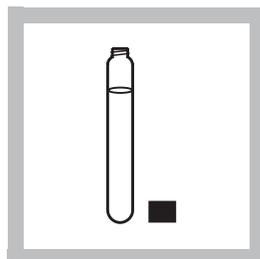
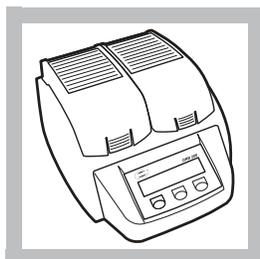
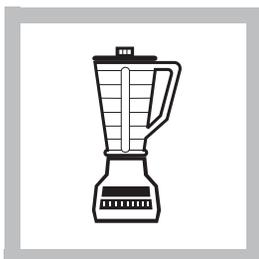
\*\* This item must be purchased separately.



# OXYGEN DEMAND, CHEMICAL

Method 8000  
For water, wastewater and  
seawater

## Reactor Digestion Method\* USEPA approved for reporting wastewater analysis\*\* Digestion



**1.** Homogenize 500 mL of sample for 2 minutes in a blender.

*Note: For the 0-15,000 mg/L range, homogenize 100 mL of sample. Pour the blended sample into a 250-mL beaker. Stir with a magnetic stirrer while withdrawing a sample aliquot. This improves accuracy and reproducibility.*

**2.** Turn on the DRB 200 Reactor. Preheat to 150 °C.

*Note: See DRB 200 user manual for selecting pre-programmed temperature applications.*

**3.** Remove the cap of a COD Digestion Reagent Vial for the appropriate range:

Sample Conc. Range (mg/L)	COD Digestion Reagent Vial Type
0 to 150	Low Range
0 to 1500	High Range
0 to 15,000	High Range Plus

*Note: The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The light striking the vials during the test will not affect results.*

**4.** Hold the vial at a 45-degree angle. Pipet 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) of sample into the vial.

*Note: For the 0-15,000 mg/L range, pipet only 0.20 mL of sample, not 2.00 mL of sample, using a TenSette Pipet. For greater accuracy analyze a minimum of three replicates and average the results.*

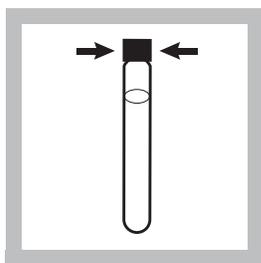
*Note: Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If spills occur, wash with running water.*

**Caution:** Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

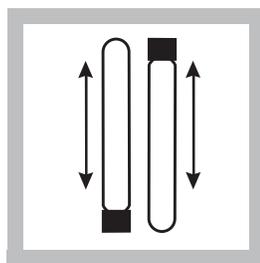
\* Jirka, A.M.; Carter, M.J. *Analytical Chemistry*, 1975, 47(8). 1397.

\*\* *Federal Register*, April 21, 1980, 45(78), 26811-26812. The 0-15,000 mg/L range is **not** USEPA approved.

## OXYGEN DEMAND, CHEMICAL, continued

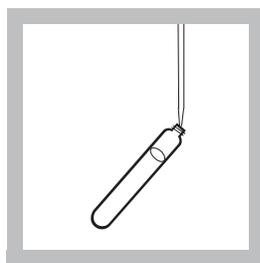


**5.** Replace the vial cap tightly. Rinse the outside of the COD vial with deionized water and wipe the vial clean with a paper towel.



**6.** Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated DRB 200 Reactor.

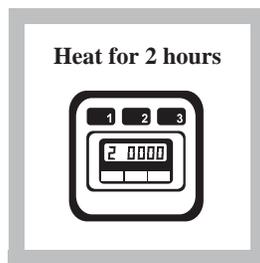
*Note:* The vial will become very hot during mixing.



**7.** Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) deionized water for the sample.

*Note:* Be sure the pipet is clean.

*Note:* One blank must be run with each set of samples. Run samples and blanks with vials from the same lot number (lot # is on the container label).

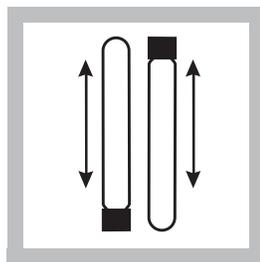


**8.** Heat the vials for 2 hours.

*Note:* Many samples are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until the reading remains unchanged. Cool vials to room temperature for final measurement.

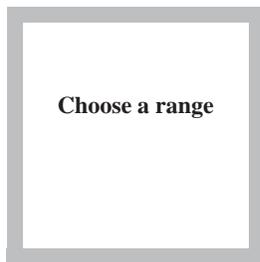


**9.** Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.



**10.** Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.

*Note:* If a pure green color appears in the reacted sample, measure the COD and, if necessary, repeat the test with a diluted sample.

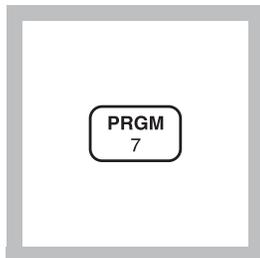


**11.** Use one of the following analytical techniques to measure the COD:

- Colorimetric method, 0-150 mg/L COD
- Colorimetric method, 0-1,500 mg/L COD
- Colorimetric method, 0-15,000 mg/L COD

## OXYGEN DEMAND, CHEMICAL, continued

### Colorimetric Determination, 0 to 150 mg/L COD

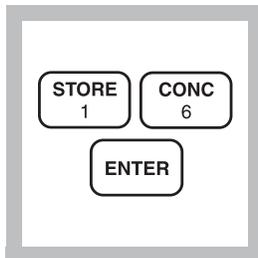


**1.** Enter the stored program number for chemical oxygen demand (COD), low range.

Press: **PRGM**

The display will show:

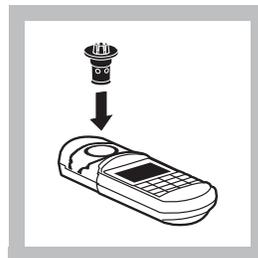
**PRGM ?**



**2.** Press: **16 ENTER**

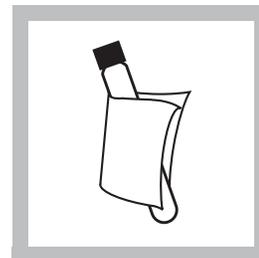
The display will show **mg/L, COD** and the **ZERO** icon.

*Note:* For alternate form ( $O_2$ ), press the **CONC** key.

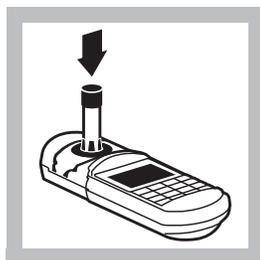


**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note:* For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.



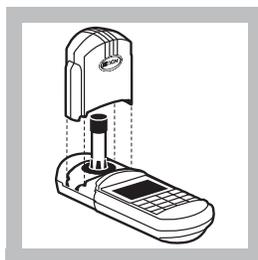
**4.** Clean the outside of the blank with a towel. *Note:* Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.



**5.** Place the blank in the adapter.

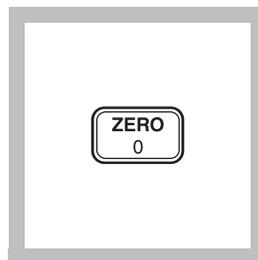
Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**6.** Tightly cover the vial with the instrument cap.

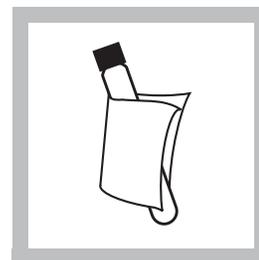
*Note:* The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.



**7.** Press: **ZERO**

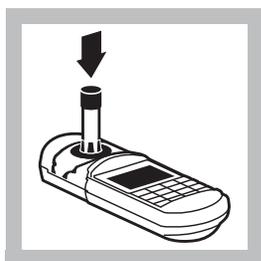
The cursor will move to the right, then the display will show:

**0 mg/L COD**



**8.** Clean the outside of the sample vial with a towel.

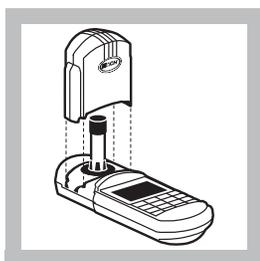
## OXYGEN DEMAND, CHEMICAL, continued



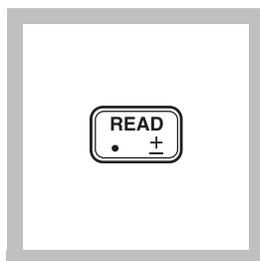
**9.** Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



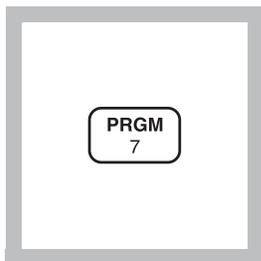
**10.** Tightly cover the vial with the instrument cap.



**11.** Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

### Colorimetric Determination, 0 to 1,500 and 0 to 15,000 mg/L COD

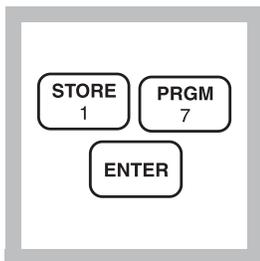


**1.** Enter the stored program number for chemical oxygen demand, high range.

Press: **PRGM**

The display will show:

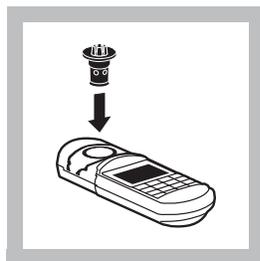
**PRGM ?**



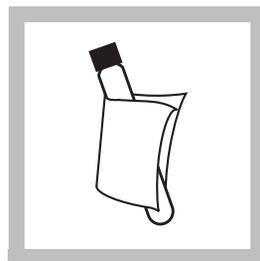
**2.** Press: **17 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon.

*Note: For alternate form (O<sub>2</sub>), press the **CONC** key.*



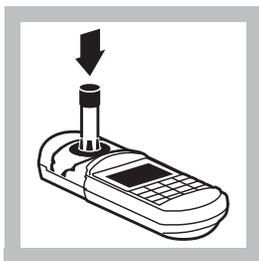
**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



**4.** Clean the outside of the blank with a towel.

*Note: Wiping with a damp towel followed by a dry one will remove fingerprints or other marks.*

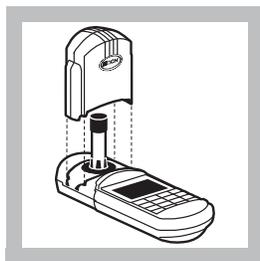
## OXYGEN DEMAND, CHEMICAL, continued



**5.** Place the blank in the adapter.

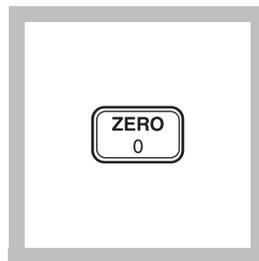
Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**6.** Tightly cover the sample cell with the instrument cap.

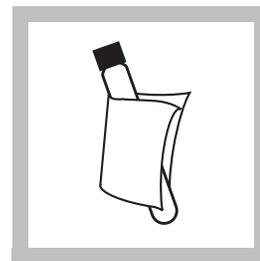
*The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.*



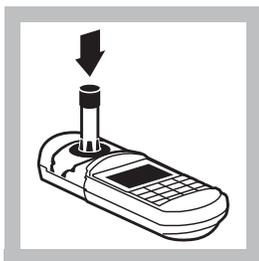
**7.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0 mg/L COD**



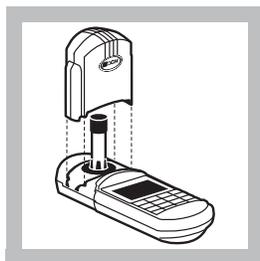
**8.** Clean the outside of the sample vial with a towel.



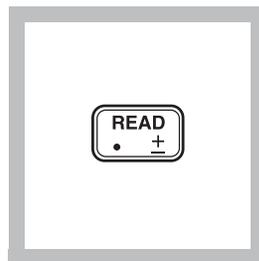
**9.** Place the sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**10.** Tightly cover the sample cell with the instrument cap.



**11.** Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

*Note:* When using High Range Plus COD Digestion Reagent Vials, multiply the reading by 10.

*Note:* For most accurate results with samples near 1,500 or 15,000 mg/L COD, repeat the analysis with a diluted sample.

## OXYGEN DEMAND, CHEMICAL, continued

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### Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see *Correction for Volume Additions* (Section 1) for more information.

### Accuracy Check

#### Standard Solution Method

Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2.0 mL as the sample volume. The expected result will be 100 mg/L COD. As an alternative, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to make a 100-mg/L standard.

Check the accuracy of the 0 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Alternatively, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water. Use 2.0 mL of one of these solutions as the sample volume.

Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

### Method Performance

#### Precision

Program #16: In a single laboratory, using a standard solution of 100 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 2$  mg/L COD.

Program #17: In a single laboratory, using a standard solution of 1000 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 16$  mg/L COD. For more information on Hach's precision statement, see *Section 1*.

## OXYGEN DEMAND, CHEMICAL, continued

**Estimated Detection Limit (EDL)** The EDL for program 16 is 4 mg/L COD. The EDL for program 17 is 30 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

**Alternate reagents** Mercury-free COD2 Reagents can provide a mercury-free testing option for non-reporting purposes. For process control applications, COD2 reagents will eliminate mercury waste and save on disposal costs. These reagents are fully compatible with test procedures and calibration curves programmed into the DR/2400 spectrophotometer. Determine chloride and ammonia for accurate results.

*Note: Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.*

### Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to the level specified in column 1 in *Table 1*. Samples with higher chloride concentrations should be diluted. Dilute the sample enough to reduce the chloride concentration to the level given in column 2.

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ( $\text{HgSO}_4$ ) to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to the level given in column 3.

**Table 1**

	Column 1	Column 2	Column 3
Vial Type Used	Maximum $\text{Cl}^-$ concentration in sample (mg/L)	Maximum $\text{Cl}^-$ concentration of diluted samples (mg/L)	Maximum $\text{Cl}^-$ concentration in sample when 0.50 $\text{HgSO}_4$ added (mg/L)
Low Range	2000	1000	8000
High Range	2000	1000	4000
High Range Plus	20,000	10,000	40,000

## OXYGEN DEMAND, CHEMICAL, continued

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### Blanks for Colorimetric Determination

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (420 or 610 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of deionized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

### Summary of Method

The mg/L COD results are defined as the mg of O<sub>2</sub> consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion (Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup>) to green chromic ion (Cr<sup>3+</sup>). When the 0-150 mg/L colorimetric method is used, the amount of Cr<sup>6+</sup> remaining is determined. When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr<sup>3+</sup> produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex the chloride interference.

### Pollution Prevention and Waste Management

Final samples will contain mercury (D009), silver (D011), and chromium (D007) at concentration levels regulated by the Federal RCRA. Please see *Section 3* for further information on proper disposal of these materials.

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### REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Low Range, 0 to 150 mg/L COD.....	1 to 2 vials .....	25/pkg.....	21258-25
High Range, 0 to 1,500 mg/L COD .....	1 to 2 vials .....	25/pkg.....	21259-25
High Range Plus, 0 to 15,000 mg/L COD .....	1 to 2 vials .....	25/pkg.....	24159-25
Water, deionized.....	varies .....	4 L.....	272-56

### REQUIRED APPARATUS

Blender, Osterizer, 120 V, 14 speed.....	1 .....	each.....	26160-00
Blender, Osterizer, 240 V, 14 speed.....	1 .....	each.....	26160-02
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	

## OXYGEN DEMAND, CHEMICAL, continued

### REQUIRED APPARATUS (continued)

Description	Qty/Test	Unit	Cat. No.
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....			LTV082.52.40001
COD/TNT Adapter.....	1.....	each .....	48464-00
Pipet, TenSette, 0.1 to 1.0 mL.....	1.....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	1.....	50/pkg .....	21856-96
Pipet, volumetric, Class A, 2.00 mL .....	1.....	each .....	14515-36
Pipet Filler, safety bulb .....	1.....	each .....	14651-00
Test Tube Rack.....	1 to 2 racks .....	each .....	18641-00

### ALTERNATE REAGENTS\*

COD2, LR, 0 to 150 mg/L COD .....	1-2 vials .....	25/pkg .....	25650-25
COD2, HR, 0 to 1500 mg/L COD.....	1-2 vials .....	25/pkg .....	25651-25
COD2, HR, 0 to 1500 mg/L COD.....	1-2 vials .....	150/pkg .....	25651-15
COD2, HR, 0 to 15,000 mg/L COD.....	1-2 vials .....	25/pkg .....	28343-25

### OPTIONAL REAGENTS

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 0 to 150 mg/L COD .....	150/pkg .....	21258-15
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD .....	150/pkg .....	21259-15
COD Standard Solution, 300 mg/L .....	200 mL .....	12186-29
COD Standard Solution, 1000 mg/L .....	200 mL .....	22539-29
Mercuric Sulfate.....	28.3 grams .....	1915-20
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules.....	16/pkg .....	28335-10
Potassium Acid Phthalate, ACS .....	500 g .....	315-34
Potassium Dichromate Standard Solution, 0.25 N.....	1000 mL* .....	1809-53
Sulfuric Acid, ACS .....	500 mL** .....	979-49
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28331-49

### OPTIONAL APPARATUS

Balance, analytical, 115 V.....	each .....	28014-01
Balance, analytical, 230 V.....	each .....	28014-02
Beaker, 250 mL .....	each .....	500-46H
Cylinder, graduated, 5 mL.....	each .....	508-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....		LTV082.53.30001

\* Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.

\*\* Contact Hach for larger sizes.

## OXYGEN DEMAND, CHEMICAL, continued

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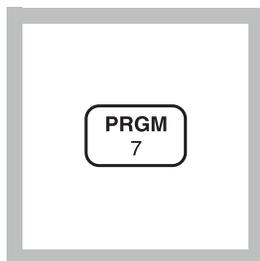
### OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm .....	LTV082.52.30001	
Electromagnetic Stirrer, 120 V, with electrode stand.....	each.....	45300-01
Electromagnetic Stirrer, 230 V, with electrode stand.....	each.....	45300-02
Flask, volumetric, Class A, 1000 mL .....	each.....	14574-53
Flask, volumetric, Class A, 100 mL .....	each.....	14574-42
pH Paper, 1 to 11 pH units.....	5 rolls/pkg.....	391-33
Pipet, serological, 5 mL .....	each.....	532-37
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 10 mL.....	each.....	14515-38
Spoon, measuring, 0.5 g.....	each.....	907-00
Stir Bar, 22.2 x 4.76 mm (7/8" x 3/16").....	each.....	45315-00
Stir Bar Retriever .....	each.....	15232-00
Timer .....	each.....	26304-00

#### *For Technical Assistance, Price and Ordering*

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

**OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater****Manganese III Digestion Method\* (without chloride removal)**

**1.** Enter the stored program number for Manganese III COD.

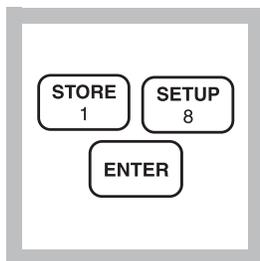
Press: **PRGM**

The display will show:

**PRGM ?**

*Note: If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

*Note: Preheat the COD Reactor to 150 °C for use later in the procedure.*



**2.** Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon.

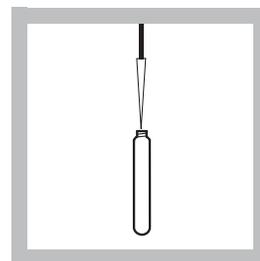
*Note: For alternate forms (O<sub>2</sub>), press the **CONC** key.*



**3.** Homogenize 100 mL of sample for 30 seconds in a blender.

*Note: Blending promotes even distribution of solids and improves accuracy and reproducibility.*

*Note: Continue mixing the sample while pipetting if suspended solids are present.*



**4.** If chloride is not present in significant amounts<sup>†</sup>, pipet 0.50 mL of homogenized sample into a Mn III COD vial. Cap and invert several times to mix.

*Note: If the sample COD value is not between 20-1000 mg/L dilute the sample with deionized water to obtain a range of 20-1000 mg/L COD. Multiply the final result by the dilution factor.*

<sup>†</sup> To determine if chloride will interfere, run the sample with and without the chloride removal procedure and compare the results.

**Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.**

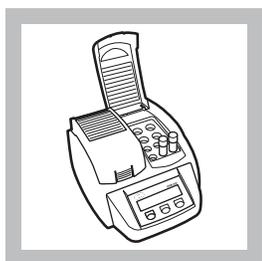
\* U.S. Patent 5,556,787

## OXYGEN DEMAND, CHEMICAL, continued



**5.** Prepare a blank (see note) by substituting 0.50 mL of deionized water for the sample. Continue with step 9 of this procedure.

*Note: The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range should be about 1.36-1.43.*

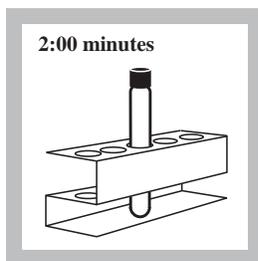


**6.** Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

*Note: Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

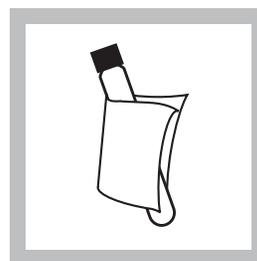
*Note: Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

*Note: See DRB 200 user manual for selecting pre-programmed temperature applications.*



**7.** Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

*Note: Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*



**8.** Remove the vials from the water and wipe with a clean, dry paper towel.

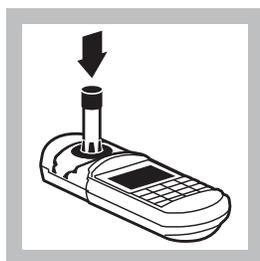
Invert the vials several times to mix.

## OXYGEN DEMAND, CHEMICAL, continued



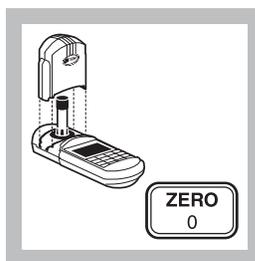
**9.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note:* For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.



**10.** Place the blank in the sample cell adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



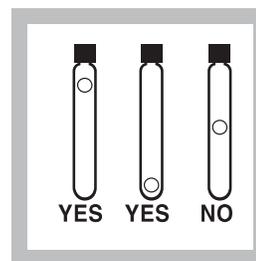
**11.** Tightly cover the sample cell with the instrument cap.

*Note:* Clean the COD vial with a towel to remove fingerprints or other marks.

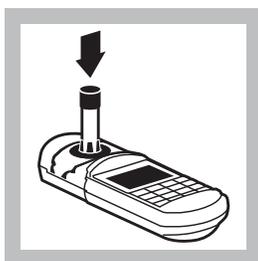
Press: **ZERO**

The cursor will move to the right, then the display will show:

**0 mg/L COD**

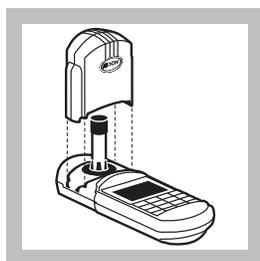


**12.** If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



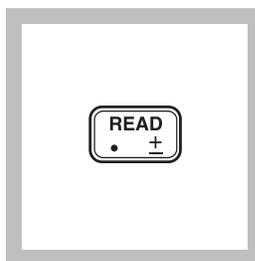
**13.** Place the sample in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**14.** Tightly cover the sample cell with the instrument cap.

*Note:* Clean the COD vial with a towel to remove fingerprints or other marks.



**15.** Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

*Note:* Adjust the result for any sample dilution in Steps 4 or 6.

## OXYGEN DEMAND, CHEMICAL, continued

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### Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions (Section 1)* for more information.

### Accuracy Check

#### Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be 800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

### Method Performance (for Manganic III COD without the chloride removal procedure)

#### Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

#### Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

## OXYGEN DEMAND, CHEMICAL, continued

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routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

### Summary of Method

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

## OXYGEN DEMAND, CHEMICAL, continued

### REQUIRED REAGENTS

Description	Quantity Required		
	Per Test	Unit	Cat. No.
Manganese III COD Reagent Vials, 20-1000 mg/L .....	1 .....	25/pkg.....	26234-25
Sulfuric Acid, concentrated .....	1 mL.....	4 Kg.....	979-09
Water, deionized.....	varies .....	4 L.....	272-56

### REQUIRED APPARATUS

Adapter, COD/TNT .....	1 .....	each.....	48464-00
Blender, Osterizer, 120 Vac, 14-speed.....	1 .....	each.....	26747-00
Blender Container, 118 mL.....	1 .....	2/pkg.....	26748-00
Cap, with inert Teflon liner, for mixing bottle.....	varies .....	12/pkg.....	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....		LTV082.52.40001	
Forceps, extra fine point .....	1 .....	each.....	26696-00
Mixing Bottle, glass, for sample + acid.....	1 .....	each.....	24276-06
Pipet, TenSette, 1.0 to 10.0 mL.....	1 .....	each.....	19700-10
Pipet Tips, for 19700-10 TenSette .....	2.....	250/pkg.....	21997-25
Pipet, TenSette, 0.1 to 1.0 mL.....	1 .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette .....	2.....	1000/pkg.....	21856-28
Test Tube Rack, stainless steel.....	1 .....	each.....	18641-00

### OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD .....	200 mL.....	26726-29	
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules .....	16/pkg.....	28335-10	
Potassium Acid Phthalate .....	500 g.....	315-34	
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL.....	28332-49	
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL.....	28331-49	

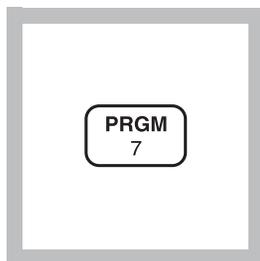
### OPTIONAL APPARATUS

Dispenser for sulfuric acid.....	each.....	25631-37	
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....		LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm .....		LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm .....		LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm .....		LTV082.52.30001	

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater****Manganese III Digestion Method\* (with chloride removal)**

**1.** Enter the stored program number for Manganese III COD.

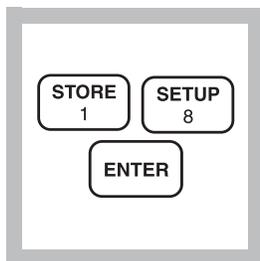
Press: **PRGM**

The display will show:

**PRGM ?**

*Note: If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

*Note: Preheat the COD Reactor to 150 °C for use later in the procedure.*



**2.** Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon.

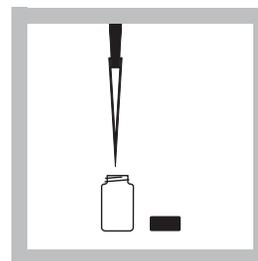
*Note: For alternate forms (O<sub>2</sub>), press the **CONC** key.*



**3.** Homogenize 100 mL of sample for 30 seconds in a blender.

*Note: Blending promotes even distribution of solids and improves accuracy and reproducibility.*

*Note: Continue mixing the sample while pipetting if suspended solids are present.*



**Chloride Removal Procedure**

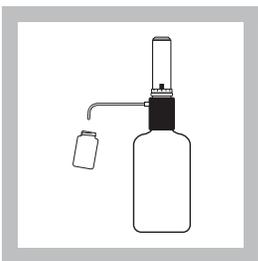
**4.** Using a TenSette Pipet or a pipet and safety bulb, pipet 9.0 mL of homogenized sample into an empty glass mixing cell. If the sample COD exceeds 1000 mg/L, dilute the sample as described in Table 1.

*Note: If suspended solids are present, continue mixing the sample while pipetting.*

**Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.**

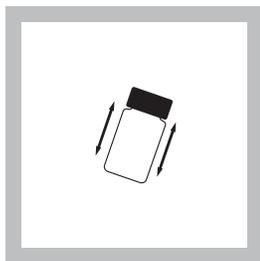
\* U.S. Patent 5,556,787

## OXYGEN DEMAND, CHEMICAL, continued



**5.** Using an automatic dispenser or TenSette Pipet, add 1.0 mL of concentrated sulfuric acid to the mixing cell.

*Note:* Mixing concentrated sulfuric acid and water is not additive. Adding 1.0 mL of concentrated sulfuric acid to 9.0 mL of sample does not result in a final volume of 10.0 mL. This factor is built into the calibration curve.



**6.** Cap the cell tightly and invert it several times. The solution will become hot. Cool to room temperature before proceeding.

*Note:* Acidified samples are stable for several months when refrigerated at 4 °C.

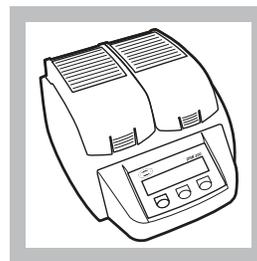


**7.** Prepare a blank (see note) by repeating Steps 4-6, substituting 9.0 mL of deionized water for the sample.

*Note:* The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range, when using chloride removal, should be about 1.31-1.36.

*Note:* Use a clean pipet or rinse it thoroughly.

*Note:* One blank must be run with each lot of reagents. Run all samples and blanks with the same lot of vials (lot number is on the container label).



**8.** If not already on, turn on the DRB 200 Reactor and heat to 150 °C.

*Note:* See DRB 200 user manual for selecting pre-programmed temperature applications.

## OXYGEN DEMAND, CHEMICAL, continued

**Table 1 Dilution Table (for use with Chloride Removal Procedure Only)**

Sample (mL)	Deionized Water (mL)	Range (mg/L COD)	Multiplication Factor
6.0	3.0	30-1500	1.5
3.0	6.0	60-3000	3
1.0	8.0	180-9000	9
0.5	8.5	360-18000	18

All dilutions require that the ratio of sample to sulfuric acid remain at 9:1. For other dilutions that are not listed in Table 1, simply add the sample volume + deionized water and divide by the sample volume to obtain the multiplication factor.

**Example:**

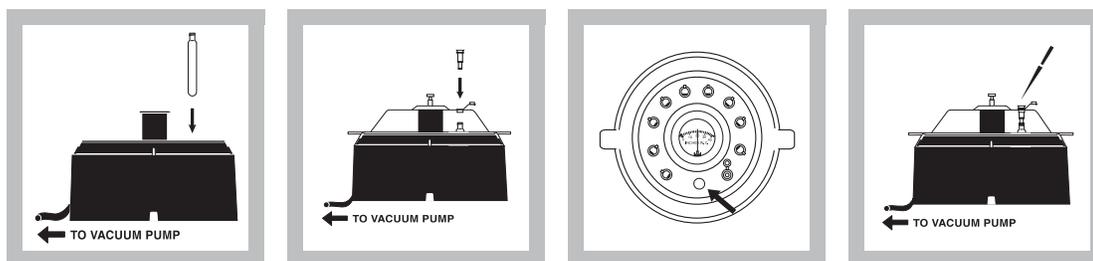
Dilute the sample to a range of 90-4500 mg/L COD

$$\text{Sample Volume (2.0 mL) + Deionized water (7.0 mL) = Total Volume (9.0 mL)}$$

$$\text{Multiplication Factor} = \frac{\text{Total Volume}}{\text{Sample Volume}} = \frac{9.0 \text{ mL}}{2.0 \text{ mL}} = 4.5$$

Standard test range is 20-1000 mg/L COD. Example Test Range = 4.5 (20) to 4.5 (1000) = 90-4500 mg/L COD

It is best to use 0.5 mL or more of sample for diluting. If sample values exceed 18,000 mg/L COD, use a separate sample dilution before the sample chloride removal procedure.



**9.** Label each Mn III COD vial and remove the cap. Place the vial in one of the numbered holes in the Vacuum Pretreatment Device (VPD)\* base.

*Note: The VPD must be attached to a vacuum pump (not an aspirator-type vacuum) that can create a vacuum of 20 to 25 inches of mercury.*

**10.** Place the VPD top on the base. Insert a fresh Chloride Removal Cartridge (CRC)\*\* directly above each Mn III COD Reagent Vial. Plug any open holes in the VPD top using the stoppers provided.

**11.** Turn the vacuum pump on and adjust the vacuum regulator valve on top of the VPD until the internal gauge reads 20 inches of water.

*Note: The optimum setting allows the sample to flow through the CRC in about 30 to 45 seconds.*

**12.** Pipet 0.60 mL of acidified sample (made in Steps 4-6) into the CRC. Pipet 0.60 mL of acidified blank into another CRC.

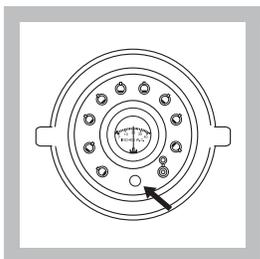
*Note: If the sample does not flow through the CRC, increase the vacuum until flow starts, then reduce the vacuum to 20 inches of water. Proceed as usual.*

\* Patent Pending.

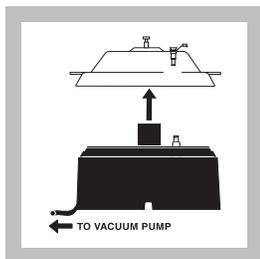
\*\* U.S. patents 5,667,754 and 5,683,914.

## OXYGEN DEMAND, CHEMICAL, continued

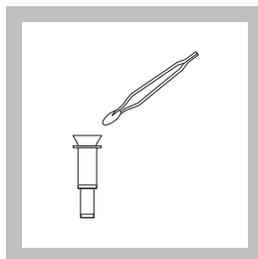
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**13.** Close the vacuum regulator valve completely to achieve full vacuum. After one minute under full vacuum, slide the VPD back and forth several times to dislodge any drops clinging to the cartridge.



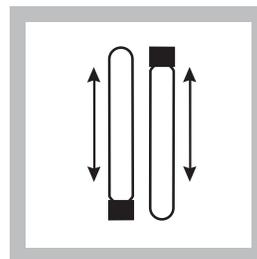
**14.** Open the VPD regulator valve to release the vacuum. Turn the pump off. Remove the VPD top and set it beside the base.



**15.** Use forceps to remove the filter from the top of each CRC. Place each filter in the corresponding Mn III COD Vial (use the numbers on the VPD as a guide).

*Note:* If the sample does not contain suspended solids, it is not necessary to transfer the filter to the digestion vial.

*Note:* To avoid cross contamination, clean forceps tips between samples by wiping with a clean towel or rinsing with deionized water.



**16.** Remove the Mn III COD vial from the vacuum chamber and replace the original cap. Screw the cap on tightly. Invert several times to mix.

*Note:* Dispose of the used Chloride Removal Cartridge. Do not reuse it.

## OXYGEN DEMAND, CHEMICAL, continued

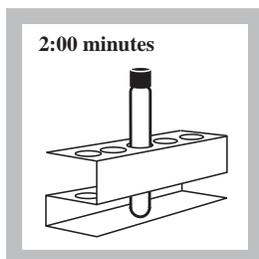


**17.** Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

*Note: Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

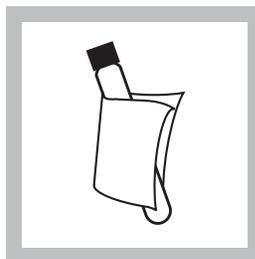
*Note: Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

*Note: See DRB 200 user manual for selecting pre-programmed temperature applications.*



**18.** Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

*Note: Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*



**19.** Remove the vials from the water and wipe with a clean, dry paper towel.

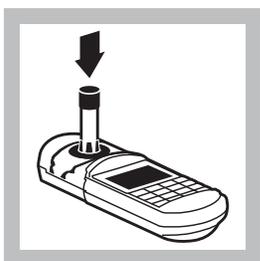
Invert the vials several times to mix.



**20.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

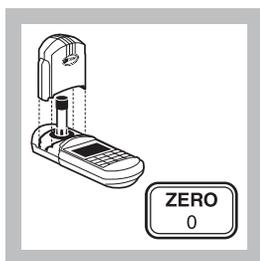
*Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

## OXYGEN DEMAND, CHEMICAL, continued



**21.** Place the blank in the sample cell adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



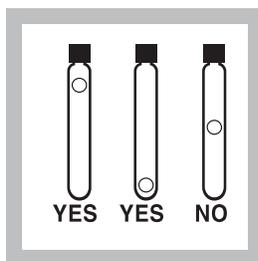
**22.** Tightly cover the sample cell with the instrument cap.

*Note:* Clean the COD vial with a towel to remove fingerprints or other marks.

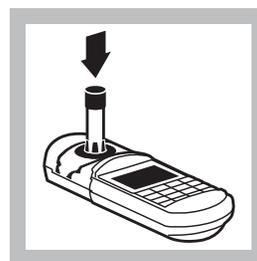
Press: **ZERO**

The cursor will move to the right, then the display will show:

**0 mg/L COD**



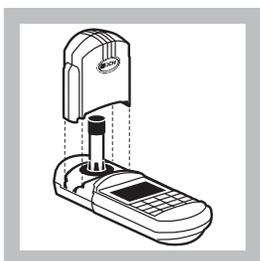
**23.** If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



**24.** Place the sample in the adapter.

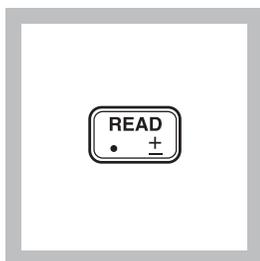
Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**25.** Tightly cover the sample cell with the instrument cap.

*Note:* Clean the COD vial with a towel to remove fingerprints or other marks.



**26.** Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

*Note:* Adjust the result for any sample dilution.

## OXYGEN DEMAND, CHEMICAL, continued

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### Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions (Section 1)* for more information.

### Accuracy Check

#### Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be 800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

### Method Performance (for Manganic III COD without the chloride removal procedure)

#### Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

#### Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

## **OXYGEN DEMAND, CHEMICAL, continued**

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routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

### **Summary of Method**

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

## OXYGEN DEMAND, CHEMICAL, continued

### REQUIRED REAGENTS

Description	Quantity Required		
	Per Test	Unit	Cat. No.
Chloride Removal Cartridges (CRC) .....	1 .....	25/pkg .....	26618-25
Manganese III COD Reagent Vials, 20-1000 mg/L.....	1 .....	25/pkg .....	26234-25
Sulfuric Acid, concentrated.....	1 mL .....	4 Kg .....	979-09
Water, deionized .....	varies .....	4 L .....	272-56

### REQUIRED APPARATUS

Adapter, COD/TNT .....	1 .....	each .....	48464-00
Blender, Osterizer, 120 Vac, 14-speed .....	1 .....	each .....	26747-00
Blender Container, 118 mL .....	1 .....	2/pkg .....	26748-00
Cap, with inert Teflon liner, for mixing bottle .....	varies .....	12/pkg .....	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....		LTV082.52.40001	
Forceps, extra fine point.....	1 .....	each .....	26696-00
Mixing Bottle, glass, for sample + acid .....	1 .....	each .....	24276-06
Pipet, TenSette, 1.0 to 10.0 mL.....	1 .....	each .....	19700-10
Pipet Tips, for 19700-10 TenSette.....	2 .....	250/pkg .....	21997-25
Pipet, TenSette, 0.1 to 1.0 mL.....	1 .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette.....	2 .....	1000/pkg .....	21856-28
Test Tube Rack, stainless steel .....	1 .....	each .....	18641-00
Vacuum Pretreatment Device (VPD) .....	1 .....	each .....	49000-00
Vacuum Pump, 115 V.....	1 .....	each .....	14697-00
Vacuum Pump, 230V.....	1 .....	each .....	14697-02

### OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD.....	200 mL .....	26726-29
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules.....	16/pkg .....	28335-10
Potassium Acid Phthalate.....	500 g .....	315-34
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28331-49

### OPTIONAL APPARATUS

Dispenser for sulfuric acid .....	each .....	25631-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.52.30001	

For Technical Assistance, Price and Ordering

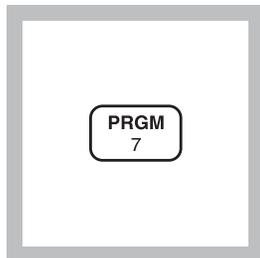
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**OXYGEN, DISSOLVED, High Range (0 to 15.0 mg/L O<sub>2</sub>)****HRDO Method**

For water and wastewater

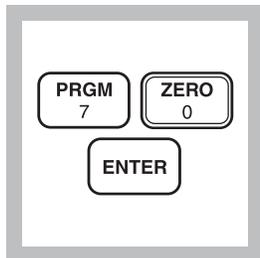


1. Enter the stored program number for dissolved oxygen, high range.

Press: **PRGM**

The display will show:

**PRGM ?**

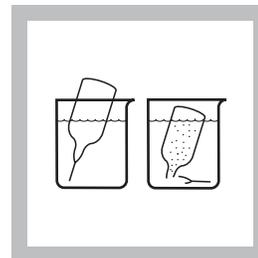


2. Press: **70 ENTER**

The display will show **mg/L, O<sub>2</sub>** and the **ZERO** icon.

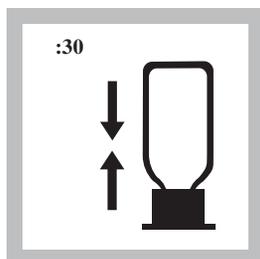


3. Fill a sample cell (the blank) with at least 10 mL of sample. Fill a blue ampul cap with sample. Collect at least 40 mL of sample in a 50-mL beaker.



4. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

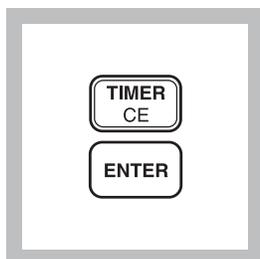
*Note: Keep the tip immersed while the ampul fills completely.*



5. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake for about 30 seconds.

*Note: Accuracy is not affected by undissolved powder.*

*Note: The cap prevents contamination with atmospheric oxygen.*

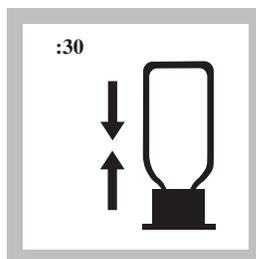


6. Press:

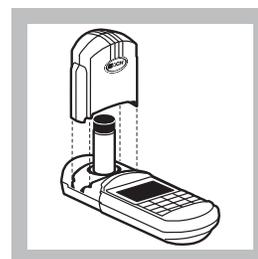
**TIMER ENTER**

A 2-minute reaction period will begin.

*Note: The two-minute period allows oxygen which was degassed during aspiration to redissolve in the sample and react.*



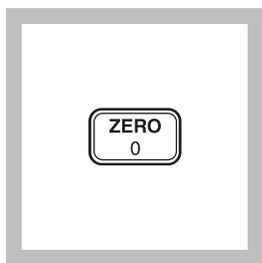
7. When the timer beeps, shake the ampul for 30 seconds.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## OXYGEN, DISSOLVED, High Range, continued

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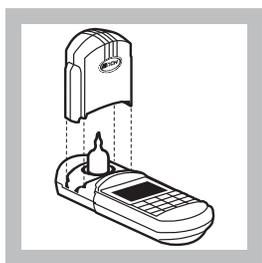


**9. Press: ZERO**

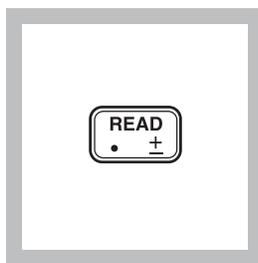
The cursor will move to the right, then the display will show:

**0.0 mg/L O<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**10. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap. Wait approximately 30 seconds for the air bubbles to disperse from the light path.**



**11. Press: READ**

The cursor will move to the right, then the result in mg/L O<sub>2</sub> will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

The main consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, it should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate over-all condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

## OXYGEN, DISSOLVED, High Range, continued

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### Accuracy Check

The results of this procedure may be compared with the results of a dissolved oxygen meter (Cat. No. 51815-01).

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 8.0 mg/L O<sub>2</sub> determined by the Winkler method and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.41 mg/L O<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit for program 70 is 0.10 mg/L O<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Cr <sup>3+</sup>	Greater than 10 mg/L
Cu <sup>2+</sup>	Greater than 10 mg/L
Fe <sup>2+</sup>	Greater than 10 mg/L
Mg <sup>2+</sup>	Magnesium is commonly present in seawater and causes a negative interference. If the sample contains more than 50% seawater, the oxygen concentration obtained by this method will be 25% less than the true oxygen concentration. If the sample contains less than 50% seawater, the interference will be less than 5%.
Mn <sup>2+</sup>	Greater than 10 mg/L
Ni <sup>2+</sup>	Greater than 10 mg/L
NO <sub>2</sub> <sup>-</sup>	Greater than 10 mg/L

### Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, a yellow color forms, which turns purple as the oxygen reacts with the reagent. The color developed is proportional to the concentration of dissolved oxygen.

## OXYGEN, DISSOLVED, High Range, continued

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### REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps .....	1 ampul .....	25/pkg.....	25150-25

### REQUIRED APPARATUS

Beaker, 50 mL.....	1 .....	each.....	500-41H
Caps, ampul, blue.....	varies .....	25/pkg.....	1731-25
Sample Cell, 10-20-25 mL, w/ cap.....	1 .....	6/pkg.....	24019-06

### OPTIONAL REAGENTS AND APPARATUS

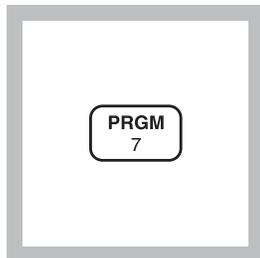
AccuVac Dissolved Oxygen Sampler .....		each.....	24051-00
AccuVac Snapper Kit.....		each.....	24052-00
AccuVac Drainer.....		each.....	41036-00
BOD bottle and stopper, 300 mL.....		each.....	621-00
Dissolved Oxygen Meter, Portable HQ 10 .....		each.....	51815-01
Dissolved Oxygen Reagent Set (Buret Method).....	100 tests.....		23514-00
Dissolved Oxygen Reagent Set (Digital Titrator Method) .....	50 tests.....		22722-00

Dissolved oxygen may also be determined by titrimetric methods.  
Request Publication 8042 for additional information.

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**OXYGEN, DISSOLVED, Low Range (0 to 1000 µg/L O<sub>2</sub>) For boiler feedwater****Indigo Carmine Method (Using AccuVac Ampuls)**

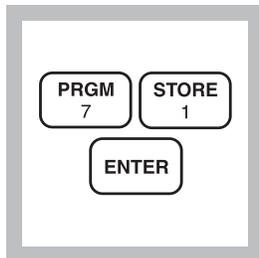
**1.** Enter the stored program number for low range dissolved oxygen (O<sub>2</sub>).

Press: **PRGM**

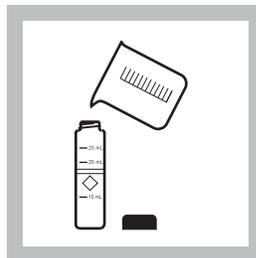
The display will show:

**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

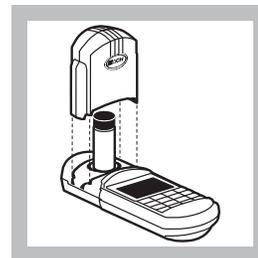


**2.** Press: **71 ENTER**  
The display will show **µg/L, O<sub>2</sub>** and the **ZERO** icon.

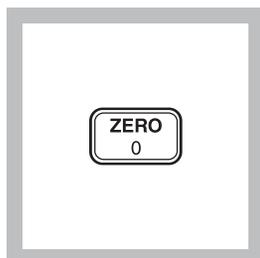


**3.** Fill a sample cell with at least 10 mL of sample (the blank).

*Note: Samples must be analyzed immediately and cannot be stored.*



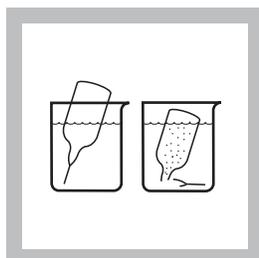
**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



**5.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

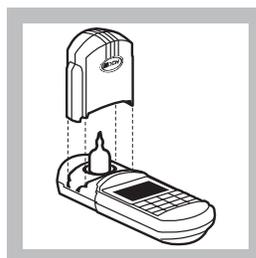
**0 µg/L O<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



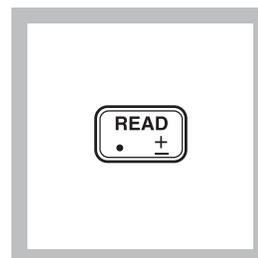
**6.** Collect at least 40 mL of sample in a 50-mL beaker. Fill a Low Range Dissolved Oxygen AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampul fills completely.*



**7.** Immediately place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

*Note: The ampuls will contain a small piece of wire to maintain reagent quality. The solution color will be yellow.*



**8.** Press: **READ**  
The cursor will move to the right, then the result in µg/L dissolved oxygen will be displayed.

*Note: Use the initial reading. The reading is stable for 30 seconds. After 30 seconds, the ampul solution will absorb oxygen from the air.*

## OXYGEN, DISSOLVED, Low Range, continued

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### Sampling and Storage

The main consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

### Accuracy Check

The reagent blank for this test can be checked by following these steps:

- a) Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.
- b) Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample and break the tip. Keep the tip immersed while the ampul fills completely.
- c) Determine the dissolved oxygen concentration according to the preceding procedure. The result should be  $0 \pm 1 \mu\text{g/L}$ .

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 500  $\mu\text{g/L O}_2$  and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 2 \mu\text{g/L O}_2$ . For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program #71 is 10  $\mu\text{g/L O}_2$ . For more information on the estimated detection limit, see *Section 1*.

## OXYGEN, DISSOLVED, Low Range, continued

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### Interferences

Interfering Substance	Interference Levels and Treatments
Hydrazine	100,000 fold excess will begin to reduce the oxidized form of the indicator solution.
Sodium hydrosulfite	Reduces the oxidized form of the indicator solution and will cause a significant interference.

Excess amounts of sodium thioglycolate, sodium ascorbate, sodium ascorbate + sodium sulfite, sodium ascorbate + cupric sulfate, sodium nitrite, sodium sulfite, sodium thiosulfate, and hydroquinone do not cause significant interference.

### Summary of Method

When the vacuum-sealed AccuVac ampul is broken open in a sample containing dissolved oxygen, the yellow reagent solution turns blue. The blue color is proportional to the dissolved oxygen concentration.

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### REQUIRED REAGENTS & APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Low Range Dissolved Oxygen AccuVac Ampuls...	1 ampul.....	25/pkg .....	25010-25
Beaker, 50 mL .....	1 .....	each .....	500-41H
Sample Cell, 10-20-25 mL, w/cap .....	1 .....	6/pkg .....	24019-06

### OPTIONAL REAGENTS AND APPARATUS

AccuVac Snapper Kit .....	each .....	24052-00
Sodium Hydrosulfite, technical grade .....	500 g .....	294-34

### *For Technical Assistance, Price and Ordering*

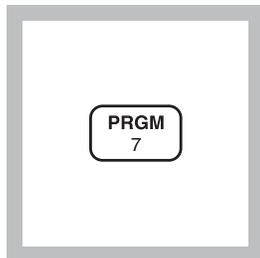
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**OZONE (0 to 0.25 mg/L O<sub>3</sub>, 0 to 0.75 mg/L O<sub>3</sub> or 0 to 1.50 mg/L O<sub>3</sub>)**

**Indigo Method (Using AccuVac Ampuls)**

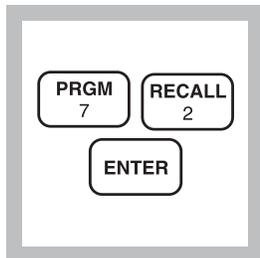


**1.** Enter the stored program number for Ozone (O<sub>3</sub>) AccuVac ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

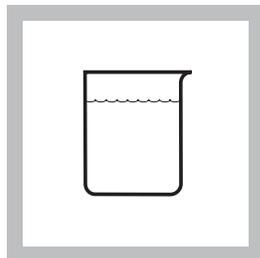


**2.** Press: **72 ENTER** for low range ozone

Press: **73 ENTER** for mid range ozone

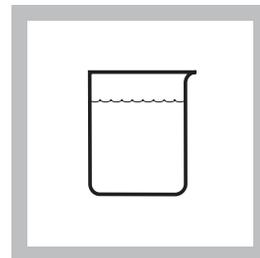
Press: **74 ENTER** for high range ozone.

The display will show **mg/L, O<sub>3</sub>** and the **ZERO** icon.



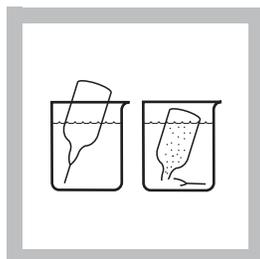
**3.** Gently collect at least 40 mL of sample in a 50-mL beaker.

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis. See Sampling and Storage following these steps for proper collection.*



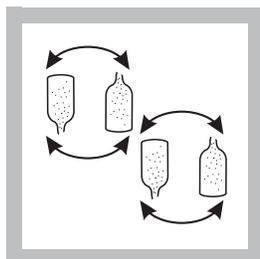
**4.** Collect at least 40 mL of ozone-free water (blank) in another 50-mL beaker.

*Note: Ozone-free water used for the blank may be deionized water or tap water.*



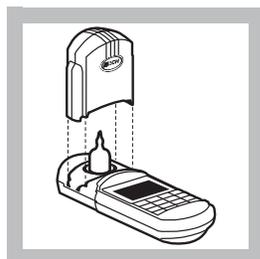
**5.** Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and one ampul with the blank.

*Note: Keep the tip immersed while the ampul fills.*



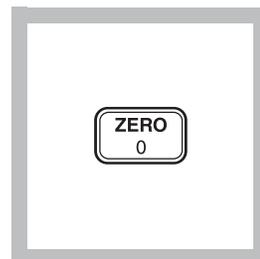
**6.** Quickly invert both ampuls several times to mix. Wipe off any liquid or fingerprints.

*Note: Part of the blue color will be bleached if ozone is present. (The sample will be lighter than the blank.)*



**7.** Place the **sample** AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

*Note: Standardization for this procedure is intentionally reversed.*

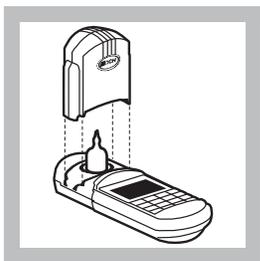


**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L O<sub>3</sub>**

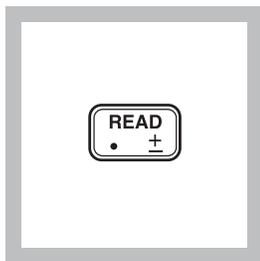
## OZONE, continued

---



**9.** Place the AccuVac ampul containing the **blank** into the cell holder. Tightly cover the ampul with the instrument cap.

*Note: Standardization for this procedure is intentionally reversed.*



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L ozone will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

---

## Sampling

The chief consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample or disturbing the sample by stirring or shaking will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

## Stability of Indigo Reagent

Indigo is light-sensitive. Therefore, the AccuVac Ampuls should be kept in the dark at all times.

However, the indigo solution decomposes slowly under room light after filling with sample. The blank ampul can be used for multiple measurements during the same day.

## Method Performance

### Precision

In a single laboratory, using standard solutions of 0.15, 0.28 and 0.96 mg/L ozone for the low, mid and high range, respectively, and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.01$ ,  $\pm 0.02$  and  $\pm 0.02$  mg/L O<sub>3</sub> for the low, mid and high range tests, respectively. For more information on Hach's precision statement, see *Section 1*.

## OZONE, continued

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### Estimated Detection Limit

The estimated detection limit for the programs #72, #73, and #74 is 0.02 mg/L O<sub>3</sub>. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The reagent formulation adjusts the sample pH to 2.5 after the ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone. The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent chlorine interference. No transfer of sample is needed in the procedure. Therefore, ozone loss due to sampling is eliminated.

---

### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Ozone AccuVac Ampuls				
Select one or more based on range:				
0-0.25 mg/L.....	2 ampuls.....	25/pkg.....		25160-25
0-0.75 mg/L.....	2 ampuls.....	25/pkg.....		25170-25
0-1.50 mg/L.....	2 ampuls.....	25/pkg.....		25180-25
Water, deionized.....	varies.....	4 L.....		272-56

### REQUIRED APPARATUS

Beaker, 50 mL.....	2.....	each.....		500-41H
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### OPTIONAL APPARATUS

AccuVac Snapper Kit.....		each.....		24052-00
AccuVac Ampule sampler.....		each.....		24051-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

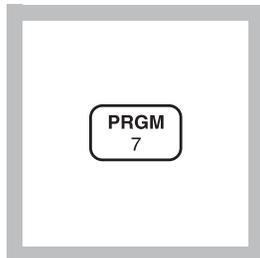
Outside the U.S.A.—Contact the Hach office or distributor serving you.



## pH (6.5 to 8.5 pH units)

## Colorimetric pH Determination Using Phenol Red

For water and wastewater

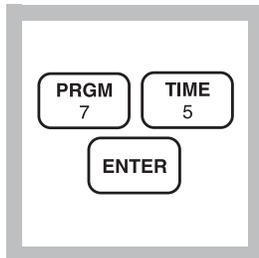


1. Enter the stored program number for the pH method.

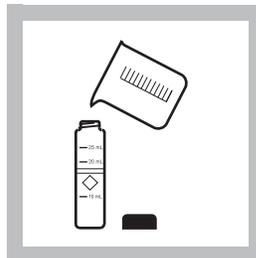
Press: **PRGM**

The display will show:

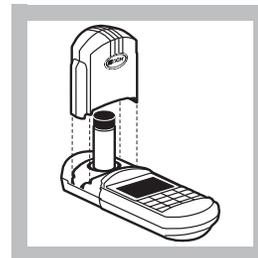
**PRGM ?**



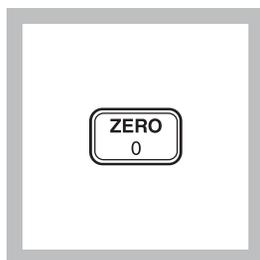
2. Press: **75 ENTER**  
The display will show **PH** and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample (the blank).

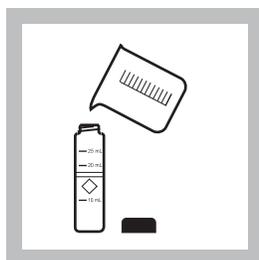


4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



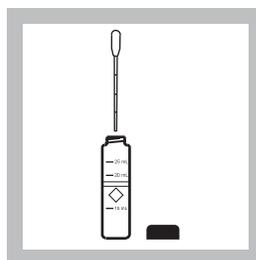
5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**6.0 PH**

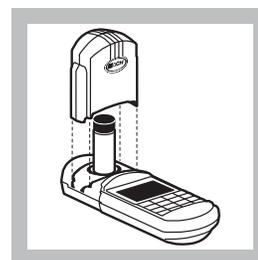


6. Fill another cell with 10 mL of sample.

*Note: Sample temperature must be 21-29 °C.*



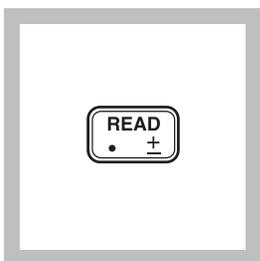
7. Using a disposable dropper, add 1 mL of Phenol Red Indicator Solution to the cell (the prepared sample). Cap the sample cell and invert twice to mix.



8. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

## pH, continued

---



### 9. Press: **READ**

The cursor will move to the right, then the result in pH units will be displayed.

*Note: Use of the Standard Adjust feature is highly recommended. See Accuracy Check.*

*Note: Any reading below 6.5 pH units will be erroneous.*

---

## Sampling and Storage

Analyze samples immediately for best results.

## Accuracy Check

### Standard Solution Method

Using a clear pH 7.0 buffer solution as the sample, perform the pH procedure as described above.

## Method Performance

### Precision

In a single laboratory using a standard solution of pH 7.0 and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 0.1 pH units.

### Estimated Detection Limit

The estimated detection limit for program 75 is a pH of 6.5.

## pH, continued

---

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 7.0 buffer solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **7.0** to edit the standard concentration to match that of the standard used. See *Section 1, Standard Curve Adjustment* for more information. Press **ENTER** to complete the curve adjustment.

### Interferences

Chlorine does not interfere at levels of 6 mg/L or lower.

Salt water (sea water) will interfere and cannot be analyzed using this method.

### Summary of Method

This method uses a sulfonphthalein indicator (Phenol Red) to determine pH colorimetrically. Phenol Red has a working range of pH 6.8 (yellow) to 8.2 (red).

---

## REQUIRED REAGENTS & APPARATUS

Description	Quantity Required		
	Per Test	Units	Cat. No.
Dropper, 0.5 & 1.0 mL marks .....	1 .....	20/pkg.....	21247-20
Phenol Red Indicator Solution, spec grade .....	1.0 mL.....	50 mL.....	26575-12
Sample Cells, 10-20-25 mL, w/ cap.....	2.....	6/pkg.....	24019-06

## OPTIONAL REAGENTS

pH 7.0 Buffer Solution .....	500 mL.....	12222-49
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## OPTIONAL APPARATUS

Description	Units	Cat. No.
Thermometer, -20 to 110 °C, Non-Mercury.....	each.....	26357-02

### *For Technical Assistance, Price and Ordering*

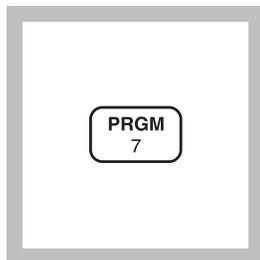
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**PHOSPHONATES (0-2.5 to 0-125 mg/L)**

For water, wastewater, and seawater

**Persulfate UV Oxidation Method\***

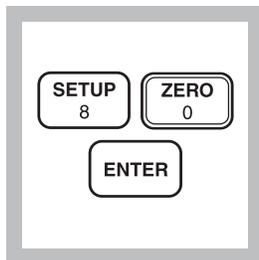
**1.** Enter the stored program number for phosphonates.

Press: **PRGM**

The display will show:

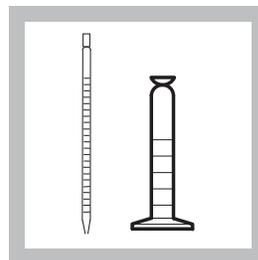
**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



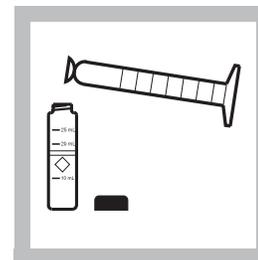
**2.** Press: **80 ENTER**

The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.



**3.** Choose the appropriate sample size from *Table 1* below. Pipet the chosen sample volume into a 50-mL graduated mixing cylinder. Dilute the sample to 50 mL with deionized water. Mix well.

*Note: Clean glassware with 1:1 hydrochloric acid, followed by a deionized water rinse. Do not use commercial detergents containing phosphates to clean glassware.*



**4.** Fill a sample cell to the 10-mL mark with diluted sample from Step 3 (label this as the blank).

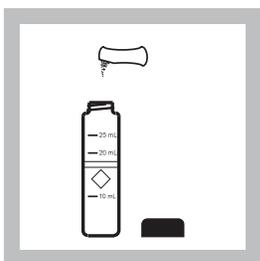
Fill another sample cell to the 25-mL mark with diluted sample from Step 3 (label this as the sample).

**Table 1**

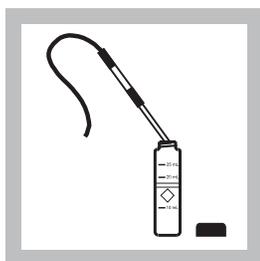
Expected Range (mg/L phosphonate)	Sample Volume (mL)
0-2.5	50
0-5	25
0-12.5	10
0-25	5
0-125	1

\* Adapted from Blystone, P.; Larson, P., *A Rapid Method for Analysis of Phosphonate Compounds*, International Water Conference, Pittsburgh, Pa. (Oct. 26-28, 1981).

## PHOSPHONATES, continued



**5.** Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the cell labeled as “sample”. Swirl to mix. This cell contains the prepared sample.

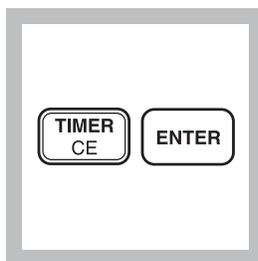


**6.** Insert the ultraviolet (UV) lamp into the prepared sample.

*Note:* Wear UV safety goggles while the lamp is on.

*Note:* Do not handle the lamp surface. Fingerprints will etch the glass. Wipe lamp with a soft, clean tissue between samples. Do not use detergents with phosphates to wash glassware.

*Note:* A specially designed cord adapter is available for performing two digestions with a single power supply. A second UV lamp is required.



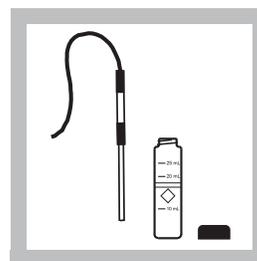
**7.** Turn on the UV lamp to digest the prepared sample.

Press: **TIMER ENTER**

A 10-minute reaction period will begin.

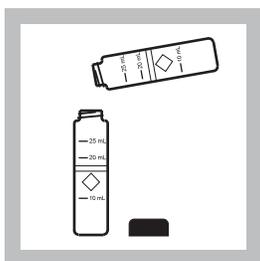
*Note:* Phosphonates are converted to orthophosphate in this step.

*Note:* The digestion step may take less time. Contaminated samples or a weak lamp could result in incomplete digestion. Check efficiency by running a longer digestion to see if readings increase.

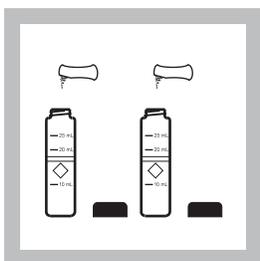


**8.** When the timer beeps, turn off the UV lamp. Remove it from the sample cell.

## PHOSPHONATES, continued

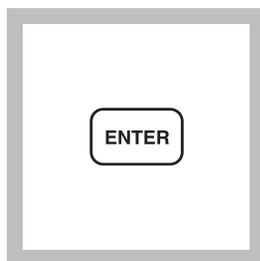


**9.** Pour 10 mL of sample from the cell labeled as “sample” into a second clean, dry sample cell. This is the prepared sample.



**10.** Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow for 10-mL samples to each sample cell. Swirl immediately to mix.

*Note:* A blue color will form if phosphate is present. Sample and blank cells may develop color.

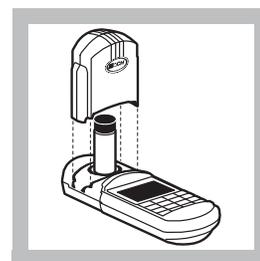


**11.** The display will show: **2:00 TIMER 2**

Press: **ENTER**

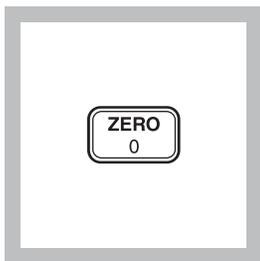
A two-minute reaction period will begin.

*Note:* If sample is colder than 15 °C, 4 minutes are required for color development.



**12.** When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note:* Perform Steps 12-15 within three minutes after the timer beeps.

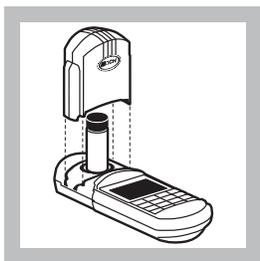


**13.** Press: **ZERO**

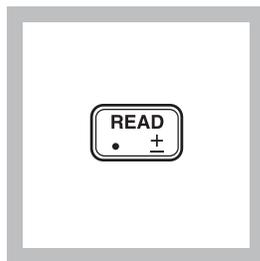
The cursor will move to the right, then the display will show:

**0.0 mg/L PO<sub>4</sub>**

*Note:* If Reagent Blank Correction is on, the display may flash “limit”. See Section 1.



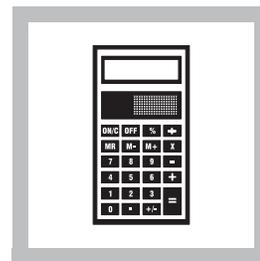
**14.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**15.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate will be displayed. Multiply this value by the appropriate multiplier from Table 2 to obtain the actual concentration of phosphonates as phosphate in the sample.

*Note:* Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).



**16.** Results may be expressed in terms of a specific active phosphonate by using the appropriate conversion factor and the equation found in Table 3.

## PHOSPHONATES, continued

Table 2

Sample Volume (mL) (chosen in Step 3)	Multiplier
50	0.1
25	0.2
10	0.5
5	1.0
1	5.0

Phosphate concentration = Instrument Reading x Multiplier

Table 3

Phosphonate Type	Conversion Factor
PBTC	2.84
NTP	1.050
HEDPA	1.085
EDTMPA	1.148
HMDTMPA	1.295
DETPMPA	1.207
HPA	1.49

Active Phosphonate (mg/L) = Phosphate concentration from Step 15 x Conversion Factor

### Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use a commercial detergent. If prompt analysis is impossible, adjust the pH to 2 or less with about 2 mL of sulfuric acid, ACS, per liter of sample. Store at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See *Section 1* for more information on dilution factors, cleaning instructions, etc.

### Accuracy Check

Ideally, a solution containing a known amount of the phosphonate product being used should be prepared. This will check the UV conversion of phosphonate to orthophosphate.

### Interferences

When testing a 5-mL sample volume, the following may interfere when present in concentrations exceeding those listed below:

The interference levels will decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10.00 mL, copper will begin to interfere above 50 mg/L.

## PHOSPHONATES, continued

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

Phosphites and organophosphorus compounds other than phosphonates react quantitatively. Meta and polyphosphates do not interfere.

Interfering Substance	Level	Interfering Substance	Level
Aluminum	100 mg/L	EDTA	100 mg/L
Arsenate	all levels	Iron	200 mg/L
Benzotriazole	10 mg/L	Nitrate	200 mg/L
Bicarbonate	1000 mg/L	NTA	250 mg/L
Bromide	100 mg/L	Orthophosphate	15 mg/L
Calcium	5000 mg/L	Silica	500 mg/L
CDTA	100 mg/L	Silicate	100 mg/L
Chloride	5000 mg/L	Sulfate	2000 mg/L
Chromate	100 mg/L	Sulfide	All levels
Copper	100 mg/L	Sulfite	100 mg/L
Cyanide <sup>1</sup>	100 mg/L	Thiourea	10 mg/L
Diethanoldithiocarbamate	50 mg/L		

<sup>1</sup> Increase the UV digestion to 30 minutes.

### Summary of Method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV catalyzed oxidation of phosphonate to orthophosphate. Range may be as low as 0 to 2.5 mg/L or as high as 0 to 125 mg/L.

Phosphonate is converted to orthophosphate during the UV digestion. Both the sample and the blank will develop color if orthophosphate is present in the sample. The increase in color in the sample is proportional to the phosphate produced in the digestion.

## PHOSPHONATES, continued

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### REQUIRED REAGENTS

Phosphonates Reagent Set (100 tests) ..... 24297-00  
Includes: (2) 21060-69, (1) 20847-69

Description	Quantity Required		Unit	Cat. No
	Per Test			
PhosVer 3 Phosphate Reagent Powder Pillows ....	2 pillows	.....	100/pkg	..... 21060-69
Potassium Persulfate Pillow for Phosphonate .....	1 pillow	.....	100/pkg	..... 20847-69
Water, deionized.....	varies	.....	4 L	..... 272-56

### REQUIRED APPARATUS

Cylinder, mixing, graduated, 50 mL.....	1	.....	each	..... 1896-41
Goggles, UV safety.....	1	.....	each	..... 21134-00
Pipet, serological, 25 mL.....	1	.....	each	..... 2066-40
Pipet Filler, safety bulb.....	1	.....	each	..... 14651-00
Sample Cell, 10-20-25 mL, w/cap.....	2	.....	6/pkg	..... 24019-06
UV Lamp with power supply, 115 V, with goggles.....	1	.....	each	..... 20828-00
OR				
UV Lamp with power supply, 230 V.....	1	.....	each	..... 20828-02

### OPTIONAL REAGENTS

Hydrochloric Acid, 6.0 N (1:1).....	500 mL	.....	884-49
Sulfuric Acid, ACS.....	500 mL	.....	979-49

### OPTIONAL APPARATUS

pH Paper, 1 to 11 pH units.....	5 rolls/pkg	.....	391-33	
Pipet, serological, 2 mL.....	.....	.....	each	..... 532-36
Pipet, TenSette, 1-10 mL.....	.....	.....	each	..... 19700-10
Pipet Tips, for 19700-10 Tensette Pipet.....	50/pkg	.....	21997-96	
Thermometer, -20 to 110 °C, Non-Mercury.....	.....	.....	each	..... 1877-01

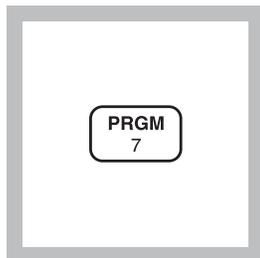
### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**PHOSPHORUS, REACTIVE (0 to 2.50 mg/L PO<sub>4</sub><sup>3-</sup>)** For water, wastewater, seawater**(Also called Orthophosphate) PhosVer 3 (Ascorbic Acid) Method\***

(Powder Pillows or AccuVac Ampuls) USEPA Accepted for wastewater analysis reporting\*\*

**Using Powder Pillows**

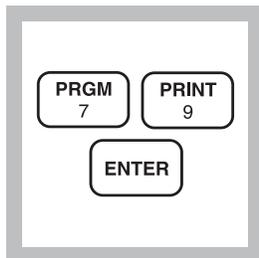
**1.** Enter the stored program number for reactive phosphorus, ascorbic acid method.

Press: **PRGM**

The display will show:

**PRGM ?**

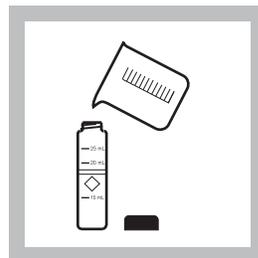
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **79 ENTER**

The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.

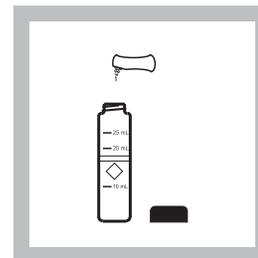
*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*



**3.** Fill a sample cell with 10 mL of sample.

*Note: For samples with extreme pH, see Interferences following these steps.*

*Note: Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*



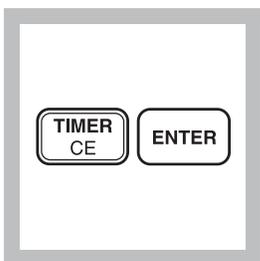
**4.** Add the contents of one PhosVer 3 Phosphate Powder Pillow for 10-mL sample to the cell (the prepared sample). Shake for 15 seconds.

*Note: A blue color will form if phosphate is present.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

\*\* Procedure is equivalent to USEPA method 365.2 and Standard Method 4500-PE for wastewater.

## PHOSPHORUS, REACTIVE, continued

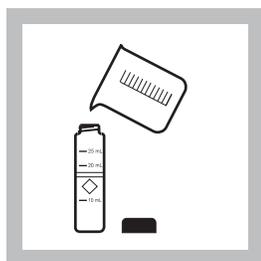


5. Press:

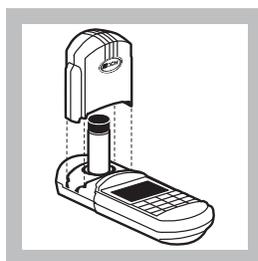
**TIMER ENTER**

A two-minute reaction period will begin. Perform Steps 6-8 during this period.

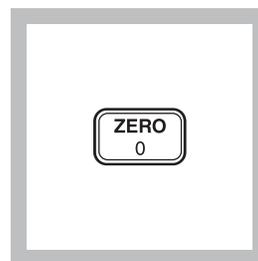
*Note: If the acid-persulfate digestion was used, an 8-10 minute reaction period is required.*



6. Fill another sample cell with 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

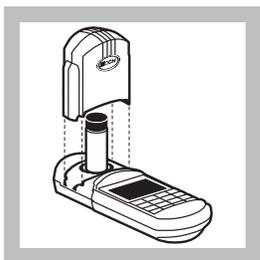


8. Press: **ZERO**

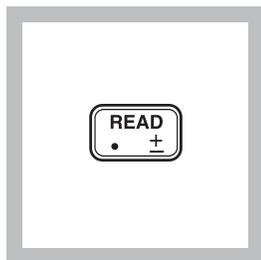
The cursor will move to the right, then the display will show:

**0.00 mg/L PO4**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



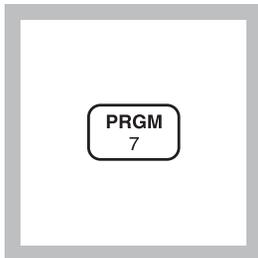
10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate ( $\text{PO}_4^{3-}$ ) will be displayed.

*Note: Standard Adjust may be performed using a 2.0-mg/L  $\text{PO}_4^{3-}$ -standard; see Section 1.*

## PHOSPHORUS, REACTIVE, continued

### Using AccuVac Ampuls



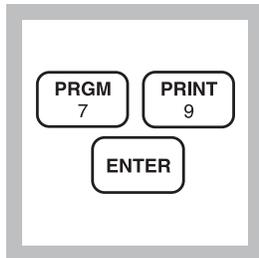
**1.** Enter the stored program number for reactive phosphorus-ascorbic acid method.

Press: **PRGM**

The display will show:

**PRGM ?**

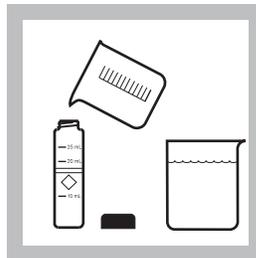
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **79 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

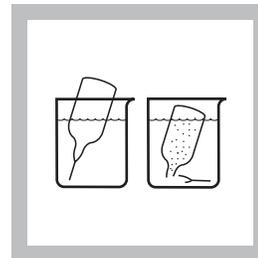
*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*



**3.** Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

*Note: For samples with extreme pH, see Interferences.*

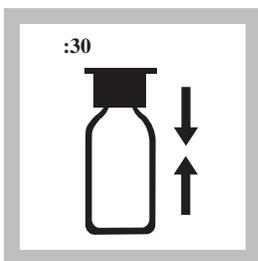
*Note: Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergent containing phosphates to clean glassware.*



**4.** Fill a PhosVer 3 Phosphate AccuVac Ampul with sample.

*Note: Keep the tip immersed while the ampul fills completely.*

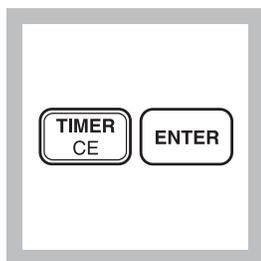
## PHOSPHORUS, REACTIVE, continued



**5.** Place an ampul cap securely over the tip of the ampul. Shake the ampul for about 30 seconds. Wipe off any liquid or fingerprints.

*Note:* A blue color will form if phosphate is present.

*Note:* Accuracy is not affected by undissolved powder.

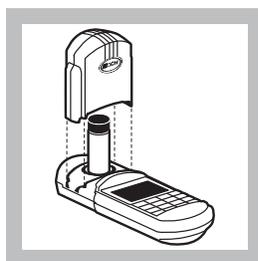


**6.** Press:

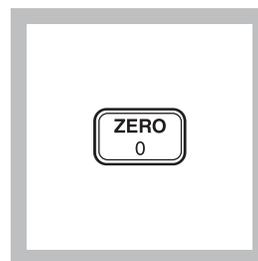
**TIMER ENTER**

A two-minute reaction period will begin. Perform Steps 7-8 during this period.

*Note:* Use an 8-10 minute reaction period if determining total phosphorus following the acid-persulfate digestion.



**7.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

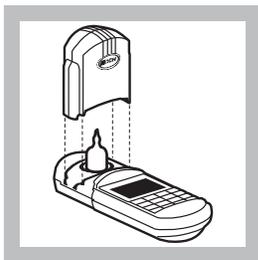


**8.** Press: **ZERO**

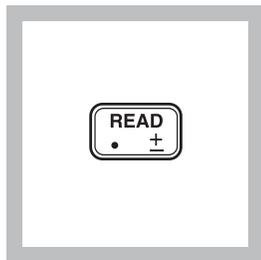
The cursor will move to the right, then the display will show:

**0.00 mg/L PO<sub>4</sub>**

*Note:* If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



**9.** After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO<sub>4</sub><sup>3-</sup>) will be displayed.

*Note:* Standard Adjust may be performed using a 2.0-mg/L PO<sub>4</sub><sup>3-</sup> standard; see Section 1.

## PHOSPHORUS, REACTIVE, continue

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### Sampling and Storage

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing samples at 4 °C. Warm to room temperature before testing.

### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 50 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.
- d) For analysis with AccuVacs, transfer solutions to dry, clean 50 mL beakers to fill the AccuVac ampules. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.
- e) Analyze each standard addition sample as described in the procedure. The phosphate concentration should increase 0.2 mg/L  $\text{PO}_4^{3-}$  for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions in Section 1*.

#### Standard Solution Method

Prepare a 2.0 mg/L  $\text{PO}_4^{3-}$  standard solution by pipetting 4.0 mL of Phosphate Standard Solution, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid-washed Class A 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix. Use this solution in place of the sample in the procedure to insure the accuracy of the test. The mg/L  $\text{PO}_4^{3-}$  reading should be 2.00 mg/L.

## PHOSPHORUS, REACTIVE, continued

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### Method Performance

#### Precision

In a single laboratory using a standard solution of 1.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.05$  mg/L  $\text{PO}_4^{3-}$ .

In a single laboratory using a standard solution of 1.00 mg/L  $\text{PO}_4^{3-}$  and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.03$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L  $\text{PO}_4$ . For more information on the estimated detection limit, see *Section 1*.

#### Interference

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Hydrogen sulfide	All levels
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Turbidity or color	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add the contents of one Phosphate Pretreatment Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. pH 2 to 10 is recommended.

#### Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

## PHOSPHORUS, REACTIVE, continued

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Cat. No.
	Per Test	Unit	
PhosVer 3 Phosphate Reagent Powder Pillows			
10 mL sample size .....	1 Pillow .....	100/pkg.....	21060-69
Sample Cell, 10-20-25 mL, w/cap .....	2.....	6/pkg.....	24019-06

### REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent AccuVac Ampuls ....	1 ampul .....	25/pkg.....	25080-25
Beaker, 50 mL .....	1.....	each.....	500-41
Cap, ampul, blue .....	1.....	25/pkg.....	1731-25
Sample Cell, 10-20-25 mL, w/cap .....	1.....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....	500mL .....	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....	500 mL.....	884-49
Phosphate Standard Solution, 1mg/L .....	500mL.....	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....	20/pkg.....	171-20
Phosphate Standard Solution, Voluette Ampul, 50 mg/L, 10 mL .....	16/pkg.....	171-10
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL * MDB.....	2450-32
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49
Water, deionized .....	4 L.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each.....	24052-00
Ampule Breaker Kit for 10-ml ampules.....	each.....	21968-00
Aspirator, vacuum .....	each.....	2131-00
Cylinder, graduated, mixing, 25 mL, tall (3 required) .....	each.....	20886-40
Filter Holder, 47 mm, 300 mL, graduated.....	each.....	13529-00
Filter, membrane, 47 mm, 0.45 microns .....	100/pkg.....	13530-00
Flask, filtering, 500 mL.....	each.....	546-49
Flask, volumetric, Class A, 100 mL.....	each.....	14574-42
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>Sension</i> <sup>TM</sup> 1, portable with electrode .....	each.....	51700-10
Pipet, 2 mL serological .....	each.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL TenSette Pipet.....	each.....	19700-01
Pipet Tips, for 19700-01 .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 .....	1000/pkg.....	21856-28
Pipet Filler, safety bulb .....	each.....	14651-00
Pipet, volumetric, Class A, 4.00 mL .....	each.....	14515-04
PourRite Ampule Breaker Kit.....	each.....	24846-00

Outside the U.S.A.—Contact the Hach office or distributor serving you.

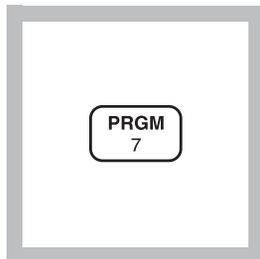
\* Larger sizes available.



**PHOSPHORUS, REACTIVE (0.00 to 5.00 mg/L PO<sub>4</sub><sup>3-</sup>)**

**PhosVer 3 Method, Test 'N Tube Procedure**  
 USEPA accepted for reporting wastewater analysis\*

For water, wastewater, and seawater



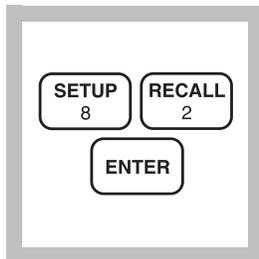
**1.** Enter the stored program number for reactive phosphorus (PO<sub>4</sub><sup>3-</sup>), Test 'N Tube.

Press: **PRGM**

The display will show:

**PRGM ?**

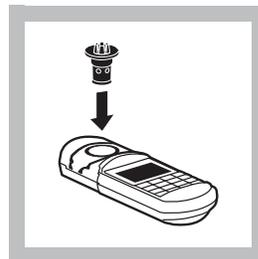
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



**2.** Press: **82 ENTER**

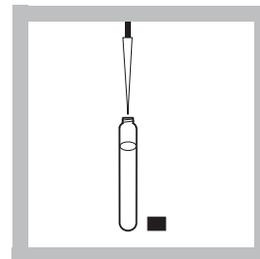
The display will show **mg/L, PO4** and the **ZERO** icon.

*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*



**3.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



**4.** Use a TenSette Pipet to add 5.0 mL of sample to a Reactive Phosphorus Test 'N Tube Dilution Vial. Cap and mix.

*Note: For samples with extreme pH, see the Interference section.*

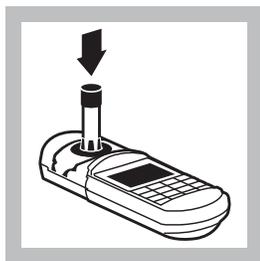
\* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P E for wastewater.

## PHOSPHORUS, REACTIVE, continued



5. Clean the outside of the vial with a towel.

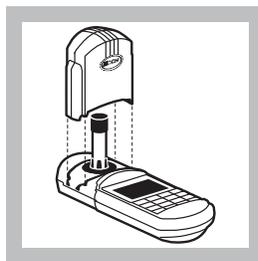
*Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



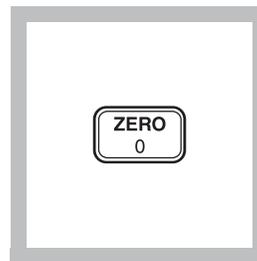
6. Place the sample vial into the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



7. Tightly cover the sample vial with the instrument cap.

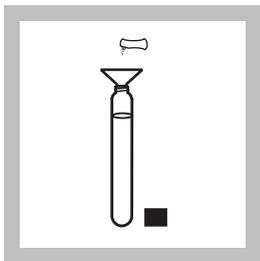


8. Press: **ZERO**

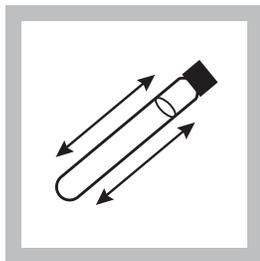
The cursor will move to the right, then the display will show:

**0.00 mg/L PO<sub>4</sub>**

*Note: For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 Reagent.*

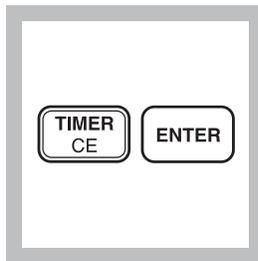


9. Using a funnel, add the contents of one PhosVer 3 Phosphate Powder Pillow to the vial.



10. Cap the vial tightly and shake for 10-15 seconds.

*Note: The powder will not completely dissolve.*

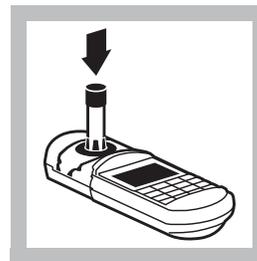


11. Press:  
**TIMER ENTER**

A 2-minute reaction time will begin.

*Note: Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

*Note: A blue color will develop if phosphate is present.*



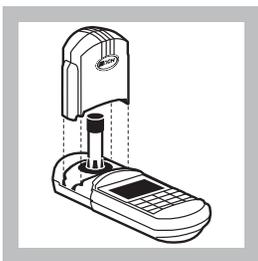
12. Immediately after the timer beeps, place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

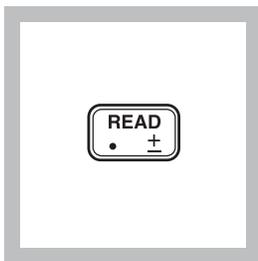
*Note: Do not move the vial from side to side as this can cause errors.*

## PHOSPHORUS, REACTIVE, continued

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**13.** Tightly cover the vial with the instrument cap.



**14.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate ( $\text{PO}_4^{3-}$ ) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing at 4 °C. Warm to room temperature before analyzing the sample.

### Accuracy Check

*Note: Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

## PHOSPHORUS, REACTIVE, continued

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- d) Analyze each sample as described in the procedure; use 5.0 mL of the prepared standard additions for each test. The concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, prepare a 1.0-mg/L  $\text{PO}_4^{3-}$  standard by pipetting 2 mL of solution from a Phosphate Voluette Ampule Standard for Phosphate, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid-washed, Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 5.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.08$  mg/L  $\text{PO}_4^{3-}$ .

### Estimated Detection Limit (EDL)

The EDL for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## PHOSPHORUS, REACTIVE, continued

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### Interferences

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	6 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: <ol style="list-style-type: none"><li>1. Measure 25 mL of sample into a 50-mL beaker.</li><li>2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops.</li><li>3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with <i>step 1</i>.</li></ol>
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

### Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

## PHOSPHORUS, REACTIVE, continued

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### Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

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### REQUIRED REAGENTS

Reactive Phosphorus Test 'N Tube Reagent Set.....50 tests ..... 27425-45  
Includes: (1) 21060-46, (50) Orthophosphate Dilution Vials\*

Description	Quantity Required		
	Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows .....	1	50/pkg	21060-46
50 Orthophosphate Test 'N Tube Dilution Vials .....	1	50/pkg	..... *

### REQUIRED APPARATUS

COD/TNT Adapter .....	1	each	48464-00
Funnel, micro .....	1	each	25843-35
Pipet, TenSette, 1 to 10 mL.....	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet .....	1	50/pkg	21997-96
Test Tube Rack .....	1-3	each	18641-00

### OPTIONAL REAGENTS

Bromine Water, 30 g/L.....	29 mL	.....	2211-20
Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....	500mL	.....	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1).....	500 mL	.....	884-49
Phenol Solution, 30 g/L .....	29 mL	.....	2112-20
Phosphate Standard Solution, 1 mg/L as PO <sub>4</sub> <sup>3-</sup> .....	500 mL	.....	2569-49
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL .....	16/pkg	.....	171-10
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....	20/pkg	.....	171-20H
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL	.....	28332-49
Water, deionized.....	4 L	.....	272-56

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\* These items are not sold separately.

## PHOSPHORUS, REACTIVE, continued

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### OPTIONAL APPARATUS

Ampule Breaker, Pour Rite (2-mL ampule).....	each.....	24846-00
Ampule Breaker Kit .....	each.....	21968-00
Aspirator, vacuum .....	each.....	2131-00
Cylinder, graduated, mixing, 25 mL (3 required) .....	each.....	20886-40
Dispenser, Repipet Jr., 2 mL .....	each.....	22307-01
Filter Holder, 47 mm, 300 mL, graduated.....	each.....	13529-00
Filter, membrane, 47 mm, 0.45 microns .....	100/pkg.....	13530-00
Flask, filtering, 500 mL.....	each.....	546-49
Flask, volumetric, Class A, 100 mL.....	each.....	14574-42
pH Indicator Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
pH Meter, <i>sension</i> <sup>TM</sup> I, portable with electrode.....	each.....	51700-10
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 .....	50 pkg.....	21856-96
Pipet Tips, for 19700-01 .....	1000/pkg.....	21856-28
Pipet Filler, Safety Bulb .....	each.....	14651-00
Pipet, volumetric, Class A, 5.00 mL .....	each.....	14515-37
Pipet, volumetric, Class A, 2.00 mL .....	each.....	14515-36

### *For Technical Assistance, Price and Ordering*

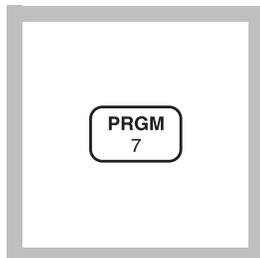
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**PHOSPHORUS, REACTIVE (0 to 30.0 mg/L PO<sub>4</sub><sup>3-</sup>)****Amino Acid Method\***

For water, wastewater, seawater



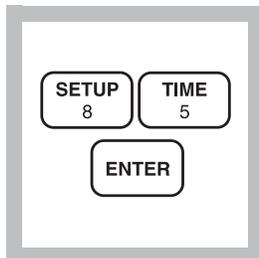
1. Enter the stored program number for reactive phosphate (PO<sub>4</sub><sup>3-</sup>), amino acid method.

Press: **PRGM**

The display will show:

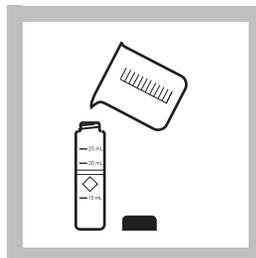
**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

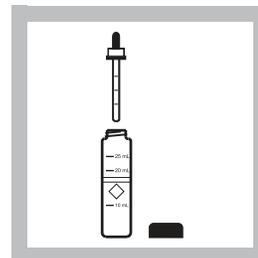


2. Press: **85 ENTER**  
The display will show **mg/L, PO4** and the **ZERO** icon.

*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press **CONC**.*



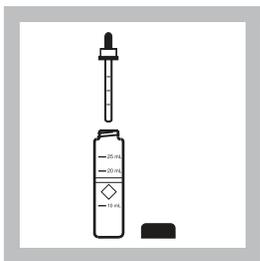
3. Fill a 25-mL sample cell with 25 mL of sample.



4. Add 1 mL of Molybdate Reagent using a 1-mL calibrated dropper.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

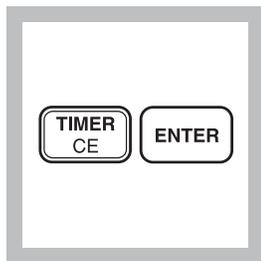
## PHOSPHORUS, REACTIVE, continued



**5.** Add 1 mL of Amino Acid Reagent Solution. Cap and invert several times to mix (the prepared sample).

*Note:* A blue color will form if phosphate is present.

*Note:* You may substitute the contents of one Amino Acid Reagent Powder Pillow for 1 mL of Amino Acid Reagent Solution.



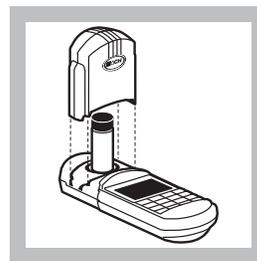
**6.** Press: **TIMER ENTER**

A 10-minute reaction period will begin.

*Note:* Perform Step 7 while the timer is running.



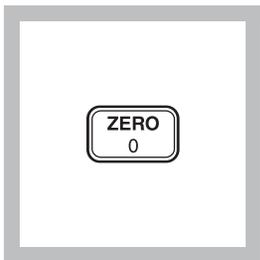
**7.** Pour 25 mL of sample (the blank) into a sample cell.



**8.** When the timer beeps, the display will show:

**mg/L PO<sub>4</sub>**

Place the blank into the cell holder. Cover the sample cell with the instrument cap.

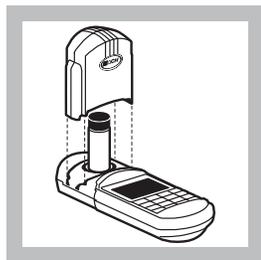


**9.** Press: **ZERO**

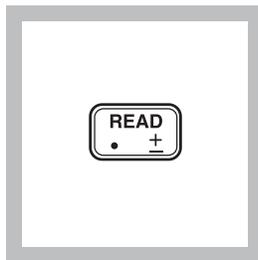
The cursor will move to the right, then the display will show:

**0.0 mg/L PO<sub>4</sub>**

*Note:* If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



**10.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**11.** Press: **READ**

The cursor will move to the right, then the result in mg/L PO<sub>4</sub> will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

### Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

## PHOSPHORUS, REACTIVE, continued

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Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) for up to 48 hours.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$ .
- b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples. Mix well.
- c) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L orthophosphate ( $\text{PO}_4^{3-}$ ).
- d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

#### Standard Solution Method

Prepare a 10.0-mg/L phosphate standard by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L as  $\text{PO}_4^{3-}$  into a 50-mL volumetric flask. Dilute to volume with deionized water.

Or, prepare a 10.0-mg/L  $\text{PO}_4^{3-}$  standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate PourRite Ampule Standard, 500 mg/L as  $\text{PO}_4^{3-}$ , into a 50-mL volumetric flask. Dilute to volume with deionized water.

Substitute this standard for the sample and perform the test as described. The mg/L  $\text{PO}_4^{3-}$  reading should be 10 mg/L.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 15.0 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.12$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit

The estimated detection limit for program 85 is 0.14 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1*.

## PHOSPHORUS, REACTIVE, continued

### Interferences

Interfering Substance	Interference Levels and Treatments
Calcium (Ca <sup>2+</sup> )	Greater than 10,000 mg/L as CaCO <sub>3</sub>
Chloride	Greater than 150,000 mg/L as Cl <sup>-</sup>
Colored samples	Add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
High salt levels (Na <sup>+</sup> )	May cause low results. To eliminate this interference, dilute the sample until two successive dilutions yield about the same result.
Magnesium	Greater than 40,000 mg/L as CaCO <sub>3</sub>
Nitrites (NO <sub>2</sub> <sup>-</sup> )	Bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid to the sample. Swirl to mix. Continue with Step 4.
Phosphates, high levels (PO <sub>4</sub> <sup>3-</sup> )	As the concentration of phosphate increases, the color changes from blue to green, then to yellow and finally to brown. The brown color may suggest a concentration as high as 100,000 mg/L PO <sub>4</sub> <sup>3-</sup> . If a color other than blue is formed, dilute the sample and retest.
Sulfide (S <sup>2-</sup> )	Sulfide interferes. For samples with sulfide concentration less than 5 mg/L, sulfide interference may be removed by oxidation with Bromine Water as follows: <ol style="list-style-type: none"> <li>1. Measure 50mL of sample into a 125-mL flask.</li> <li>2. Add Bromine Water dropwise with constant swirling until permanent yellow color develops.</li> <li>3. Add Phenol Solution dropwise until the yellow color just disappears. Use this sample in Steps 3 and 7.</li> </ol>
Temperature	For best results, sample temperature should be 21 ±3 °C (70 ±5 °F).
Turbidity	May give inconsistent results for two reasons. Some suspended particles may dissolve because of the acid used in the test. Also, desorption of orthophosphate from particles may occur. For highly turbid samples, add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

### Summary of Method

In a highly acidic solution, ammonium molybdate reacts with orthophosphate to form molybdophosphoric acid. This complex is then reduced by the amino acid reagent to yield an intensely colored molybdenum blue compound.

### REQUIRED REAGENTS

High Range Reactive Phosphorus Reagent Set (100 Test) ..... **Cat. No.** 22441-00

## PHOSPHORUS, REACTIVE, continued

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Includes: (1) 1934-32, (1) 2236-32

Description	Quantity Required		Units	Cat. No.
	Per Test			
Amino Acid Reagent .....	1 mL	.....100 mL	MDB*	.....1934-32
Molybdate Reagent .....	1 mL	.....100 mL	MDB*	.....2236-32

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap .....	2	.....6/pkg	.....24019-06
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### OPTIONAL REAGENTS

Description		Units	Cat. No.
Amino Acid Reagent Powder Pillow .....		100/pkg	.....804-99
Bromine Water, 30 g/L .....		29 mL	.....2211-20
Hydrochloric Acid Solution, 1:1 (6 N) .....		500 mL	.....884-49
Phenol Solution, 30 g/L .....		29 mL	.....2112-20
Phosphate Standard Solution, 50 mg/L PO <sub>4</sub> <sup>3-</sup> .....		500 mL	.....171-49
Phosphate Standard Solution, PourRite ampule, 500 mg/L PO <sub>4</sub> <sup>3-</sup> , 2 mL .....		20/pkg	.....14242-20
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL	MDB	..... 2450-32
Sulfamic Acid .....		113 g	.....2344-14
Sulfuric Acid Standard Solution, 10 N .....		1 L	.....931-53
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....		500 mL	.....28331-49
Water, deionized .....		4L	.....272-56

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\* Larger sizes available.

## PHOSPHORUS, REACTIVE, continued

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### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit, PourRite.....	each.....	24846-00
Aspirator, vacuum.....	each.....	2131-00
Cylinder, graduated, 50 mL.....	each.....	508-41
Cylinder, graduated, mixing, 25 mL.....	each.....	20886-40
Filter Holder, 47 mm, 300 mL, graduated.....	each.....	13529-00
Filter, membrane, 47 mm, 0.45 microns.....	100/pkg.....	13530-00
Flask, filtering, 500 mL.....	each.....	546-49
Flask, erlenmeyer, 125 mL.....	each.....	505-43
Flask, volumetric, Class A, 50 mL.....	each.....	14574-41
pH Indicator Paper, 1 to 11 pH.....	5 rolls/pkg.....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> <b>I</b> , portable with electrode.....	each.....	51700-10
Pipet, serological, 2.0 mL.....	each.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet.....	50/pkg.....	21856-96
Pipet Tips, for 19700-01.....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 10.00 mL.....	each.....	14515-38
Pipet Filler, safety bulb.....	each.....	12189-00
Spoon, measuring, 0.05 g.....	each.....	492-00
Thermometer, -20 to 110 °C, Non-Mercury.....	each.....	26357-02

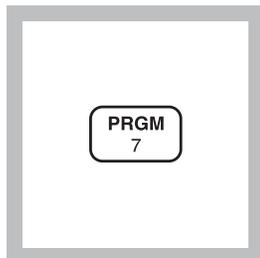
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**PHOSPHORUS, REACTIVE (0 to 45.0 mg/L PO<sub>4</sub><sup>3-</sup>) For water and wastewater**

(Also called Orthophosphate) Molybdovanadate Method\*  
(Reagent Solution or AccuVac Ampuls)

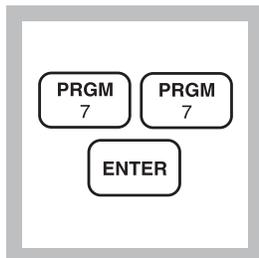
**Using Reagent Solution**

**1.** Enter the stored program number for high range phosphate (PO<sub>4</sub><sup>3-</sup>) reagent solution.

Press: **PRGM**

The display will show:

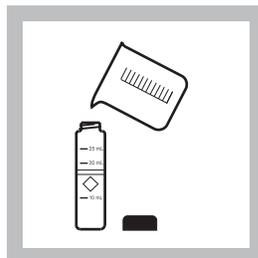
**PRGM ?**



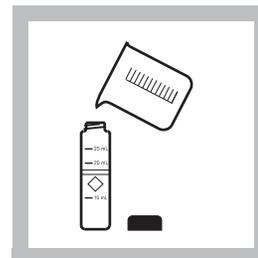
**2.** Press: **77 ENTER**

The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.

*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*

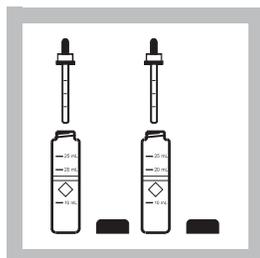


**3.** Fill a sample cell with 25 mL of deionized water (the blank).



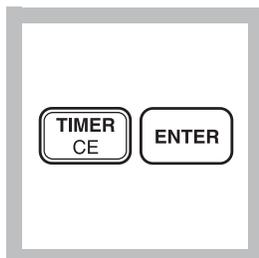
**4.** Fill another sample cell with 25 mL of sample (the prepared sample).

*Note: For best results, the sample temperature should be 20-25 °C.*



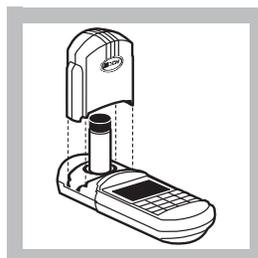
**5.** Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Cap the cells and invert to mix.

*Note: A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank, because of the reagent.*

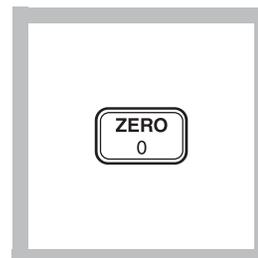


**6.** Press: **TIMER ENTER**

A five-minute reaction period will begin.



**7.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



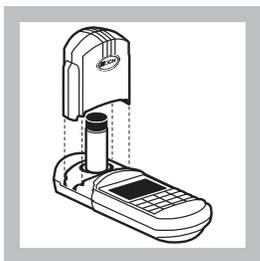
**8.** Press: **ZERO**

The cursor will move to the right, then the display will show:

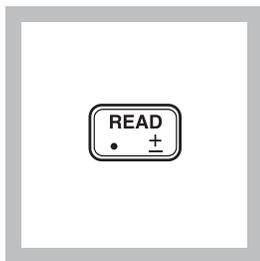
**0.0 mg/L PO<sub>4</sub>**

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## PHOSPHORUS, REACTIVE, continued



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

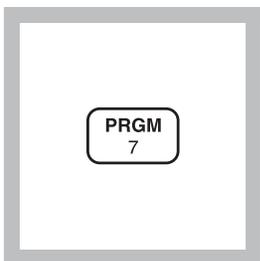


**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

### Using AccuVac Ampuls

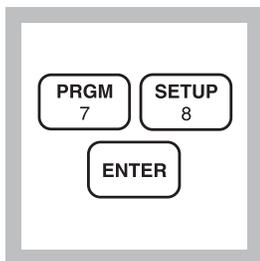


**1.** Enter the stored program number for high range phosphate ( $\text{PO}_4^{3-}$ )-AccuVac Ampuls.

Press: **PRGM**

The display will show:

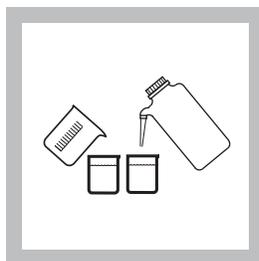
**PRGM ?**



**2.** Press: **78 ENTER**

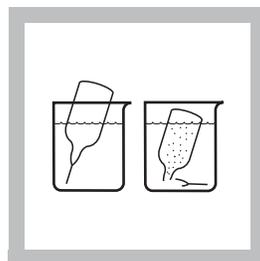
The display will show **mg/L, PO4** and the **ZERO** icon.

*Note: For alternate forms (P,  $\text{P}_2\text{O}_5$ ), press the **CONC** key.*



**3.** Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

*Note: For best results, sample temperature should be 20-25 °C.*

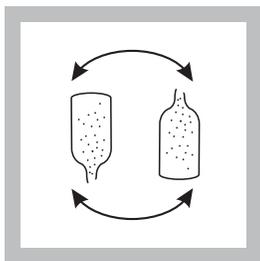


**4.** Fill a Molybdo-vanadate Reagent AccuVac Ampul with sample. Fill a second AccuVac Ampul with deionized water (the blank).

*Note: Keep the tip immersed while the ampul fills completely.*

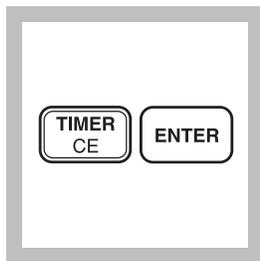
## PHOSPHORUS, REACTIVE, continued

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**5.** Invert the ampul several times to mix, then wipe off any liquid or fingerprints.

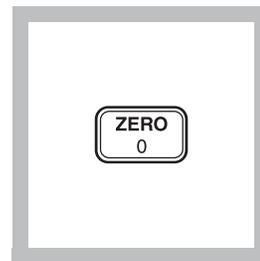
*Note: A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank because of the reagent.*



**6.** Press: **TIMER ENTER**  
A five-minute reaction period will begin.

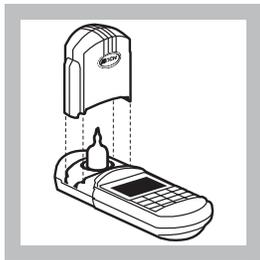


**7.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

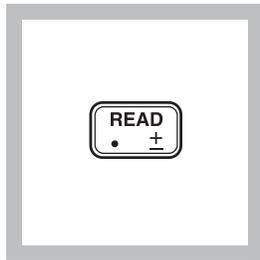


**8.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.0 mg/L PO<sub>4</sub>**



**9.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**10.** Press: **READ**  
The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

## PHOSPHORUS, REACTIVE, continued

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Analyze samples immediately for best results. If prompt analysis is impossible, preserve samples by filtering immediately and storing at 4 °C for up to 48 hours.

### Accuracy Check

#### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and invert to mix well.
- d) For analysis with AccuVac Ampuls, transfer the spiked samples to clean, dry 50-mL beakers to facilitate filling of the ampuls. For analysis with reagent solution, transfer the spiked samples to 25-mL sample cells.
- e) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L  $\text{PO}_4^{3-}$ .
- f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

#### Standard Solution Method

Obtain a Hach Phosphate Standard Solution, 10.0 mg/L as phosphate. Using this solution as the sample, perform the phosphate procedure as described above.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* for more information.

## PHOSPHORUS, REACTIVE, continued

### Method Performance

#### Precision

In a single laboratory using a standard solution of 30.0 mg/L  $\text{PO}_4^{3-}$ , two lots of reagent, and the instrument, a single operator obtained a standard deviation of  $\pm 0.1$  mg/L  $\text{PO}_4^{3-}$  for the reagent solution method and a standard deviation of  $\pm 0.2$  for the AccuVac Ampul method.

#### Estimated Detection Limit

The estimated detection limit for program 77 is 0.3 mg/L  $\text{PO}_4^{3-}$  and 0.4 mg/L  $\text{PO}_4^{3-}$  for program 78. For more information on the estimated detection limit, see *Section 1*.

### Interferences

#### Interfering Substances and Suggested Treatment

Interfering Substance	Interference Level and Treatment
Arsenate	Only interferes if sample is heated.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if sample is heated.
Sulfide	Causes a negative interference. Remove interference as follows: <ol style="list-style-type: none"> <li>1. Measure 50 mL of sample into an erlenmeyer flask.</li> <li>2. Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops.</li> <li>3. Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with step 4 of the procedure (step 3 if using the AccuVac procedure).</li> </ol>
Extreme pH or highly buffered samples	May exceed buffering capacity of reagents. See Section 1, <i>pH Interferences</i> . Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause negative interference
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, $\text{Al}^{3+}$ , $\text{Fe}^{3+}$ , $\text{Mg}^{2+}$ , $\text{Ca}^{2+}$ , $\text{Ba}^{2+}$ , $\text{Sr}^{2+}$ , $\text{Li}^+$ , $\text{Na}^+$ , $\text{K}^+$ , $\text{NH}_4^+$ , $\text{Cd}^{2+}$ , $\text{Mn}^{2+}$ , $\text{NO}_3^-$ , $\text{NO}_2^-$ , $\text{SO}_4^{2-}$ , $\text{SO}_3^{2-}$ , $\text{Pb}^{2+}$ , $\text{Hg}^+$ , $\text{Hg}^{2+}$ , $\text{Sn}^{2+}$ , $\text{Cu}^{2+}$ , $\text{Ni}^{2+}$ , $\text{Ag}^+$ , $\text{U}^{4+}$ , $\text{Zr}^{4+}$ , $\text{AsO}_3^-$ , $\text{Br}^-$ , $\text{CO}_3^{2-}$ , $\text{ClO}_4^-$ , $\text{CN}^-$ , $\text{IO}_3^-$ , $\text{SiO}_4^{4-}$ .	

### Summary of Method

## PHOSPHORUS, REACTIVE, continued

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

### REQUIRED REAGENTS AND APPARATUS (using Reagent Solution)

Description	Quantity Required		Units	Cat. No.
	Per Test			
Molybdovanadate Reagent .....	2.0 mL	100 mL*	MDB	20760-32
Sample Cell, 10-20-25 mL, w/ cap .....	2		6/pkg	24019-06
Water, deionized.....	25 mL		4 L	272-56

### REQUIRED REAGENTS AND APPARATUS (using AccuVac Ampuls)

Molybdovanadate Reagent AccuVac Ampuls .....	2		25/pkg	25250-25
Beaker, 50 mL.....	2		each	500-41H
Water, deionized.....	25 mL		4 L	272-56

### OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L.....	29 mL*	2211-20
Hydrochloric Acid Solution, 1:1 (6.0 N).....	500 mL	884-49
Phenol Solution, 30 g/L .....	29 mL	2112-20
Phosphate Standard Solution, 10.0 mg/L as PO <sub>4</sub> <sup>3-</sup> .....	946 mL	14204-16
Phosphate Standard Solution, Voluette Ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL .....	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL* MDB	2450-32
Sulfuric Acid, ACS .....	500 mL*	979-49
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL	28331-49

### OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each	24052-00
Ampule Breaker Kit.....	each	21968-00
Cylinder, graduated, 25 mL .....	each	508-40
Cylinder, graduated, mixing, 25-mL.....	each	20886-40
Dispenser, fixed volume, 1.0 mL Repipet Jr.....	each	21113-02
Flask, erlenmeyer, 50 mL .....	each	505-41
Flask, volumetric, Class A, 50 mL .....	each	14574-41
pH Paper, 1 to 11 pH units.....	5 rolls/pkg	391-33
pH Meter, <i>Sensio</i> <sup>TM</sup> <b>I</b> , portable with electrode .....	each	51700-10

\* Contact Hach for larger sizes.

## PHOSPHORUS, REACTIVE, continued

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### OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Pipet, serological, 2.0 mL.....	each .....	532-36
Pipet, TenSette, 0.1 to 1.0 mL.....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Thermometer, -20 to 110 °C.....	each .....	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

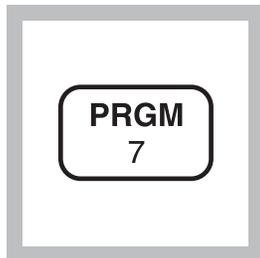
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**PHOSPHORUS, REACTIVE, HR (0.0 to 100.0 mg/L PO<sub>4</sub><sup>3-</sup>)**

Molybdovanadate Method\*, Test 'N Tube™ Procedure

For water and wastewater

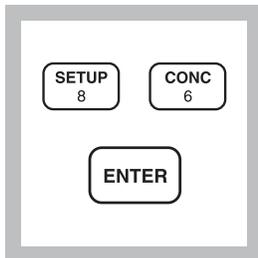


1. Enter the stored program number for phosphorus, reactive, high range, Test 'N Tube.

Press: **PRGM**

The display will show:

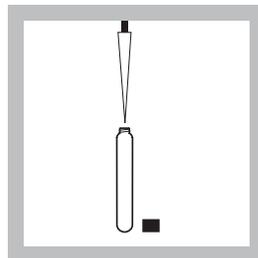
**PRGM ?**



2. Press: **86 ENTER**

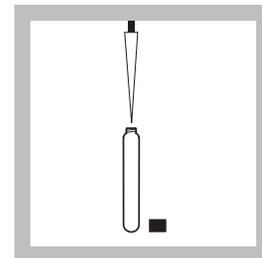
The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.

*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*



3. Use a TenSette® Pipet to add 5.0 mL of deionized water to a Reactive High Range Phosphorus Test 'N Tube Vial (the blank).

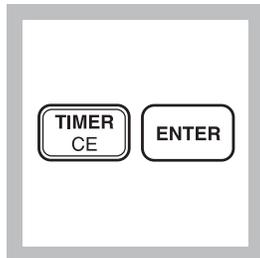
Cap and invert to mix.



4. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive High Range Phosphorus Test 'N Tube Vial (the sample).

Cap and invert to mix.

*Note: For samples with extreme pH, see the Interference section.*



5. Press:

**TIMER ENTER**

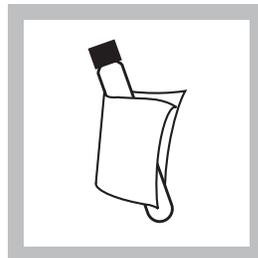
A 7-minute reaction period will begin.

*Note: This reaction time is for samples at 23 °C (73 °F). If the sample temperature is 13 °C (55 °F), wait 15 minutes. If the sample temperature is 33 °C (91 °F), wait two minutes.*



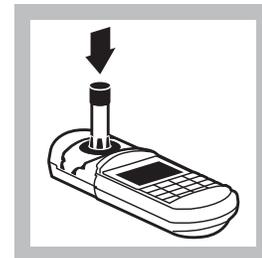
6. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note: A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



7. Clean the outside of the vials with a towel.

*Note: Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

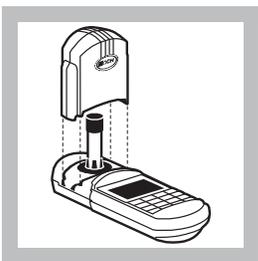


8. When the timer sounds, place the blank vial into the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

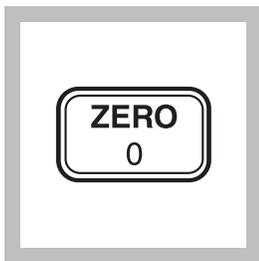
*Note: Do not move the vial from side to side as this can cause errors.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## PHOSPHORUS, REACTIVE, HR, continued



**9.** Tightly cover the sample cell with the instrument cap.

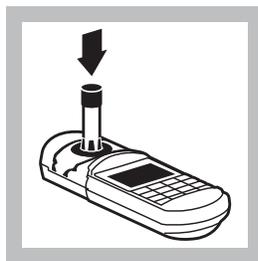


**10.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.0 mg/L PO<sub>4</sub>**

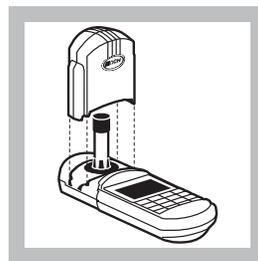
*Note: Reagent blanks for each lot of reagent may be used more than once. At room temperature, the reagent blank is stable for as long as three weeks; then prepare a new one.*



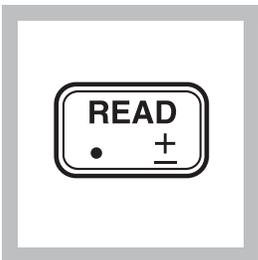
**11.** Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



**12.** Tightly cover the vial with the instrument cap.



**13.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO<sub>4</sub><sup>3-</sup>) will be displayed.

*Note: For best results, use Standard Adjust with each new lot of reagent. (See Accuracy Check.)*

## PHOSPHORUS, REACTIVE, HR, continued

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning the glassware used in this test.

For best results, analyze the samples immediately after collection. If prompt analysis is impossible, preserve the samples for up to 748 hours by filtering immediately and storing at 4 °C. The sample should have a neutral (6–8) pH and be at room temperature before analysis.

### Accuracy Check

*Note: Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

#### Standard Additions Method

- a. Fill three 10-mL graduated mixing cylinders with 10 mL of sample.
- b. Snap the neck off a Voluette™ Ampule of Phosphate Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$  (Cat. No. 14242-10).
- c. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of sample prepared in *step a*. Mix well.
- d. Analyze each sample from *step c* as described in the procedure; use 5.0 mL of the prepared sample for each test. The concentration should increase as follows: 5 mg/L, 10 mg/L, and 15 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e. If these increases do not occur, see *Standard Additions* in *Section 1* of the *DR/890 Procedures Manual* for more information.

#### Standard Solution Method

To check accuracy, prepare an 80 mg/L  $\text{PO}_4^{3-}$  standard by pipetting 8.0 mL of solution from a 10-mL Voluette Ampule of Phosphate Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$ , into an acid-cleaned 50-mL Class A volumetric flask. Fill to the line with deionized water. Substitute this standard for the sample and perform the procedure as described.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/mL  $\text{PO}_4^{3-}$  standard solution, press the **SETUP** key and

## PHOSPHORUS, REACTIVE, HR, continued

scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

### Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated. <sup>1</sup>
Iron, ferrous	Blue color caused by ferrous iron does not interfere if the iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Sulfide	Causes a negative interference. Remove interference as follows: <ol style="list-style-type: none"> <li>1. Measure 50 mL of sample into an Erlenmeyer flask.</li> <li>2. Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops.</li> <li>3. Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with <i>step 1</i> of the procedure.</li> </ol>
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See <i>pH Interferences in Section 1</i> of the <i>DR/890 Procedure Manual</i> . Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, cold (less than 20 °C)	Causes a negative interference.
Temperature, hot (greater than 25 °C)	Causes a positive interference.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al <sup>3+</sup> , Fe <sup>3+</sup> , Mg <sup>2+</sup> , Ca <sup>2+</sup> , Ba <sup>2+</sup> , Sr <sup>2+</sup> , Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Cd <sup>2+</sup> , Mn <sup>2+</sup> , NO <sub>3</sub> <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , SO <sub>3</sub> <sup>2-</sup> , Pb <sup>2+</sup> , Hg <sup>+</sup> , Hg <sup>2+</sup> , Sn <sup>2+</sup> , Cu <sup>2+</sup> , Ni <sup>2+</sup> , Ag <sup>+</sup> , U <sup>4+</sup> , Zr <sup>4+</sup> , AsO <sub>3</sub> <sup>-</sup> , Br <sup>-</sup> , CO <sub>3</sub> <sup>2-</sup> , ClO <sub>4</sub> <sup>-</sup> , CN <sup>-</sup> , IO <sub>3</sub> <sup>-</sup> , SiO <sub>4</sub> <sup>4-</sup> .	

<sup>1</sup> Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

## PHOSPHORUS, REACTIVE, HR, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 80.0 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 3.0$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit (EDL)

The EDL for program 86 is 7.0 mg/L  $\text{PO}_4^{3-}$ . For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

### Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

### Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

### Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

### Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.

## PHOSPHORUS, REACTIVE, HR, continued

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6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	86	29	0
2	4	30	80
3	73	31	50
4	0	32	79
5	0	33	53
6	0	34	0
7	0	35	62
8	65	36	166
9	56	37	246
10	217	38	148
11	21	39	63
12	66	40	63
13	157	41	78
14	197	42	252
15	30	43	4
16	0	44	76
17	0	45	128
18	0	46	0
19	0	47	15
20	80	48	1
21	79	49	164
22	52	50	0
23	0	51	0
24	0	52	0
25	80	53	0
26	0	54	80
27	0	55	0
28	0	56	255

## PHOSPHORUS, REACTIVE, HR, continued

### REQUIRED REAGENTS

High Range Reactive Phosphorus Test 'N Tube™ Reagent Set.....50 vials.....27673-45  
Includes: (50) Reactive High Range Phosphorus Test 'N Tube™ Vials\*, (2) 272-42

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Reactive High Range Phosphorus Test 'N Tube™ Vials.....	1.....	50/pkg.....	* .....
Water, deionized.....	.....	100 mL.....	272-42

### REQUIRED APPARATUS

COD/TNT Adapter for DR/800 Series.....	1.....	each.....	48464-00
Pipet, TenSette®, 1 to 10 mL.....	1.....	each.....	19700-10
Pipet Tips, for 19700-10 TenSette® Pipet.....	1.....	50/pkg.....	21997-96
Test Tube Rack.....	1-3.....	each.....	18641-00

### OPTIONAL REAGENTS

Bromine Water, 30 g/L.....	29 mL** .....	2211-20
Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....	500 mL.....	884-49
Phenol Solution, 30 g/L.....	29 mL.....	2112-20
Phosphate Standard Solution, PourRite ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL.....	20/pkg.....	14242-20
Phosphate Standard Solution, Voluette ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL.....	16/pkg.....	14242-10
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL.....	28331-49

### OPTIONAL APPARATUS

Ampule Breaker Kit .....	each.....	21968-00
Aspirator, vacuum .....	each.....	2131-00
Cylinder, graduated, mixing, 10 mL, 3 required .....	each.....	20886-38
Filter Holder, 47 mm, 300 mL, graduated.....	each.....	13529-00
Filter, membrane, 47 mm, 0.45 microns .....	200/pkg.....	13530-00
Flask, filtering, 500 mL.....	each.....	546-49
Flask, volumetric, Class A, 50-mL .....	each.....	14574-41
pH Indicator Paper, 1 to 11 pH units.....	5 rolls/pkg.....	391-33
pH Meter, <i>sensio</i> ™1, portable with electrode .....	each.....	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet.....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 TenSette® Pipet.....	1000/pkg.....	21856-28
Pipet Tips, for 19700-10 TenSette® Pipet.....	250/pkg.....	21997-25
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 8.00-mL.....	each.....	14515-08
PourRite™ Ampule Breaker .....	each.....	24846-00

\* These items are not sold separately.

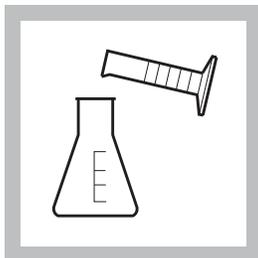
\*\* Larger sizes available.



## PHOSPHORUS, ACID HYDROLYZABLE

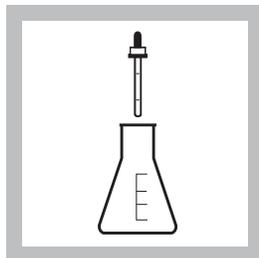
### Hydrolysis to Orthophosphate Method\*

For water, wastewater, seawater



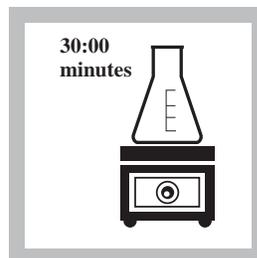
**1.** Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

*Note:* Wash all glassware with 6 N hydrochloric acid. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.



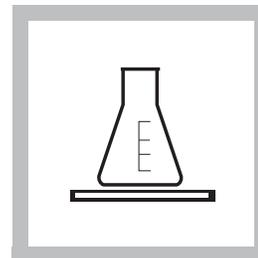
**2.** Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

*Note:* Use the 1-mL calibrated dropper provided.



**3.** Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

*Note:* Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.

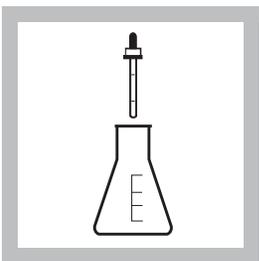


**4.** Cool the hot prepared sample to room temperature.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

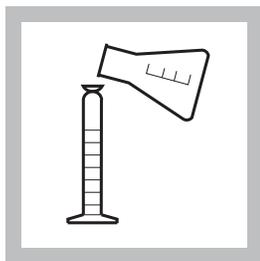
## PHOSPHORUS, ACID HYDROLYZABLE, continued

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**5.** Add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the prepared sample. Swirl to mix.

*Note:* Use the 1-mL calibrated dropper provided.



**6.** Pour the prepared sample into a graduated cylinder. Add deionized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

*Note:* Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result. Make sure both results are in the same chemical form and units.

## **PHOSPHORUS, ACID HYDROLYZABLE, continued**

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### **Sampling and Storage**

Analyze samples immediately after collection for best results. If prompt analysis is not possible, samples may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

### **Interferences**

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

### **Summary of Method**

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to reactive orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the “acid hydrolyzable” phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

## PHOSPHORUS, ACID HYDROLYZABLE, continued

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### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....			500mL.....	28330-49
Sodium Hydroxide Solution, 5.0 N .....	2 mL	.....100 mL	* MDB.....	2450-32
Sulfuric Acid Solution, 5.25 N .....	2 mL	.....100 mL	* MDB .....	2449-32
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....			500 mL.....	28332-49
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....			500 mL.....	28331-49

### REQUIRED APPARATUS

Cylinder, graduated, 25 mL .....	2.....	each.....	508-40
Flask, erlenmeyer, 50 mL .....	1.....	each.....	505-41

### OPTIONAL REAGENTS

Hydrochloric Acid, 6 N .....	500 mL .....	884-49
Water, deionized .....	4L .....	272-56

### OPTIONAL APPARATUS

Cylinder, graduated, 50 mL .....	each.....	508-41
Flask, erlenmeyer, 125 mL .....	each.....	505-43
Hot Plate, 4" diameter, 120 Vac .....	each.....	12067-01
Hot Plate, 4" diameter, 240 Vac .....	each.....	12067-02
Pad, cooling, 4" x 4" .....	each.....	18376-00
pH indicator Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>sensio</i> <sup>TM</sup> <b>I</b> , portable with electrode .....	each.....	51700-10
Thermometer, -20 to 110 °C, Non-Mercury .....	each.....	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

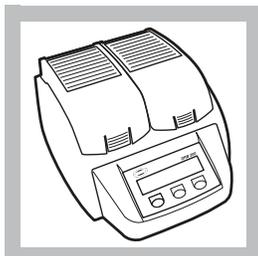
Outside the U.S.A.—Contact the Hach office or distributor serving you.

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\* Contact Hach for larger sizes.

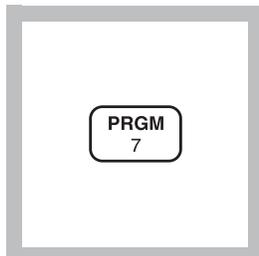
**PHOSPHORUS, ACID HYDROLYZABLE (0.00 to 5.00 mg/L PO<sub>4</sub><sup>3-</sup>)****PhosVer 3 with Acid Hydrolysis  
Test 'N Tube™ Procedure**

For water, wastewater, and seawater



**1.** Turn on the COD Reactor. Heat to 150 °C.

*Note:* See DRB200 instrument manual for selecting preprogrammed temperature applications.



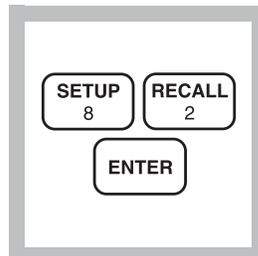
**2.** Enter the stored program number for acid hydrolyzable phosphorus (PO<sub>4</sub><sup>3-</sup>), Test 'N Tube.

Press: **PRGM**

The display will show:

**PRGM ?**

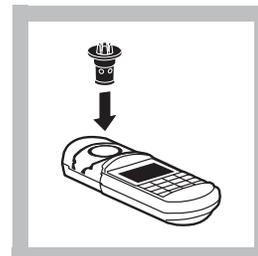
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



**3.** Press: **82 ENTER**

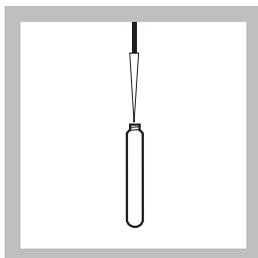
The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.

*Note:* For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.

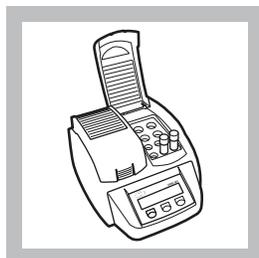


**4.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

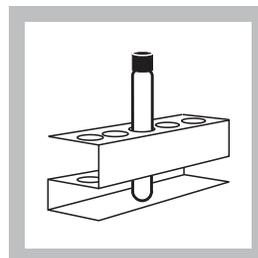
*Note:* A diffuser band covers the light path holes on the adapter to give increased performance. The band should **NOT** be removed.



**5.** Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial. Cap and mix.

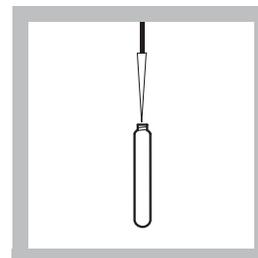


**6.** Heat the vial in the DRB200 Reactor for 30 minutes.



**7.** Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

*Note:* Vials will be hot.



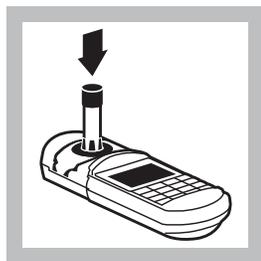
**8.** Remove the cap from the vial. Use a TenSette Pipet to add 2.0 mL of 1.00 N sodium hydroxide to the vial. Cap and mix.

## PHOSPHORUS, ACID HYDROLYZABLE, continued



**9.** Clean the outside of the vial with a towel.

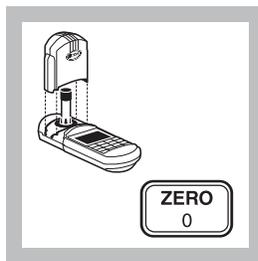
*Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



**10.** Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



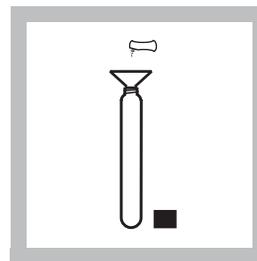
**11.** Tightly cover the vial with the instrument cap.

Press: **ZERO**

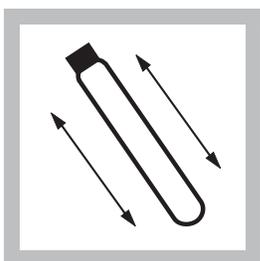
The cursor will move to the right, then the display will show:

**0.00 mg/L PO<sub>4</sub>**

*Note: For multiple samples, zero on the first sample. Read the remaining samples after adding the PhosVer 3 reagent. Subtract the reagent blank value from each reading.*

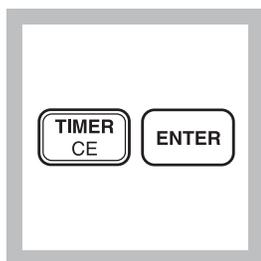


**12.** Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



**13.** Cap tightly and shake for 10-15 seconds.

*Note: The powder will not completely dissolve.*



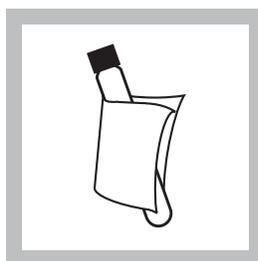
**14.** Press:

**TIMER ENTER**

A 2-minute reaction period will begin.

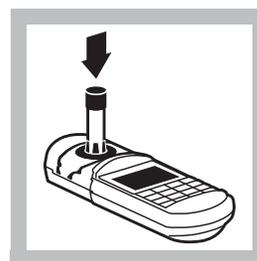
*Note: Read samples between 2 and 8 minutes after adding the PhosVer 3 reagent.*

*Note: A blue color will form if phosphate is present.*



**15.** After the timer beeps, clean the outside of the sample vial with a towel.

*Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



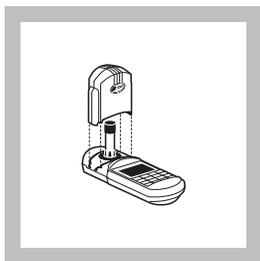
**16.** Place the prepared sample in the adapter

Push straight down on the top of the vial until it seats solidly into the adapter.

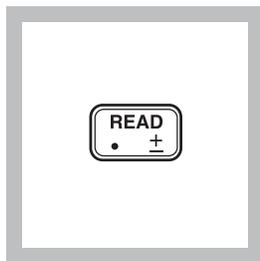
*Note: Do not move the vial from side to side as this can cause errors.*

## PHOSPHORUS, ACID HYDROLYZABLE, continued

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**17.** Tightly cover the vial with the instrument cap.



**18.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate ( $\text{PO}_4^{3-}$ ) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Section 1).

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, the sample may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

### Accuracy Check

*Note:* Clean glassware with 1:1 hydrochloric acid solution. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

## PHOSPHORUS, ACID HYDROLYZABLE, continued

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- d) Analyze each sample as described in the procedure. Use 5.0 mL of the prepared standard additions for each test; the concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Obtain a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, this can be prepared by pipetting 2 mL of a Voluette Ampule Standard for Phosphate, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid washed Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	9 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: <ol style="list-style-type: none"><li>1. Measure 25 mL of sample into a 50-mL beaker.</li><li>2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops.</li><li>3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with <i>step 1</i>.</li></ol>

## PHOSPHORUS, ACID HYDROLYZABLE, continued

Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 3.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.06$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1*.

### Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

### Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

## PHOSPHORUS, ACID HYDROLYZABLE, continued

### REQUIRED REAGENTS

Total and Acid Hydrolyzable Test 'N Tube Reagent Set..... 50 tests ..... 27427-45  
 Includes: (1) 272-42, (1) 1045-42, (1) 20847-66, (1) 21060-46, (50) Total and Acid  
 Hydrolyzable Test Vials\* (1) 27430-42

Description	Quantity Required		
	Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows .....	1	50/pkg	21060-46
Potassium Persulfate powder Pillows .....	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.0 N .....	2 mL	100 mL	1045-42
Total and Acid Hydrolyzable Test Vials .....	1	50/pkg	*
Water, deionized for reagent blanks.....	5 mL	100 mL	272-42

### REQUIRED APPARATUS

Description	Quantity Required		
	Per Test	Unit	Cat. No.
COD/TNT Adapter .....	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....		LTV082.52.40001	
Funnel, micro .....	1	each	25843-35
Pipet, TenSette, 1 to 10 mL.....	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet .....		50/pkg	21997-96
Test Tube Rack .....	1-3	each	18641-00

### OPTIONAL REAGENTS

Bromine Water, 30 g/L.....	29 mL	2211-20
Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....	500mL	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....	500 mL	884-49
Phenol Solution, 30 g/L .....	29 mL	2112-20
Phosphate Standard Solution, 1 mg/L as PO <sub>4</sub> <sup>3-</sup> .....	500 mL	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....	20/pkg	171-20H
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL .....	16/pkg	171-10
Sodium Hydroxide Standard Solution, 5.000 N .....	1000 mL	2450-53
Sulfuric Acid Standard Solution, 1.000 N .....	1 L	1270-53
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC).....	500 mL	28332-49
Water, deionized.....	4 L	272-56

\* These items are not sold separately.

## PHOSPHORUS, ACID HYDROLYZABLE, continued

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### OPTIONAL APPARATUS

Description	Units	Cat. No.
Ampule Breaker Kit, Voluette .....	each.....	21968-00
Ampule Breaker, PourRite .....	each.....	24846-00
Cylinder, graduated, mixing, 25 mL (3 required) .....	each.....	20886-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.52.30001	
Flask, volumetric, Class A, 100 mL.....	each.....	14574-42
pH Indicator Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
pH Meter, <i>sension</i> <sup>TM</sup> <b>I</b> , portable with electrode .....	each.....	51700-10
Pipet, TenSette, 0.1-1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 .....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 5.00 mL .....	each.....	14515-37
Pipet, volumetric, Class A, 2.00 mL .....	each.....	14515-36
Pipet Filler, safety bulb .....	each.....	14651-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

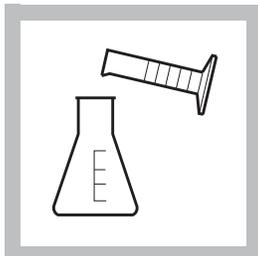
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**PHOSPHORUS, TOTAL**

For water, wastewater, and seawater

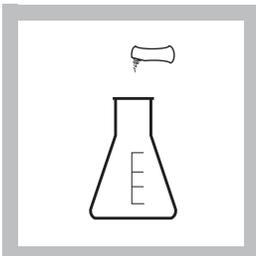
(Also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method\*  
USEPA Accepted for reporting wastewater analysis\*\*



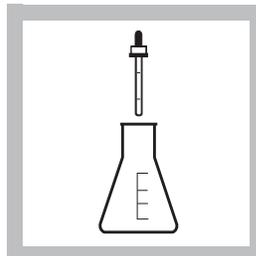
**1.** Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

*Note:* Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

*Note:* Adjust the pH of stored samples before digestion.

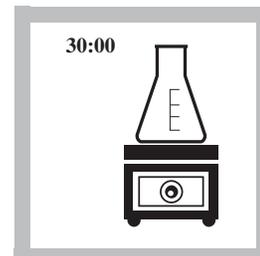


**2.** Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



**3.** Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

*Note:* Use the 1-mL calibrated dropper provided.



**4.** Place the flask on a hot plate. Boil gently for 30 minutes.

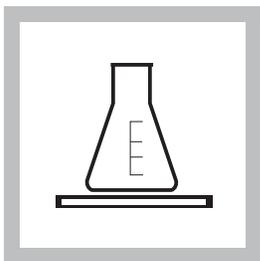
*Note:* Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

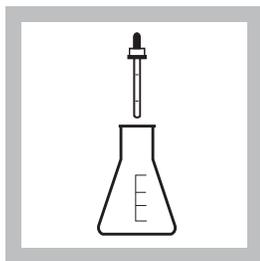
\*\* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B,5 & P E.

## PHOSPHORUS, TOTAL, continued

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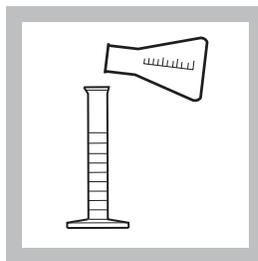


**5.** Cool the sample to room temperature.



**6.** Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

*Note:* Use the 1-mL calibrated dropper provided.



**7.** Pour the sample into a 25-mL graduated cylinder. Return the volume to 25 mL.

Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

*Note:* Use deionized water rinsings from the flask to adjust the volume.

*Note:* Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units before taking the difference.

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid-washed with 1:1 HCl and rinsed with deionized water. Do not use detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm to room temperature before testing. Correct results for volume additions; see *Volume Additions* (Section 1) for more information.

### Interferences

For turbid samples, use 50 mL of sample and double the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to add additional acid in Step 3 to drop the pH of the solution below 1.

## PHOSPHORUS, TOTAL, continued

### Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA approved for NPDES reporting.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

### REQUIRED REAGENTS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Potassium Persulfate Powder Pillows .....	1 pillow.....	100/pkg .....		2451-99
Sodium Hydroxide Solution, 5.0 N.....	2 mL .....	100 mL * MDB .....		2450-32
Sulfuric Acid Solution, 5.25 N.....	2 mL .....	100 mL * MDB .....		2449-32

### REQUIRED APPARATUS

Cylinder, graduated, 25 mL.....	2 .....	each .....		508-40
Flask, erlenmeyer, 50 mL.....	1 .....	each .....		505-41
Sample Cell, 10-20-25 mL, w/caps.....	2 .....	6/pkg .....		24019-06

### OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....	500mL .....			28330-49
Hydrochloric Acid, 6 N.....	500 mL .....			884-49
Sodium Hydroxide Solution, 5.0 N.....	1 L .....			2450-53
Sulfuric Acid .....	500 mL .....			979-49
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....			28332-49
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....			28331-49
Water, deionized .....	4L .....			272-56

\* Marked Dropper Bottle - Contact Hach for larger sizes.

## PHOSPHORUS, TOTAL, continued

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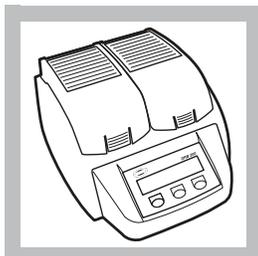
### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Cylinder, graduated, 50 mL .....	each.....	508-41
Flask, erlenmeyer, 125 mL .....	each.....	505-43
Hot Plate, 4" diameter, 120 Vac .....	each.....	12067-01
Hot Plate, 4" diameter, 240 Vac .....	each.....	12067-02
Pads, cooling, 4 x 4" .....	each.....	18376-00
pH Indicator Paper, 1 to 11 pH .....	5 rolls/pkg .....	391-33
pH Meter, <i>Sensio</i> <sup>TM</sup> <i>I</i> , portable with electrode .....	each.....	51700-10

### *For Technical Assistance, Price and Ordering*

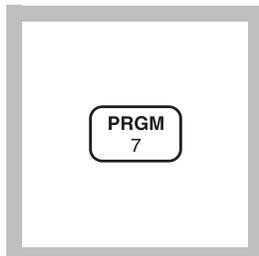
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**PHOSPHORUS, TOTAL (0.00 to 3.50 mg/L PO<sub>4</sub><sup>3-</sup>) For water, wastewater and seawater****PhosVer 3 with Acid Persulfate Digestion \* USEPA Accepted for reporting wastewater analysis \*\*  
Test 'N Tube Procedure**

**1.** Turn on the DRB200 Reactor. Heat the reactor to 150 °C.

*Note:* See DRB200 instrument manual for selecting preprogrammed temperature applications.



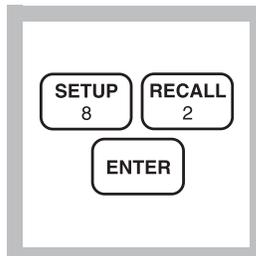
**2.** Enter the stored program number for total phosphorus, (PO<sub>4</sub><sup>3-</sup>), Test 'N Tube.

Press: **PRGM**

The display will show:

**PRGM ?**

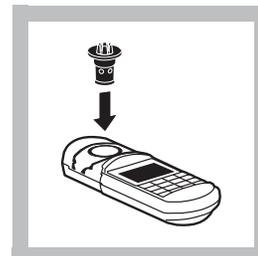
*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



**3.** Press: **82 ENTER**

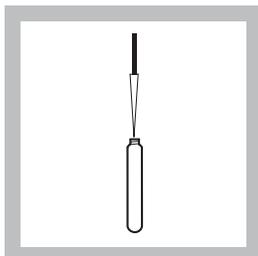
The display will show **mg/L, PO<sub>4</sub>** and the **ZERO** icon.

*Note:* For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.



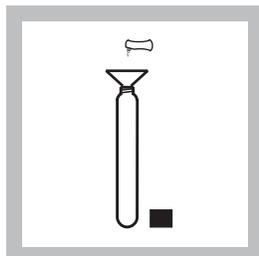
**4.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

*Note:* A diffuser band covers the light path holes on the adapter to give increased performance. The band should **NOT** be removed.

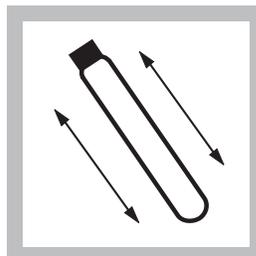


**5.** Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial.

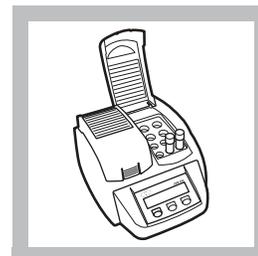
*Note:* Adjust the pH of stored samples to 6-8 before analysis.



**6.** Using a funnel, add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



**7.** Cap tightly and shake to dissolve.

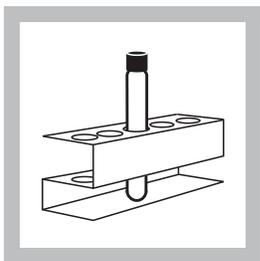


**8.** Place the vial in the DRB200 Reactor. Heat the vial for 30 minutes.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

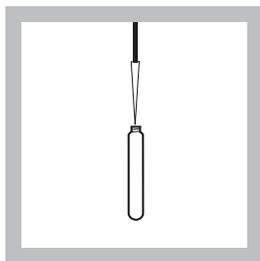
\*\* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B, 5 and P.E.

## PHOSPHORUS, TOTAL, continued

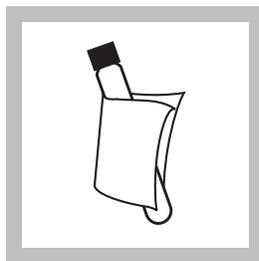


**9.** Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

*Note: Vials will be hot.*

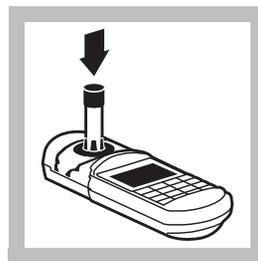


**10.** Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to the vial. Cap and mix.



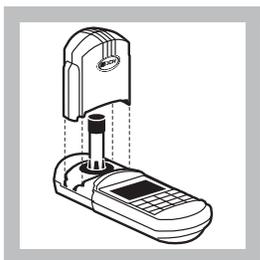
**11.** Clean the outside of the vial with a towel.

*Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*

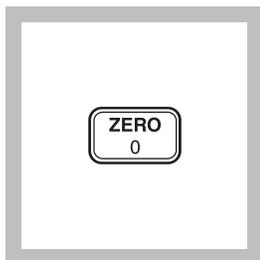


**12.** Place the sample vial in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



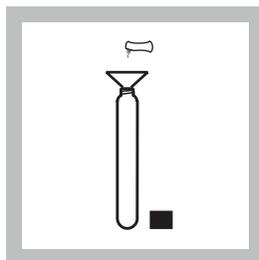
**13.** Tightly cover the vial with the instrument cap.



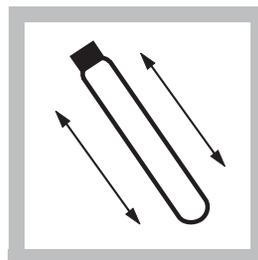
**14.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L PO<sub>4</sub>**

*Note: For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 reagent.*



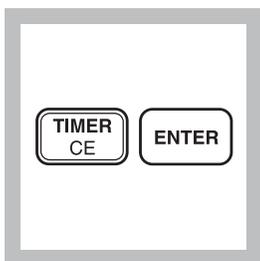
**15.** Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



**16.** Cap tightly and shake for 10-15 seconds.

*Note: The powder will not completely dissolve.*

## PHOSPHORUS, TOTAL, continued



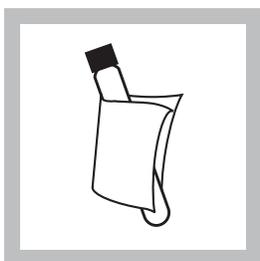
17. Press:

**TIMER ENTER**

A 2-minute waiting period will begin.

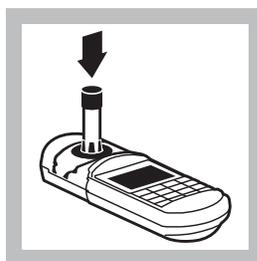
*Note: Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

*Note: A blue color will form if phosphate is present.*



18. After the timer beeps, clean the outside of the sample vial with a towel.

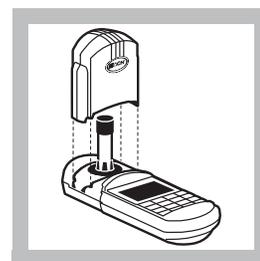
*Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



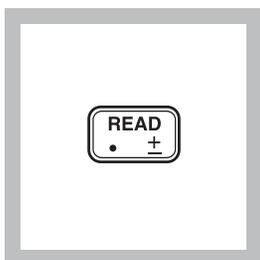
19. Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note: Do not move the vial from side to side as this can cause errors.*



20. Tightly cover the vial with the instrument cap.



21. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate ( $\text{PO}_4^{3-}$ ) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

### IMPORTANT NOTE:

The test range for total phosphate is limited to 0 to 3.5 mg/L  $\text{PO}_4^{3-}$ . Values above 3.5 mg/L may be used to estimate dilution ratios, but should NOT be used for reporting purposes. If a value above 3.5 mg/L  $\text{PO}_4^{3-}$  is obtained, dilute the sample and repeat the digestion and the colorimetric test.

## PHOSPHORUS, TOTAL, continued

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Neutralize and warm the sample to room temperature before analysis. Correct test results for volume additions; see *Volume Additions* in *Section 1*.

### Accuracy Check

*Note:* Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

### Standard Additions Method

- a) Fill three 25 mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.
- d) Analyze each sample as described in the procedure using 5.0 mL of the prepared standard additions for each test. The concentration should increase 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e) If these increases do not occur, see *Standard Additions* (Section 1).

### Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution (see Optional Reagents). Or, prepare a standard by pipetting 2 mL of solution a Voluette Ampule Standard for Phosphate Standard, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid-cleaned Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described. The mg/L  $\text{PO}_4^{3-}$  reading should be 1.0 mg/L.

OR

Prepare a 2.5 mg/L standard solution by pipetting 5 mL of a 50-mg/L Phosphate Voluette Ampule Standard into an

## PHOSPHORUS, TOTAL, continued

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acid-washed 100-mL Class A volumetric flask. Dilute to the mark with deionized water.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 3.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.06$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1*.

### Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	90 mg/L
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

Store PhosVer 3 Reagent Powder Pillows in a cool, dry environment.

## PHOSPHORUS, TOTAL, continued

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### Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

### Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

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### REQUIRED REAGENTS

Total Phosphorus Test 'N Tube Reagent Set ..... 50 tests ..... 27426-45  
Includes: (1) 272-42, (1) 20847-66, (1) 21060-46, (1) 27430-42, (50) Acid Dilution Vials\*

Description	Quantity Required		
	Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows .....	1	50/pkg	21060-46
Potassium Persulfate powder Pillows .....	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N .....	2 mL	100 mL	27430-42
Test 'N Tube Acid Dilution Vials .....	1	50/pkg	*
Water, deionized for reagent blank .....	5 mL	100 mL	272-42

### REQUIRED APPARATUS

COD/TNT Adapter .....	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....		LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....		LTV082.52.40001	
Funnel, micro .....	1	each	25843-35
Test Tube Rack .....	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL.....	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet .....	varies	50/pkg	21997-96

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\* These items are not sold separately.

## PHOSPHORUS, TOTAL, continued

### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Drinking Water Standard, Inorganic, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup> .....	500mL .....	28330-49
Total and Acid Hydrolyzable Test 'N Tube Reagent Set .....	each.....	27427-45
Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....	500 mL .....	884-49
Phosphate Standard Solution, 1 mg/L as PO <sub>4</sub> <sup>3-</sup> .....	500 mL .....	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....	20/pkg.....	171-20H
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL .....	16/pkg.....	171-10
Sodium Hydroxide Standard Solution, 5.0 N .....	1 L.....	2450-53
Total and Acid Hydrolyzable Test 'N Tube Reagent Set .....	each.....	27427-45
Wastewater Effluent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC) .....	500 mL .....	28332-49
Water, deionized .....	4L .....	272-56

### OPTIONAL APPARATUS

Ampule Breaker Kit .....	each.....	21968-00
Ampule Breaker, PourRite ampules .....	each.....	24846-00
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm .....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm .....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm .....	LTV082.52.30001	
Cylinder, graduated, mixing, 25 mL (3 required) .....	each.....	20886-40
pH Indicator Paper, 1 to 11 pH units .....	5 rolls/pkg.....	391-33
pH Meter, <i>Sensio</i> <sup>TM</sup> <b>I</b> , portable with electrodes .....	each.....	51700-10
Pipet Filler, safety bulb .....	each.....	14651-00
Pipet, volumetric, Class A, 5.00 mL .....	each.....	14515-37
Pipet, volumetric, Class A, 2.00 mL .....	each.....	14515-36
Pipet, TenSette, 0.1-1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 .....	1000/pkg.....	21856-28

### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

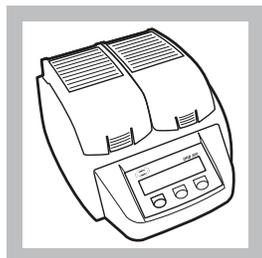
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**PHOSPHORUS, TOTAL, HR** (0.0 to 100.0 mg/L PO<sub>4</sub><sup>3-</sup>)

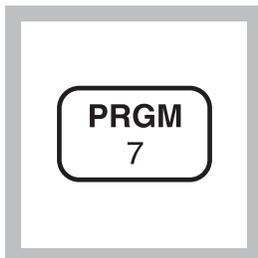
**Molybdovanadate Method with Acid Persulfate Digestion\***  
**Test 'N Tube™ Procedure**

**For water and wastewater**



**1.** Turn on the DRB200 Reactor. Heat to 150 °C.

*Note: See DRB200 instrument manual for selecting preprogrammed temperature applications*

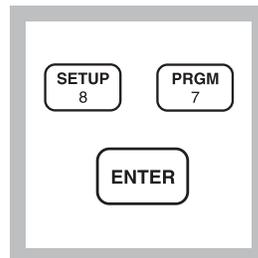


**2.** Enter the stored program number for phosphorus total high range, Test 'N Tube.

Press: **PRGM**

The display will show:

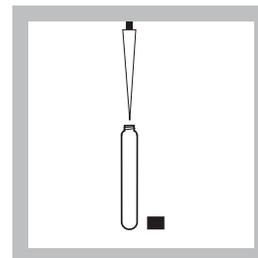
**PRGM ?**



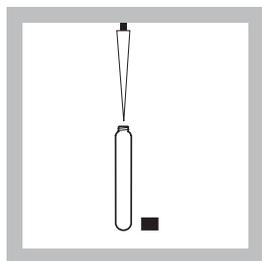
**3.** Press: **87 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

*Note: For alternate forms (P, P<sub>2</sub>O<sub>5</sub>), press the **CONC** key.*

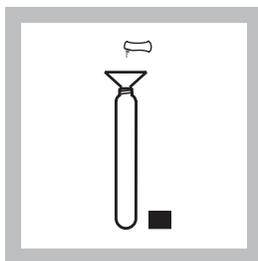


**4.** Use a TenSette® Pipet to add 5.0 mL of deionized water to a Total Phosphorus Test 'N Tube Vial (the blank).

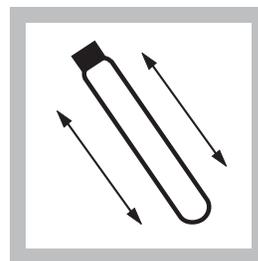


**5.** Use a TenSette Pipet to add 5.0 mL of sample to a Total Phosphorus Test 'N Tube Vial (the sample).

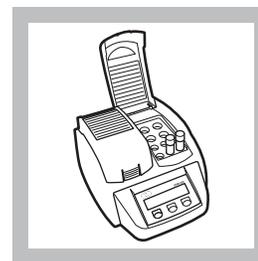
*Note: Adjust the pH of stored samples to 6–8 before analysis.*



**6.** Use a funnel to add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to each vial.



**7.** Cap tightly and shake to dissolve.

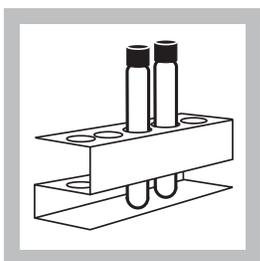


**8.** Place the vials in the DRB200 Reactor. Heat for 30 minutes.

Press: **TIMER ENTER** to time the heating period.

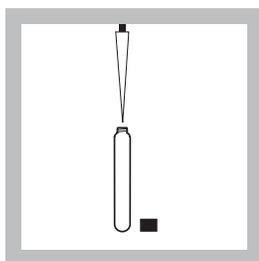
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## PHOSPHORUS, TOTAL, HR, continued

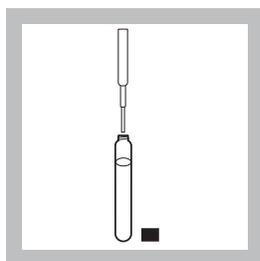


**9.** Carefully remove the vials from the reactor. Place them in a test tube rack and allow to cool to room temperature (18–25 °C).

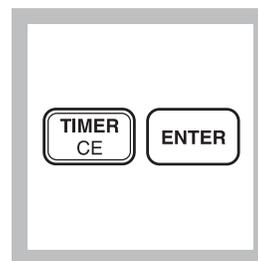
*Note:* Vials will be hot.



**10.** Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to each vial. Cap and invert to mix.



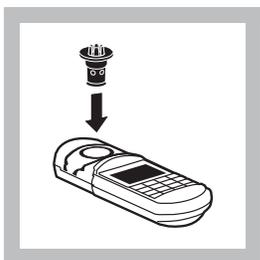
**11.** Use a polyethylene dropper to add 0.5 mL of Molybdovanadate Reagent to each vial. Cap and invert to mix.



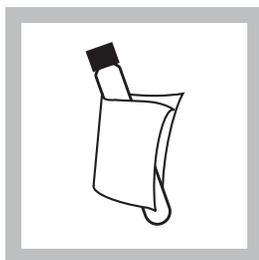
**12.** Press:  
**TIMER ENTER**

A 7-minute reaction period will begin.

*Note:* Read the samples between 7 and 9 minutes.

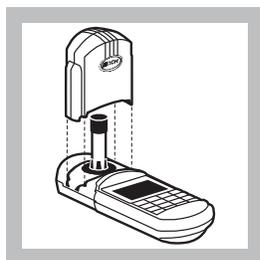


**13.** Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



**14.** Clean the outside of the vials with a towel.

*Note:* Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.

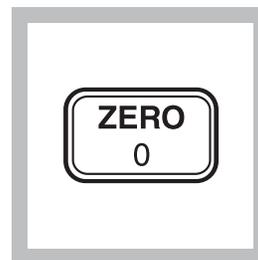


**15.** When the timer sounds, place the blank vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

*Note:* Do not move the vial from side to side as this can cause errors.



**16.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.0 mg/L PO<sub>4</sub>**

*Note:* Reagent blanks for each lot of reagents may be used more than once, but should not be used for longer than one day.

## PHOSPHORUS, TOTAL, HR, continued

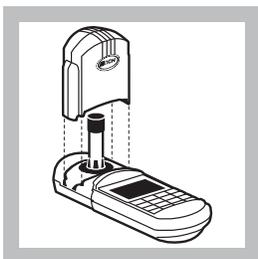
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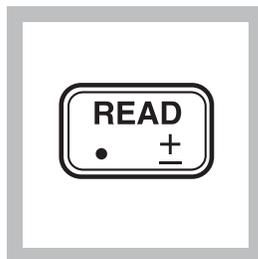
**17.** Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

*Note:* Do not move the vial from side to side as this can cause errors.



**18.** Tightly cover the vial with the instrument cap.



**19.** Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate ( $\text{PO}_4^{3-}$ ) will be displayed.

*Note:* For best results, use Standard Adjust with each new lot of reagent. (See Accuracy Check.)

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### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning the glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated  $\text{H}_2\text{SO}_4$  (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N NaOH before analysis.

Correct test results for volume additions; see *Volume Additions* in Section 1 of the *DR/890 Procedures Manual*.

## PHOSPHORUS, TOTAL, HR, continued

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### Accuracy Check

*Note: Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

#### Standard Additions Method

- a. Fill each of three 10-mL graduated mixing cylinders with 10 mL of sample.
- b. Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$  (Cat. No. 14242-10).
- c. Use a TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of the water sample prepared in *step a*. Mix well.
- d. Analyze samples from *step c* as described in the procedure. Use 5.0 mL of the prepared sample for each test. The concentration should increase: 5 mg/L, 10 mg/L, and 15 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

#### Standard Solution Method

To check accuracy, prepare an 80 mg/L standard by pipetting 8.0 mL of solution from a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as  $\text{PO}_4^{3-}$  into an acid-cleaned, Class A, 50-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/L  $\text{PO}_4^{3-}$  standard solution, press the **SETUP** key and scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1 of the Procedures Manual* for more information.

## PHOSPHORUS, TOTAL, HR, continued

### Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated. <sup>1</sup>
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See <i>pH Interferences</i> in <i>Section 1</i> of the <i>DR/890 Procedures Manual</i> . Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, Cold (less than 18 °C)	Causes a negative interference.
Temperature, Hot (greater than 25 °C)	Causes a positive interference. Post-digestion samples should be brought to room temperature (18–25 °C) before the addition of the Molybdovanadate Reagent or sodium hydroxide.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al <sup>3+</sup> , Fe <sup>3+</sup> , Mg <sup>2+</sup> , Ca <sup>2+</sup> , Ba <sup>2+</sup> , Sr <sup>2+</sup> , Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Cd <sup>2+</sup> , Mn <sup>2+</sup> , NO <sub>3</sub> <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , SO <sub>3</sub> <sup>2-</sup> , Pb <sup>2+</sup> , Hg <sup>+</sup> , Hg <sup>2+</sup> , Sn <sup>2+</sup> , Cu <sup>2+</sup> , Ni <sup>2+</sup> , Ag <sup>+</sup> , U <sup>4+</sup> , Zr <sup>4+</sup> , AsO <sub>3</sub> <sup>-</sup> , Br <sup>-</sup> , CO <sub>3</sub> <sup>2-</sup> , ClO <sub>4</sub> <sup>-</sup> , CN <sup>-</sup> , IO <sub>3</sub> <sup>-</sup> , SiO <sub>4</sub> <sup>4-</sup> .	

<sup>1</sup> Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

## PHOSPHORUS, TOTAL, HR, continued

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### Method Performance

#### Precision

In a single laboratory, using a standard solution of 80.0 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 3.0$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit

The estimated detection limit for program 87 is 7.0 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

### Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

### Sample Disposal Information

The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

### Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

## PHOSPHORUS, TOTAL, HR, continued

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### Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	87	18	0
2	4	19	0
3	73	20	80
4	0	21	79
5	0	22	52
6	0	23	0
7	0	24	0
8	0	25	80
9	0	26	0
10	0	27	0
11	0	28	0
12	66	29	0
13	175	30	80
14	48	31	50
15	32	32	79
16	0	33	53
17	0	34	0
35	62	46	0

## PHOSPHORUS, TOTAL, HR, continued

Line Number	Entry	Line Number	Entry
36	166	47	15
37	246	48	7
38	148	49	8
39	63	50	1
40	63	51	164
41	78	52	0
42	252	53	0
43	4	54	40
44	76	55	0
45	128	56	255

### REQUIRED REAGENTS

Total High Range Phosphorus Test 'N Tube™ Reagent Set ..... 50 vials ..... 27672-45  
 Includes: (50) Total Phosphorus Test 'N Tube™ Vials\*, (2) 272-42, (1) 20847-66  
 (1) 20760-26, (1) 27430-42

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Molybdovanadate Reagent .....	0.5 mL	25 mL	20760-26
Potassium Persulfate Powder Pillows.....	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N .....	2 mL	100 mL	27430-42
Total Phosphorus Test 'N Tube™ Vials.....	1	50/pkg	*
Water, deionized.....	100 mL		272-42

### REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes .....	LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes .....	LTV082.52.40001
COD/TNT Adapter, DR/800 series.....	1 ..... each ..... 48464-00
Dropper, LDPE, 0.5 to 1.0 mL.....	1 ..... 20/pkg ..... 21247-20
Pipet, TenSette®, 1 to 10 mL .....	1 ..... each ..... 19700-10
Pipet Tips, for 19700-10 TenSette® Pipet.....	varies .. 50/pkg ..... 21997-96
Test Tube Rack .....	1-3 ..... each ..... 18641-00

\* These items are not sold separately.

## PHOSPHORUS, TOTAL, HR, continued

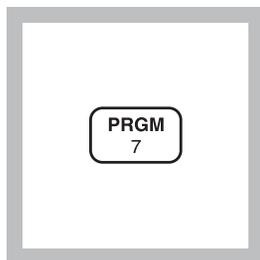
### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, PourRite™ ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2-mL	20/pkg	14242-20
Phosphate Standard Solution, Voluette™ ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10-mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Sulfuric Acid, ACS, concentrated	500 mL	979-491
Wastewater Influent Standard, Inorganic (NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC)	500 mL	28331-49

### OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 10 mL (3 required)	each	20886-38
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm	LTV082.52.30001	
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	200/pkg	13530-01
Flask, filtering, 500-mL	each	546-49
Flask, volumetric, Class A, 50-mL	each	14574-41
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, <i>sensIon</i> ™ <i>I</i> , portable with electrode	each	51700-10
Pipet Filler, Safety Bulb	each	14651-00
Pipet, TenSette®, 0.- to 1.0-mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 8.00-mL	each	14515-08
Stopper, No. 7 one hole	6/pkg	2119-07
Tubing, rubber	12 feet	560-19



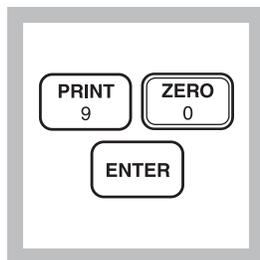
**SILICA, Low Range (0 to 1.60 mg/L)****Heteropoly Blue Method\***

1. Enter the stored program number for low range silica ( $\text{SiO}_2$ ).

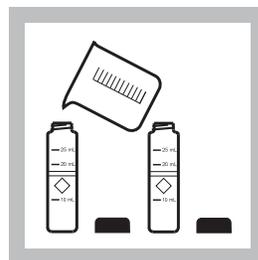
Press: **PRGM**

The display will show:

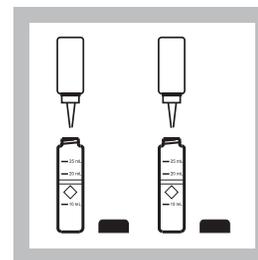
**PRGM ?**



2. Press: **90 ENTER**  
The display will show **mg/L, SiO<sub>2</sub>** and the **ZERO** icon.

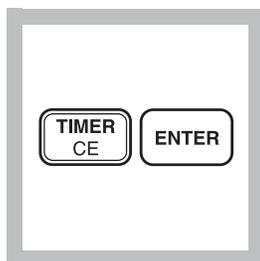


3. Fill two sample cells to the 10-mL line with sample.



4. Add 15 drops of Molybdate 3 Reagent to each sample cell. Swirl to mix.

*Note: For greatest accuracy, hold dropping bottle vertical.*

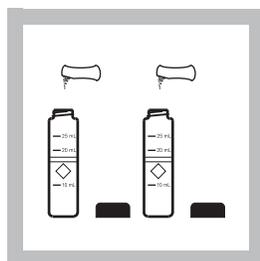


5. Press:

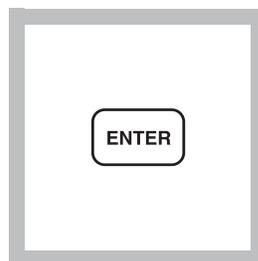
**TIMER ENTER**

A 4-minute reaction period will begin.

*Note: Reaction time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes. If the sample temperature is 30 °C (86 °F), wait 2 minutes.*



6. After the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.



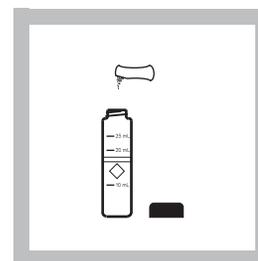
7. The display will show:

**1:00 TIMER 2**

Press: **ENTER**

A 1-minute reaction period will begin. Phosphate interference is eliminated during this period.

*Note: The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait two minutes. If the sample is 30 °C (86 °F), wait 30 seconds.*



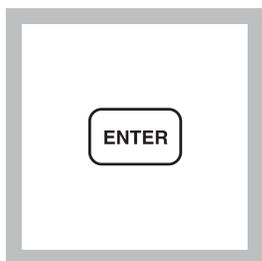
8. After the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells (the prepared sample). Invert to mix.

*Note: The sample cell without the Amino Acid F Reagent is the blank.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## SILICA, Low Range, continued

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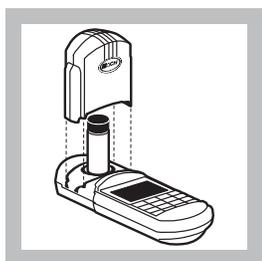
**9.** The display will show:

**2:00 TIMER 3**

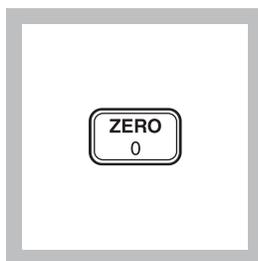
Press: **ENTER**

A 2-minute reaction period will begin.

*Note: A blue color will develop if silica is present.*



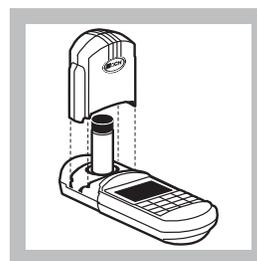
**10.** After the timer beeps, place the blank (solution without Amino Acid F Reagent) into the cell holder. Tightly cover the sample cell with the instrument cap.



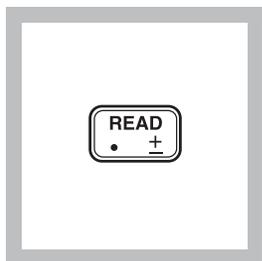
**11.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L SiO<sub>2</sub>**



**12.** Place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**13.** Press: **READ**

The cursor will move to the right, then the result in mg/L SiO<sub>2</sub> will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

## SILICA, Low Range, continued

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### Sampling and Storage

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

### Accuracy Check

#### Standard Additions Method

- a) Open a Silica Standard Solution Bottle, 25 mg/L SiO<sub>2</sub>.
- b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 10-mL samples. Mix thoroughly.
- c) Analyze each sample as described above. The silica concentration should increase 0.25 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions in Section 1* for more information.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L Standard Solution (see *Optional Reagents*), press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.00** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a instrument, a single operator obtained a standard deviation of ±0.025 mg/L silica.

#### Estimated Detection Limit (EDL)

The estimated detection limit for program 90 is 0.020 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*. If testing for very low levels of silica, use the ultra-low range silica method on the Hach DR/2010 or DR/4000 Spectrophotometers.

## SILICA, Low Range, continued

### Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Phosphate	Phosphate does not interfere at levels less than 50 mg/L PO <sub>4</sub> . At 60 mg/L PO <sub>4</sub> , an interference of -2% occurs. At 75 mg/L PO <sub>4</sub> the interference is -11%.
Iron	Large amounts of iron interfere.
Slow reacting forms of silica	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in <i>Standard Methods for the Examination of Water and Wastewater</i> under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate pretreatment.
Sulfides	Interfere at all levels
Turbidity	Eliminated by zeroing the instrument with the original sample.

### Reagent Preparation

To prepare Amino Acid F Reagent Solution, dissolve 11.4 grams of Amino Acid F Reagent Powder in 100 mL of 1.0 N Sodium Hydroxide Solution. The solution is stable for at least one month if stored in a plastic bottle.

### Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Acid reduces the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

## SILICA, Low Range, continued

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### REQUIRED REAGENTS

Low Range Silica Reagent Set, 10 mL sample (100 tests) .....			<b>Cat. No.</b>
			24593-00
Includes: (1) 22540-69, (1) 21062-69 (2) 1995-26			

<b>Description</b>	<b>Quantity Required</b>		<b>Cat. No.</b>
	<b>Per Test</b>	<b>Units</b>	
Amino Acid F Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	22540-69
Citric Acid Powder Pillows.....	2 pillows.....	100/pkg .....	21062-69
Molybdate 3 Reagent .....	28 drops .....	50 mL SCDB .....	1995-26

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06
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### OPTIONAL REAGENTS

Silica Standard Solution, 1.00 mg/L SiO <sub>2</sub> .....	500 mL .....	1106-49
Silica Standard Solution, 25 mg/L SiO <sub>2</sub> .....	236 mL .....	21225-31
Sodium Bicarbonate, ACS .....	454 g .....	776-01
Sodium Hydroxide Standard Solution, 1.000 N.....	900 mL .....	1045-53
Sulfuric Acid Standard Solution, 1.0 N .....	1000 mL .....	1270-53

### OPTIONAL APPARATUS

Bottle, 118 mL, polyethylene, oblong.....	6/pkg .....	23184-06
Dropper, 0.5- & 1.0-mL marks.....	6/pkg .....	23185-06
Pipet, serological, 2 mL, poly .....	each .....	2106-36
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 Pipet .....	1000/pkg .....	21856-28
<i>Standard Methods for the Examination of Water and Wastewater</i> .....	each .....	22708-00
Thermometer, - 20 to 110 °C, Non-Mercury.....	each .....	26357-02

### *For Technical Assistance, Price and Ordering*

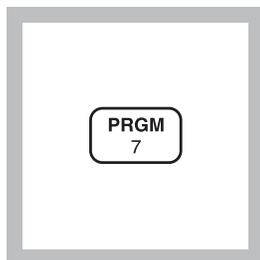
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**SILICA, High Range (0 to 75.0 mg/L)**

For water and seawater

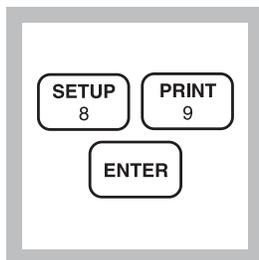
**Silicomolybdate Method**

1. Enter the stored program number for high range silica ( $\text{SiO}_2$ ).

Press: **PRGM**

The display will show:  
**PRGM ?**

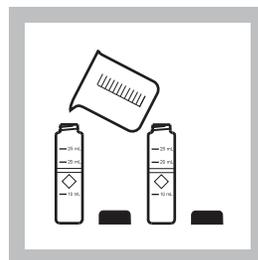
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **89 ENTER**

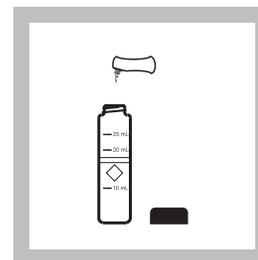
The display will show **mg/L, SiO<sub>2</sub>** and the **ZERO** icon.

*Note: For alternate form (Si), press the **CONC** key.*

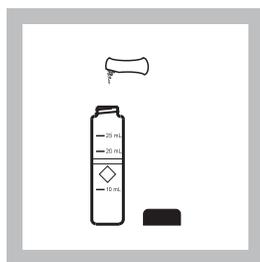


3. Fill two sample cells with 10 mL of sample. Set one aside as the blank.

*Note: Sample temperature should be 15 to 25 °C (59 to 77 °F).*

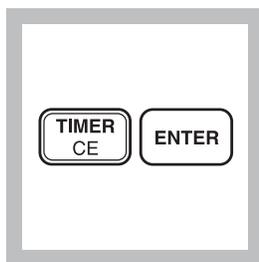


4. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.



5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Cap and invert to mix.

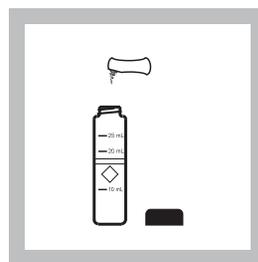
*Note: Silica or phosphate will cause a yellow color to develop.*



6. Press:

**TIMER ENTER**

A 10-minute reaction period will begin.



7. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

*Note: The yellow color due to phosphate will disappear.*



8. The display will show: **2:00 Timer 2**

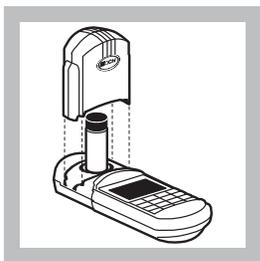
Press: **ENTER**

A two-minute reaction period will begin.

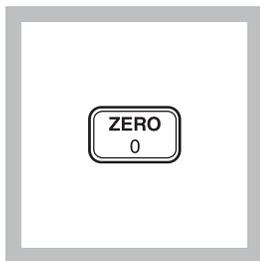
*Note: Perform Steps 9-12 within three minutes after the timer beeps.*

## SILICA, High Range, continued

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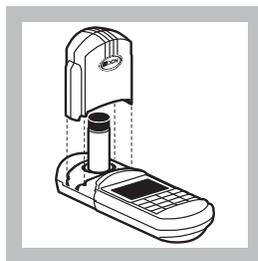


**9.** When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

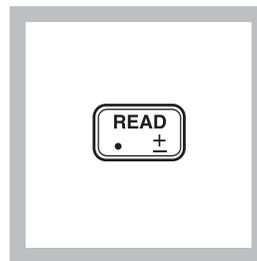


**10.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.0 mg/L SiO<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**11.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**12.** Press: **READ**  
The cursor will move to the right, then the result in mg/L silica (SiO<sub>2</sub>) will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

### Accuracy Check

#### Standard Additions Method

- a) Open a High Range Silica Standard Solution, 1000 mg/L SiO<sub>2</sub>.
- b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.
- c) Analyze each sample as described above. The silica concentration should increase 10.0 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

## SILICA, High Range, continued

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### Standard Solution Method

To check the accuracy of the method, use the Silica Standard Solutions, 25 and 50 mg/L as SiO<sub>2</sub>, listed under Optional Reagents. Analyze according to the above procedure using deionized water as the blank.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 50.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 50.0 mg/L SiO<sub>2</sub> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L silica.

### Estimated Detection Limit

The estimated detection limit for program 89 is 1.00 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

## Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe <sup>2+</sup> and Fe <sup>3+</sup> interfere.
Phosphate	Does not interfere below 50 mg/L PO <sub>4</sub> <sup>3-</sup> . At 60 mg/L PO <sub>4</sub> <sup>3-</sup> , a negative 2% interference occurs. At 75 mg/L PO <sub>4</sub> <sup>3-</sup> the interference is negative 11%.
Sulfides (S <sup>2-</sup> )	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the*

## SILICA, High Range, continued

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*Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

### Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

---

### REQUIRED REAGENTS

High Range Silica Reagent Set, 10-mL sample (100 tests) ..... 24296-00  
Includes: (1) 21074-69, (1) 21062-69, (1) 21073-69

Description	Quantity Required		Cat. No.
	Per Test	Units	
Acid Reagent Powder Pillows for High Range Silica ..	1	100/pkg	21074-69
Citric Acid Powder Pillows .....	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for HR Silica .....	1	100/pkg	21073-69

### REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap ..... 2 ..... 6/pkg ..... 24019-06

### OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L ..... 500 mL ..... 1403-49  
Silica Standard Solution, 25 mg/L ..... 236 mL ..... 21225-31  
Silica Standard Solution, 50 mg/L ..... 200 mL ..... 1117-29  
Silica Standard Solution, 1000 mg/L ..... 500 mL ..... 194-49  
Sodium Bicarbonate, ACS ..... 454 g ..... 776-01  
Sulfuric Acid Standard Solution, 1.000 N ..... 100 mL MDB ..... 1270-32  
Water, deionized ..... 4 L ..... 272-56

### OPTIONAL APPARATUS

Pipet, TenSette, 0.1 to 1.0 mL ..... each ..... 19700-01  
Pipet Tips, for 19700-01 Pipet ..... 50/pkg ..... 21856-96  
Pipet Tips, for 19700-01 Pipet ..... 1000/pkg ..... 21856-28  
*Standard Methods for the Examination of Water and Wastewater* ..... each ..... 22708-00  
Thermometer, -20 to 110 °C, Non-Mercury ..... each ..... 26357-02

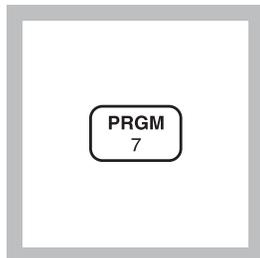
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**SILICA, Ultra High Range (0 to 200 mg/L)**

For water and seawater

**Silicomolybdate Method**

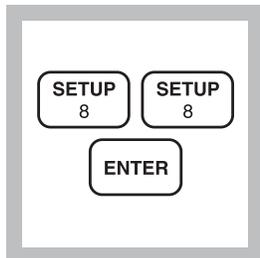
1. Enter the stored program number for ultra high range silica ( $\text{SiO}_2$ ).

Press: **PRGM**

The display will show:

**PRGM ?**

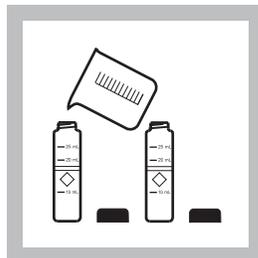
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **88 ENTER**

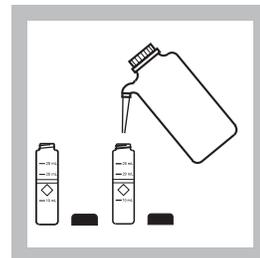
The display will show **mg/L, SiO<sub>2</sub>** and the **ZERO** icon.

*Note: For alternate form (Si), press the **CONC** key.*

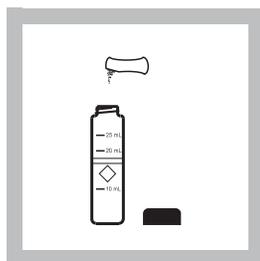


3. Fill 2 sample cells with 10 mL of sample.

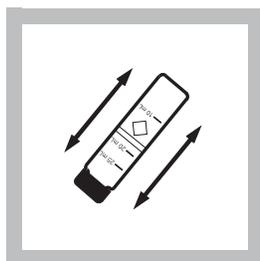
*Note: Sample temperature should be 15 to 25 °C (59 to 77 °F).*



4. Fill both sample cells to the 25-mL line with deionized water. Set one sample cell aside as the blank.

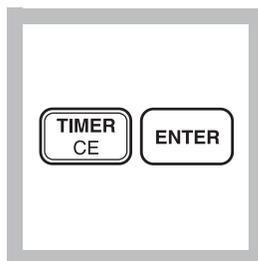


5. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.



6. Add the contents of one Acid Reagent Powder Pillow for High Range Silica to the prepared sample. Cap and invert to mix.

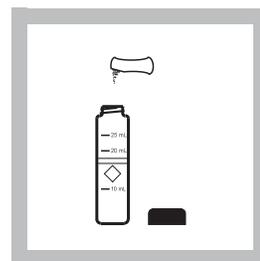
*Note: Silica or phosphate will cause a yellow color to develop.*



7. Press:

**TIMER ENTER**

A 10-minute reaction period will begin.

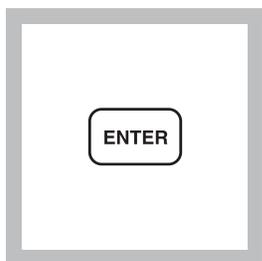


8. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

*Note: The yellow color due to phosphate will disappear.*

## SILICA, Ultra High Range, continued

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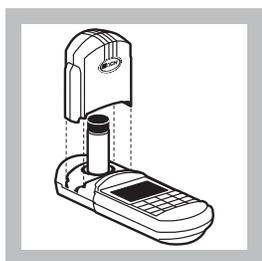


**9.** The display will show: **2:00 Timer 2**

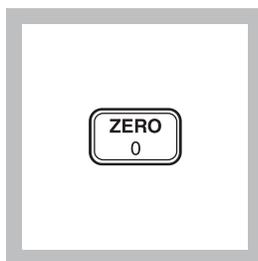
Press: **ENTER**

A two-minute reaction period will begin.

*Note: Perform Steps 10-13 within three minutes after the timer beeps.*



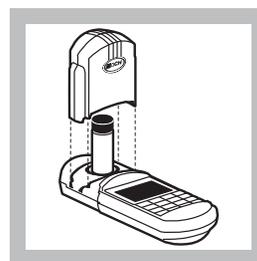
**10.** When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



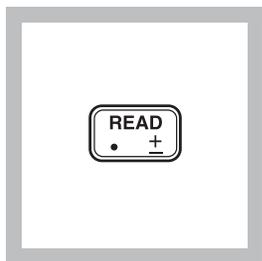
**11.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 mg/L SiO<sub>2</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**12.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**13.** Press: **READ**

The cursor will move to the right, then the result in mg/L silica (SiO<sub>2</sub>) will be displayed.

*Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

## SILICA, Ultra High Range, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

### Accuracy Check

#### Standard Additions Method

- a) Open a High Range Silica Standard Solution, 1000 mg/L SiO<sub>2</sub>.
- b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.
- c) Analyze each sample as described above. The silica concentration should increase 4 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions in Section 1* for more information.

#### Standard Solution Method

To prepare a 160-mg/L silica standard, pipet 40.0 mL of a 1000-mg/L Silica Standard Solution into a 250-mL volumetric flask. Dilute to the line with deionized water. Analyze according to the above procedure using deionized water as the blank.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 160-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the **STD** setup option. Press **ENTER** to activate the standard adjust option. Then enter **160.** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 100.0 mg/L SiO<sub>2</sub> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2.0 mg/L silica.

## SILICA, Ultra High Range, continued

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### Estimated Detection Limit

The estimated detection limit for program 88 is 3.0 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe <sup>2+</sup> and Fe <sup>3+</sup> interfere.
Phosphate	Does not interfere below 50 mg/L PO <sub>4</sub> <sup>3-</sup> . At 60 mg/L PO <sub>4</sub> <sup>3-</sup> , a negative 2% interference occurs. At 75 mg/L PO <sub>4</sub> <sup>3-</sup> the interference is negative 11%.
Sulfides (S <sup>2-</sup> )	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

### Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

## SILICA, Ultra High Range, continued

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### REQUIRED REAGENTS

			<b>Cat. No.</b>
High Range Silica Reagent Set, 25-mL sample (100 tests) .....			22443-00
Includes: (1) 1042-99, (1) 14548-99, (1) 1041-99			

Description	Quantity Required		Cat. No.
	Per Test	Units	
Acid Reagent Powder Pillows for High Range Silica... 1 .....	1	100/pkg	1042-99
Citric Acid Powder Pillows..... 1 .....	1	100/pkg	14548-99
Molybdate Reagent Powder Pillows for HR Silica..... 1 .....	1	100/pkg	1041-99
Water, deionized .....	30 mL	4 L	272-56

### REQUIRED APPARATUS

Sample 10-20-15 mL, w/ cap .....	2	6/pkg	24019-06
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### OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L .....	500 mL	1403-49
Silica Standard Solution, 25 mg/L .....	236 mL	21225-31
Silica Standard Solution, 50 mg/L .....	200 mL	1117-29
Silica Standard Solution, 1000 mg/L .....	500 mL	194-49
Sodium Bicarbonate, ACS .....	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N.....	100 mL MDB	1270-32

### OPTIONAL APPARATUS

Flask, volumetric, 250 mL, Class A.....	each	14574-46
Pipet, TenSette, 0.1 to 1.0 mL.....	each	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg	21856-96
Pipet, volumetric, Class A, 100 mL .....	each	14515-42
Pipet Filler, safety bulb .....	each	14651-00
<i>Standard Methods for the Examination of Water and Wastewater</i> .....	each	22708-00
Thermometer, -20 to 110 °C, Non-Mercury.....	each	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

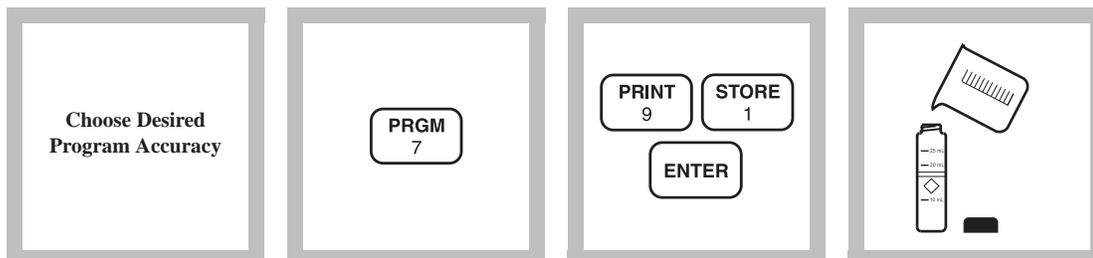
Outside the U.S.A.—Contact the Hach office or distributor serving you.



**SULFATE (0 to 70 mg/L)**

For water, wastewater, and seawater

**SulfaVer 4 Method\*** (Powder Pillows or AccuVac Ampuls); USEPA accepted for reporting wastewater analysis\*\*

**Using Powder Pillows**

**1.** A User-Entered Calibration is necessary to obtain the most accurate results. See the *User Calibration* section at the back of this procedure. Program 91 can be used for process control or applications where a high degree of accuracy is not needed.

*Note: The nature of turbidimetric tests and reagent lot variation requires user calibration for best results.*

**2.** Enter the stored program number for sulfate ( $\text{SO}_4^-$ ).  
Press: **PRGM**  
The display will show:  
**PRGM ?**

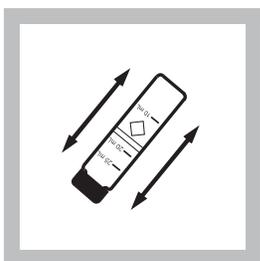
**3.** Press: **91 ENTER** or the program number selected for a user-entered calibration.  
The display will show **mg/L, SO4** and the **ZERO** icon.

**4.** Fill a clean sample cell with 10 mL of sample.  
*Note: Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

\*\* Procedure is equivalent to USEPA method 375.4 for wastewater.

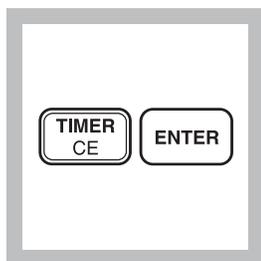
## SULFATE, continued



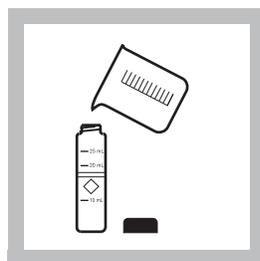
5. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and invert several times to mix.

*Note: A white turbidity will develop if sulfate is present in the sample.*

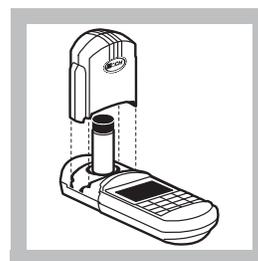
*Note: Accuracy is not affected by undissolved powder.*



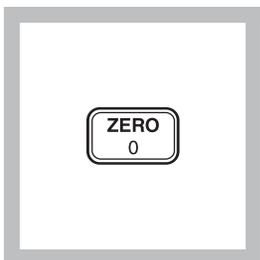
6. Press: **TIMER ENTER**  
A 5-minute reaction period will begin. Allow the cell to stand undisturbed.



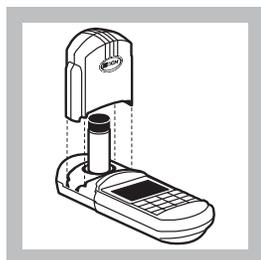
7. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).



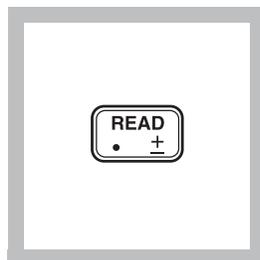
8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



9. Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0 mg/L SO<sub>4</sub>**



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



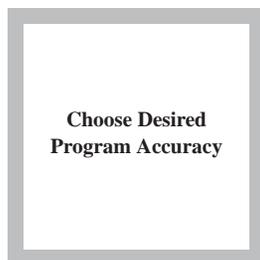
11. Press: **READ**  
The cursor will move to the right, then the result in mg/L sulfate will be displayed.

*Note: If Program 91 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

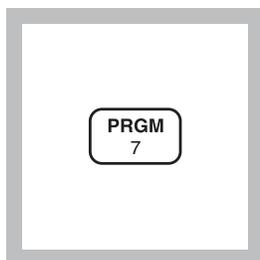
*Note: Clean the sample cells with soap and a brush.*

## SULFATE, continued

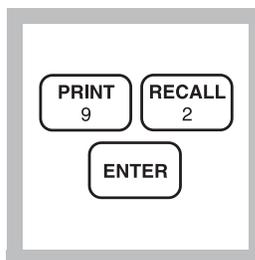
### Using AccuVac Ampuls



**1.** A User-Entered Calibration is necessary to obtain the most accurate results. See User Calibration Section at the back of this procedure. Program 92 can be used for process control or applications where a high degree of accuracy is not needed.



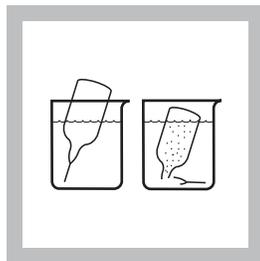
**2.** Enter the stored program number for sulfate ( $\text{SO}_4^-$ )-AccuVac Ampuls. Press: **PRGM**  
The display will show:  
**PRGM ?**



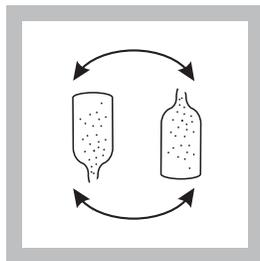
**3.** Press: **92 ENTER**  
The display will show **mg/L, SO4** and the **ZERO** icon.



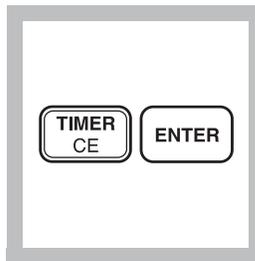
**4.** Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.  
*Note: Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*



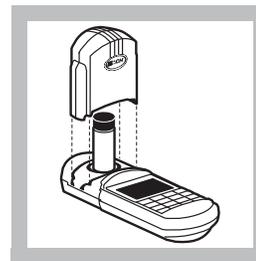
**5.** Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample.  
*Note: Keep tip immersed until the ampul fills completely.*



**6.** Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.  
*Note: A white turbidity will develop if sulfate is present.*  
*Note: Accuracy is not affected by undissolved powder.*



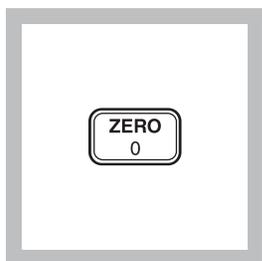
**7.** Press:  
**TIMER ENTER**  
A 5-minute reaction period will begin.  
*Note: Allow the ampul to stand undisturbed.*



**8.** After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## SULFATE, continued

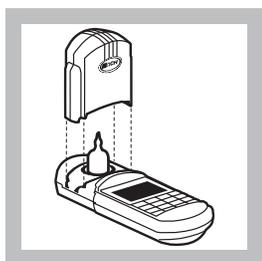
---



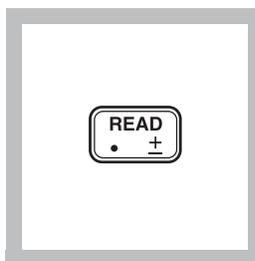
**9. Press: ZERO**

The cursor will move to the right, then the display will show:

**0 mg/L SO<sub>4</sub>**



**10.** Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



**11. Press: READ**

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

*Note: If Program 92 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

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### User- Entered Calibration

There are various programs to determine sulfate, each with a different level of accuracy. Best results are obtained by performing a user-entered calibration with each new lot of reagent. Programs 91 and 92 can be run when a high degree of accuracy is not needed. Use of the Standard Adjust feature will improve performance when using programs 91 and 92. It should NOT be used with a user calibration, as it will hinder performance.

Using Class A glassware, prepare standards of 10, 20, 30, 40, 50, 60, and 70 mg/L sulfate by pipetting 1, 2, 3, 4, 5, 6, and 7 mL of a 1000-mg/L sulfate standard into 100-mL volumetric flasks.

Dilute to the mark with deionized water and mix well.

Zero the instrument with water. The user-entered settings for sulfate are:

Program number: #101 to 105

Wavelength: 520 nm

Resolution: 0 mg/L

See *Creating User-Entered Program* in the instrument manual for specific instructions on entering a user-entered program.

## SULFATE, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples may be stored up to 28 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

### Accuracy Check

#### Standard Additions Method- Powder Pillows

- a) Snap the neck off a Sulfate Standard PourRite Ampule, 1000 mg/L  $\text{SO}_4^{2-}$ .
- b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three 10-mL samples. Mix thoroughly.
- c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Additions Method- AccuVac Ampuls

- a) Snap the neck off a Sulfate Standard PourRite Ampule, 2500 mg/L  $\text{SO}_4^{2-}$ .
- b) Fill three 25- mL graduated cylinders with 25 mL of sample. Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three cylinders. Mix thoroughly. For AccuVac Ampuls, transfer to a 50-mL beaker.
- c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Solution Method

Check the accuracy of the test by using the Sulfate Standard Solution,

50 mg/L, listed under Optional Reagents. Or, prepare this solution by pipetting 1.0 mL of a PourRite Ampule Standard for Sulfate (2500 mg/L) into a 50-mL volumetric flask. Dilute to volume with deionized water. The final concentration is 50 mg/L sulfate. Substitute this standard for the sample and proceed with the test as described in the procedure.

## SULFATE, continued

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### Standard Adjust

Standard adjust is recommended when using stored programs 91 or 92. It **should not** be used with a user-entered calibration.

To adjust the calibration curve using the reading obtained with the 50-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the **STD** setup option. Press **ENTER** to activate the standard adjust option. Then enter **50** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L sulfate.

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 3$  mg/L sulfate.

#### Estimated Detection Limit (EDL)

The EDL for program 91 is 4.9 mg/L  $\text{SO}_4$  and the EDL for program 92 is 3 mg/L  $\text{SO}_4$ . For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

The following interfere at levels above those concentrations listed:

Calcium: 20,000 mg/L as $\text{CaCO}_3$	Magnesium: 10,000 mg/L as $\text{CaCO}_3$
Chloride: 40,000 mg/L as $\text{Cl}^-$	Silica: 500 mg/L as $\text{CaCO}_3$

### Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 Sulfate Reagent to form insoluble barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. The SulfaVer 4 also contains a stabilizing agent to hold the precipitate in suspension.

## SULFATE, continued

### REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required		Cat. No.
	Per Test	Units	
SulfaVer 4 Sulfate Reagent Powder Pillows .....	1 pillow.....	100/pkg .....	21067-69
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg .....	24019-06

### REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls .....	1 ampul.....	25/pkg .....	25090-25
Beaker, 50-mL.....	1 .....	each .....	500-41H

### OPTIONAL REAGENTS

Standard, Drinking Water Inorganics, F <sup>-</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>-3</sup> , SO <sub>4</sub> <sup>-2</sup> .....	500 mL .....	28330-49
Standard, Wastewater Effluent Inorganics, NH <sub>3</sub> <sup>-N</sup> , NO <sub>3</sub> <sup>-N</sup> , PO <sub>4</sub> <sup>-3</sup> , COD, SO <sub>4</sub> <sup>-2</sup> , TOC.....	500 mL .....	28332-49
Sulfate Standard Solution, 50 mg/L .....	500 mL .....	2578-49
Sulfate Standard Solution, 1000 mg/L .....	500 mL .....	21757-49
Sulfate Standard Solution, PourRite Ampule, 2500 mg/L, 10 mL .....	16/pkg .....	14252-10
Sulfate Standard Solution, PourRite Ampule, 1000 mg/L, 2 mL .....	20/pkg .....	21757-20
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each .....	24052-00
Cylinder, graduated mixing, 25 mL .....	each .....	20886-40
Filter Paper, folded, 12.5 cm .....	100/pkg .....	1894-57
Flask, volumetric, 50 mL, Class A.....	each .....	14574-41
Funnel, poly, 65 mm.....	each .....	1083-67
Pipet, TenSette, 0.1 to 1.0 mL.....	each .....	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg .....	21856-96
Pipet, volumetric, 1.00 mL, Class A .....	each .....	14515-35
Pipet, volumetric, 2.00 mL, Class A .....	each .....	14515-36
Pipet, volumetric, 3.00 mL, Class A .....	each .....	14515-03
Pipet, volumetric, 4.00 mL, Class A .....	each .....	14515-04
Pipet, volumetric, 5.00 mL, Class A .....	each .....	14515-37
Pipet, volumetric, 6.00 mL, Class A .....	each .....	14515-06
Pipet, volumetric, 7.00 mL, Class A .....	each .....	14515-07
Pipet Filler, safety bulb .....	each .....	14651-00
PourRite Ampule Breaker .....	each .....	24846-00

### *For Technical Assistance, Price and Ordering*

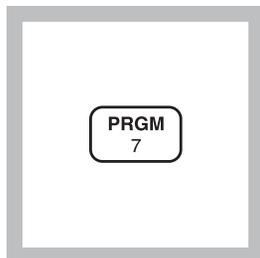
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**SULFIDE (0 to 0.70 mg/L S<sup>2-</sup>)**

For water, wastewater and seawater

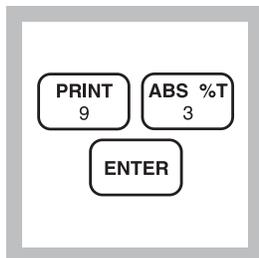
**Methylene Blue Method\* USEPA accepted for reporting wastewater analysis\*\***

1. Enter the stored program number for sulfide (S).

Press: **PRGM**

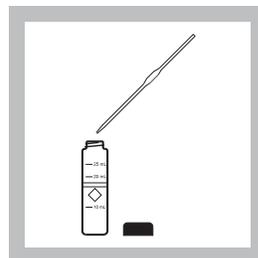
The display will show:

**PRGM ?**



2. Press: **93 ENTER**

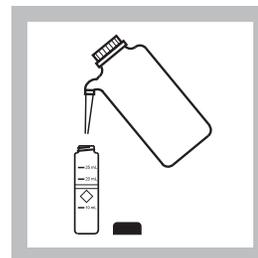
The display will show **mg/L, S** and the **ZERO** icon.



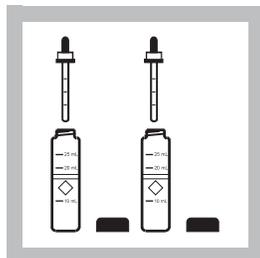
3. Pipet 25 mL of sample into a clean sample cell. This will be the prepared sample.

*Note: Samples must be analyzed immediately and cannot be preserved for later analysis. Use a pipet to avoid agitation.*

*Note: For field testing, a 25-mL graduated cylinder may be used.*

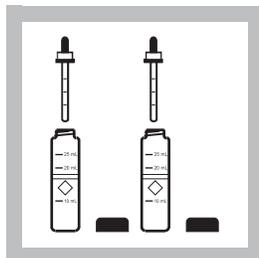


4. Fill a second sample cell with 25 mL of deionized water (the blank).



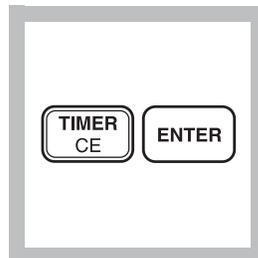
5. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

*Note: Use the calibrated 1-mL dropper.*



6. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

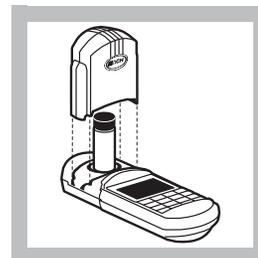
*Note: A pink color will develop, then the solution will turn blue if sulfide is present.*



7. Press:

**TIMER ENTER**

A 5-minute reaction period will begin.



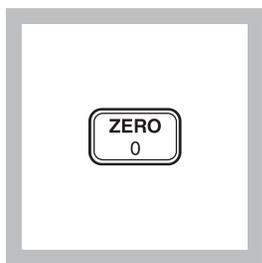
8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

\*\* Procedure is equivalent to USEPA method 376.2 or Standard Method 4500-S<sup>2-</sup>-D for wastewater.

## SULFIDE, continued

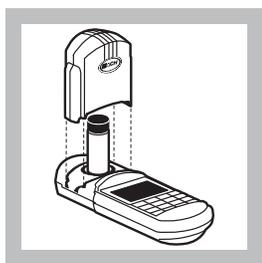
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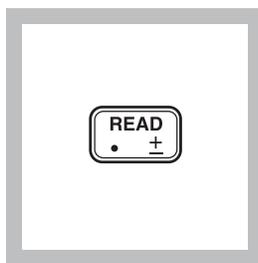
**9. Press: ZERO**

The cursor will move to the right, then the display will show:

**0.00 mg/L S**



**10.** After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**11. Press: READ**

The cursor will move to the right, then the result in mg/L sulfide will be displayed.

*Note: Some sulfide loss may occur if dilution is necessary.*

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

### Accuracy Check

Sulfide standards are unstable and must be user prepared. See Standard Methods, 4500S<sup>-</sup> for preparation and standardization directions.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 0.73 mg/L sulfide and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L sulfide.

#### Estimated Detection Limit (EDL)

The EDL for program 93 is 0.01 mg/L S<sup>2-</sup>. For more information on derivation and use of Hach's estimated detection limit, see Section 1.

## SULFIDE, continued

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### Interferences

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interfere by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. <b>1.</b> Measure 25 mL of sample into a 50-mL erlenmeyer flask. <b>2.</b> Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears. <b>3.</b> Add Phenol Solution dropwise until the yellow color just disappears. Use this solution in Step 4 in place of deionized water.

### Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

### Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N, N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after dilution.

### Pollution Prevention and Waste Management

Sulfide 2 Reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. See *Section 3* for more information on proper disposal of these materials.

## SULFIDE, continued

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### REQUIRED REAGENTS

Sulfide Reagent Set (100 tests).....	Cat. No.
Includes: (2) 1816-42, (2) 1817-42	22445-00

Description	Quantity Required		Units	Cat. No.
	Per Test			
Sulfide 1 Reagent.....	2 mL.....	100 mL	MDB.....	1816-32
Sulfide 2 Reagent.....	2 mL.....	100 mL	MDB.....	1817-32
Water, deionized.....	25 mL.....	4 L	.....	272-56

### REQUIRED APPARATUS

Cylinder, graduated, 25 mL .....	1 .....	each.....	508-40
Pipet, volumetric, Class A, 25.00 mL.....	1 .....	each.....	14515-40
Pipet Filler, safety bulb .....	1 .....	each.....	14651-00
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg.....	24019-06

### OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L.....	29 mL.....	2211-20
Phenol Solution, 30 g/L .....	29 mL.....	2112-20
Sodium Sulfide, hydrate .....	114 g.....	785-14

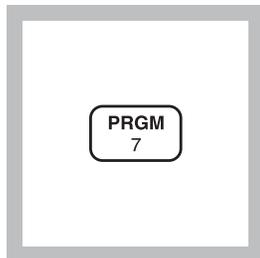
### OPTIONAL APPARATUS

Bottle, Wash, 250 mL .....	each.....	620-31
Dropper, for 1 oz. bottle.....	each.....	2258-00
Flask, erlenmeyer, 50 mL .....	each.....	505-41
<i>Standard Methods for the Examination of Water and Wastewater</i> .....	each.....	22708-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

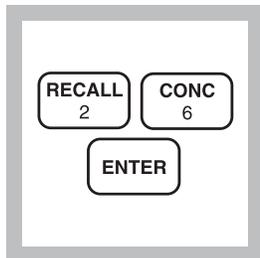
**SURFACTANTS, ANIONIC (0 to 0.300 mg/L) For water, wastewater, and seawater****(Also called: Detergents) Crystal Violet Method\***

1. Enter the stored program number for Surfactants, anionic (LAS).

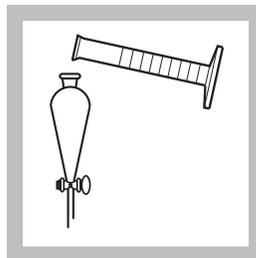
Press: **PRGM**

The display will show:

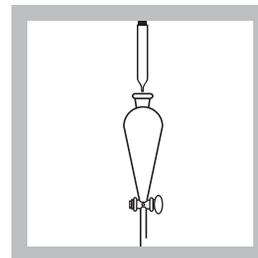
**PRGM ?**



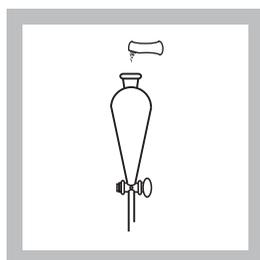
2. Press: **26 ENTER**  
The display will show **mg/L, LAS** and the **ZERO** icon.



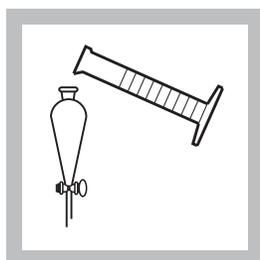
3. Fill a clean 500-mL graduated cylinder to the 300-mL mark with sample. Pour the sample into a clean 500-mL separatory funnel.



4. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.



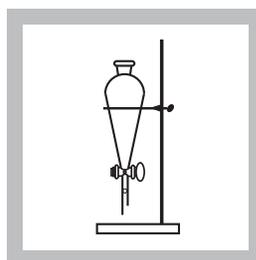
5. Add the contents of one Detergents Reagent Powder Pillow to the funnel. Stopper the funnel and shake to dissolve the powder.



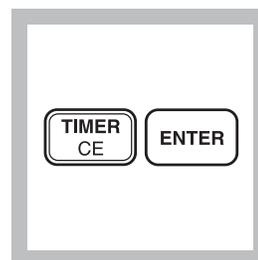
6. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.

*Note: Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.*

*Note: Use benzene only in a well-ventilated area.*



7. Place the separatory funnel in a support stand.



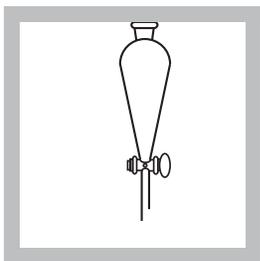
8. Press: **TIMER ENTER**

A 30-minute reaction period will begin.

*Note: Excessive agitation may cause an emulsion, requiring a longer time for phase separation. If this occurs, remove most of the water layer, then gently agitate the funnel with a clean inert object in the funnel such as a Teflon-coated magnetic stirring bar.*

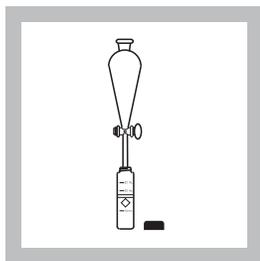
\* *Analytical Chemistry*, 38, 791(1966).

## SURFACTANTS, ANIONIC, continued



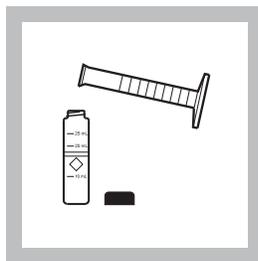
**9.** After the timer beeps, remove the stopper and drain the bottom water layer. Discard this layer.

*Note:* Benzene solutions are a regulated waste and cannot be poured down the drain. See Section 3 for proper disposal of these materials.

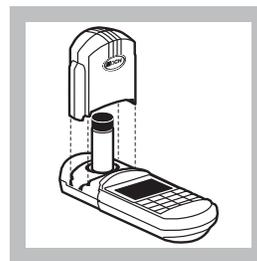


**10.** Drain the top benzene layer into a clean sample cell (the prepared sample).

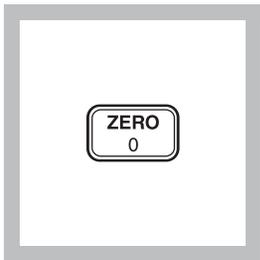
*Note:* The benzene layer cannot be filtered before color measurement. Filtration removes the blue color.



**11.** Fill another sample cell to the 25-mL mark with pure benzene (the blank).



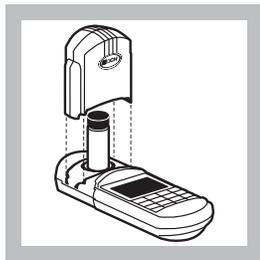
**12.** Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



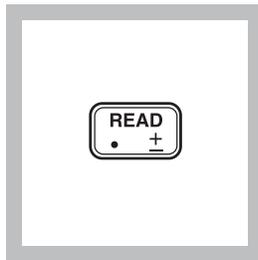
**13.** Press: **ZERO**

The cursor will move to the right, then the display will show:

**0.000 mg/L LAS**



**14.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**15.** Press: **READ**

The cursor will move to the right, then the result in mg/L anionic surfactants (LAS) will be displayed.

*Note:* Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

*Note:* Acetone may be used to clean benzene from glassware.

## SURFACTANTS, ANIONIC, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible, but they may be stored at least 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Detergent Voluette Ampule Standard Solution, 60 mg/L as LAS (The molecular weight of linear alkylate sulfonate used to make the standard is 342).
- b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 300-mL samples. Mix thoroughly.
- c) Analyze each as described above. The anionic surfactants reading should increase 0.02 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 0.150 mg/L LAS, two lots of reagent, and the instrument, a single operator obtained a standard deviation of  $\pm 0.010$  mg/L LAS as anionic surfactant.

#### Estimated Detection Limit

The estimated detection limit for program 26 is 0.020 mg/L LAS. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Perchlorate and periodate ions will interfere. High amounts of chloride, such as those levels found in brines and seawater, will cause low results.

### Summary of Method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene.

## SURFACTANTS, ANIONIC, continued

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### Pollution Prevention and Waste Management

Benzene (D018) solutions are regulated as hazardous waste by Federal RCRA. Do not pour these materials down the drain. Collect water saturated with benzene solutions for disposal with laboratory solvent wastes. See *Section 3* for more information on proper disposal of these materials.

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### REQUIRED REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Benzene, ACS .....	55 mL.....	500 mL.....	14440-49
Buffer Solution, sulfate type .....	10 mL.....	500 mL.....	452-49
Detergent Reagent Powder Pillow .....	1 pillow .....	25/pkg.....	1008-68

### REQUIRED APPARATUS

Clippers, for opening powder pillows.....	1 .....	each.....	968-00
Cylinder, graduated, 25 mL .....	1 .....	each.....	508-40
Cylinder, graduated, 50 mL .....	1 .....	each.....	508-41
Cylinder, graduated, 500 mL .....	1 .....	each.....	508-49
Funnel, separatory, 500 mL .....	1 .....	each.....	520-49
Ring, support, 4 inch.....	1 .....	each.....	580-01
Sample Cell, 10-20-25 mL, w/ cap .....	2.....	6/pkg.....	24019-06
Stand, support, 127 x 203 mm (5 x 8").....	1 .....	each.....	563-00

### OPTIONAL REAGENTS

Acetone, ACS .....	500 mL.....	14429-49
Detergent Standard Solution, Voluette ampule, 60 mg/L as LAS, 10 mL .....	16/pkg.....	14271-10

### OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....	21968-00
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 Pipet .....	1000/pkg.....	21856-28
Thermometer, -20 to 110 °C, Non-Mercury .....	each.....	26357-02

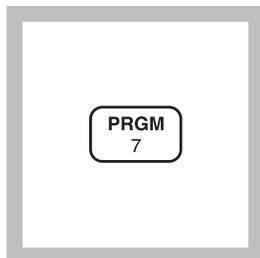
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**SUSPENDED SOLIDS (0 to 750 mg/L)**

For water and wastewater

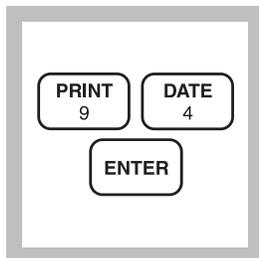
**Photometric Method\* (Also called Nonfilterable Residue)**

1. Enter the stored program number for suspended solids.

Press: **PRGM**

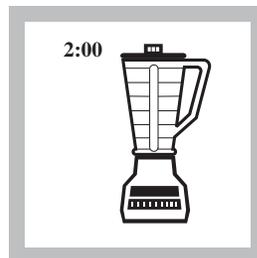
The display will show:

**PRGM ?**

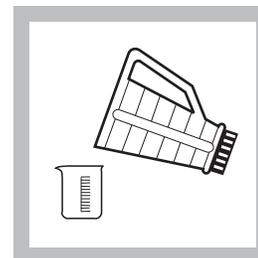


2. Press: **9** **ENTER**

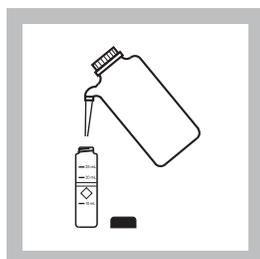
The display will show **mg/L, SuSld** and the **ZERO** icon.



3. Blend 500 mL of sample in a blender at high speed for exactly 2 minutes.

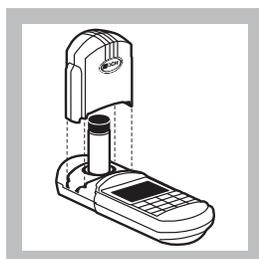


4. Pour the blended sample into a 600-mL beaker.

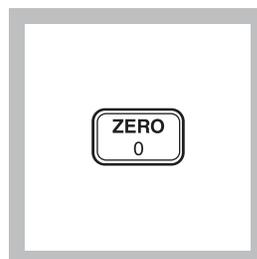


5. Fill a sample cell with 25 mL of tap water or deionized water (the blank).

*Note: Remove gas bubbles in the water by swirling or tapping the bottom of the cell on a table.*



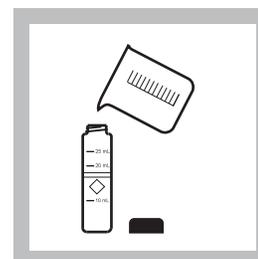
6. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



7. Press: **ZERO**

The cursor will move to the right, then the display will show:

**0 mg/L SuSld**

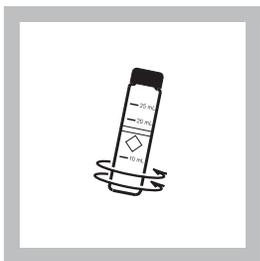


8. Stir the sample thoroughly and immediately pour 25 mL of the blended sample into a sample cell (the prepared sample).

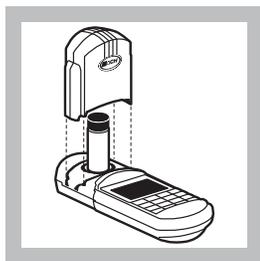
\* Adapted from *Sewage and Industrial Wastes*, 31, 1159 (1959).

## SUSPENDED SOLIDS, continued

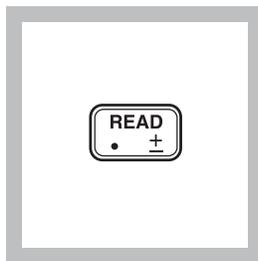
---



**9.** Swirl the prepared sample cell to remove any gas bubbles and uniformly suspend any residue.



**10.** Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



**11.** Press: **READ**  
The cursor will move to the right, then the result in mg/L suspended solids will be displayed.

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. The sample may be stored seven days by cooling to 4 °C (39 °F).

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 847.4 mg/L Suspended Solids with the instrument, a single operator obtained a standard deviation of  $\pm 18.2$  mg/L Suspended Solids.

For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program 94 is 22.1 mg/L Suspended Solids. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Calibration for this test is based on parallel samples using the gravimetric technique on sewage samples from a municipal sewage plant. For most samples, this calibration will provide satisfactory results. When higher accuracy is required, run parallel photometric and gravimetric determinations with portions of the same sample. The new calibration should be made on your particular sample using a gravimetric technique as a basis.

## SUSPENDED SOLIDS, continued

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### Summary of Method

This method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition and weighing steps that gravimetric procedures do. The USEPA specifies the gravimetric method for solids determinations, while this method is often used for checking in-plant processes.

---

### REQUIRED APPARATUS

Description	Quantity Required		Unit	Cat. No.
	Per Test			
Beaker, 600 mL, low form .....	1	.....	each	1080-52
Blender, 1.2 L, 120 V .....	each	.....	each	26161-00
Blender, 1.2 L, 240 V .....	each	.....	each	26161-02
Cylinder, graduated, 500 mL, poly.....	1	.....	each	1081-49
Pipet, serologic, 25 mL .....	1	.....	each	2066-40
Pipet, Filler, safety bulb .....	1	.....	each	14651-00

### OPTIONAL APPARATUS

Stirring Rod, glass .....	3/pkg	.....		1770-01
Wash Bottle, 250 mL.....	each	.....		620-31

### *For Technical Assistance, Price and Ordering*

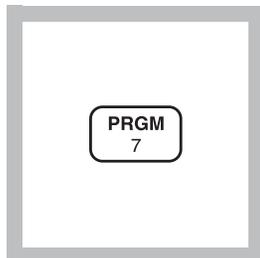
**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**



**TANNIN AND LIGNIN (0 to 9.0 mg/L)**

For water, wastewater, boiler water

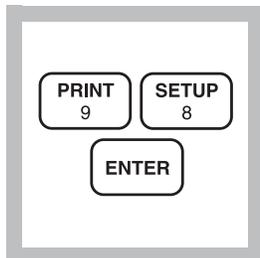
**Tyrosine Method\***

**1.** Enter the stored program number for tannin and lignin.

Press: **PRGM**

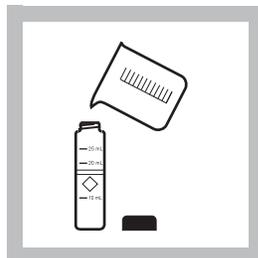
The display will show:

**PRGM ?**

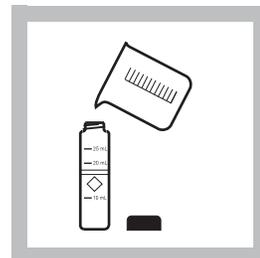


**2.** Press: **98 ENTER**

The display will show **mg/L, tanic** and the **ZERO** icon.

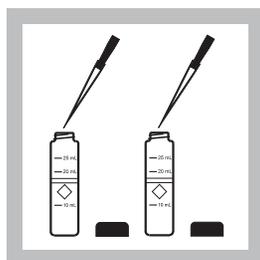


**3.** Fill a clean sample cell to the 25-mL mark with deionized water (the blank).

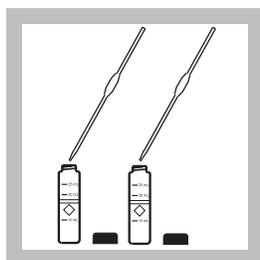


**4.** Fill a clean sample cell to the 25-mL mark with sample (the prepared sample).

*Note: Filter turbid samples and report results as mg/L soluble tannic acid.*

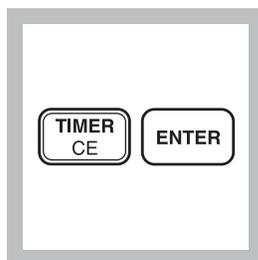


**5.** Pipet 0.5 mL of TanniVer 3 Tannin-Lignin Reagent into each cell. Swirl to mix.



**6.** Pipet 5.0 mL of Sodium Carbonate Solution into each cell. Swirl to mix.

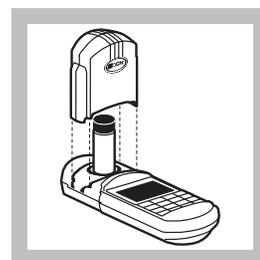
*Note: A blue color will develop if tannins and/or lignins are present.*



**7.** Press:

**TIMER ENTER**

A 25-minute reaction period will begin.

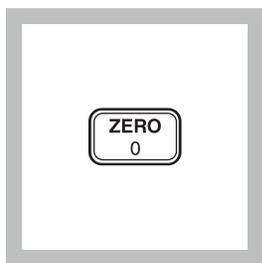


**8.** Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

\* Adapted from Kloster, M.B., *Journal American Water Works Association*, Vol. 66, No. 1, p. 44 (1974).

## TANNIN AND LIGNIN, continued

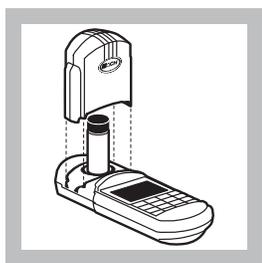
---



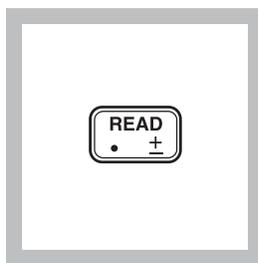
**9. Press: ZERO**

The cursor will move to the right, then the display will show:

**0.0 mg/L tanic**



**10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**



**11. Press: READ**

The cursor will move to the right, then the result in mg/L tannic acid will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

---

### Sampling and Storage

Collect samples in clean plastic or glass bottles.

### Accuracy Check

#### Standard Solution Method

Prepare a 200-mg/L tannic acid standard solution by dissolving 0.200 grams of tannic acid in deionized water and diluting to 1000 mL. Prepare this solution monthly. A 2.0 mg/L tannic acid standard is prepared by diluting 10.00 mL of the stock solution to 1000 mL with deionized water. Prepare this standard daily.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 4.0 mg/L tannic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.1$  mg/L tannic acid.

#### Estimated Detection Limit

The estimated detection limit for program 98 is 0.1 mg/L tannin and lignin. For more information on the estimated detection limit, see *Section 1*.

## TANNIN AND LIGNIN, continued

### Interferences

Substance	Interference Level and Treatment
Ferrous iron	Causes a positive interference. Two mg/L of ferrous iron produces a color equivalent to about 1 mg/L of tannic acid. To eliminate interference of ferrous iron up to 20 mg/L, add one 0.2-g scoop of sodium pyrophosphate to the sample before testing.
Sulfide	Eliminated by adding 1 mL of formaldehyde to the sample before testing the sample.

### Summary of Method

This test measures all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. Report results as total tannin and lignin expressed as mg/L tannic acid.

### REQUIRED REAGENTS

	<b>Cat. No.</b>
Tannin and Lignin Reagent Set (up to 100 tests) .....	22446-00
Includes: (2) 675-49, (1) 2560-42	

Description	Quantity Required		Cat. No
	Per Test	Unit	
Sodium Carbonate Solution .....	10 mL	500 mL	675-49
TanniVer 3 Tannin-Lignin Reagent .....	1 mL	100 mL	2560-42
Water, deionized .....	25 mL	4 L	272-56

### REQUIRED APPARATUS

Pipet Filler, safety bulb .....	1	each	14651-00
Pipet, volumetric, Class A, 5.0 mL .....	1	each	14515-37
Pipet, volumetric, Class A, 0.5 mL .....	1	each	14515-34
Sample Cell, 10-20-25-mL, w/ cap .....	2	6/pkg	24019-06

### OPTIONAL REAGENTS

Formaldehyde.....	100 mL	2059-32
Sodium Pyrophosphate, ACS.....	50 g	14295-25
Tannic Acid .....	113 g	791-14

## TANNIN AND LIGNIN, continued

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### OPTIONAL APPARATUS

Description	Unit	Cat. No
Balance, analytical, 115 V .....	each.....	28014-01
Balance, analytical, 230 V .....	each.....	28014-02
Cylinder, graduated, 25 mL .....	each.....	508-40
Filter Paper, folded, 12.5 cm.....	100/pkg.....	1894-57
Flask, volumetric, 1000 mL.....	each.....	14547-53
Funnel, poly, 65 mm .....	each.....	1083-67
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 Pipet .....	50/pkg.....	21856-96
Pipet Tips, for 19700-01 Pipet .....	1000/pkg.....	21856-28
Pipet, volumetric, Class A, 10.00 mL.....	each.....	14515-38
Pipet, Filler, safety bulb .....	each.....	14651-00
Spoon, measuring, 0.2 g.....	each.....	638-00
Weighing Boat, 67/47 mm.....	500/pkg.....	21790-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

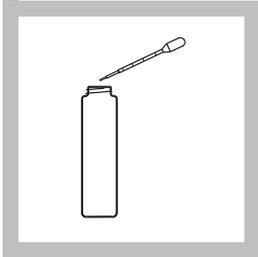
Outside the U.S.A.—Contact the Hach office or distributor serving you.

# TOXTRAK™ TOXICITY TEST\*

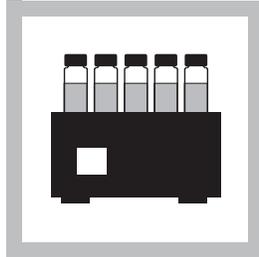
Method 10017  
For water and wastewater

## Colorimetric Method\*\*

### Inoculum Development

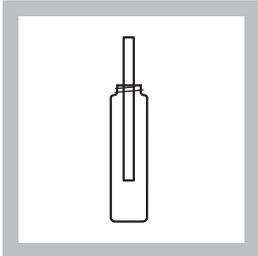


**Using Indigenous Biomass**  
**1.** Using one of the dropper pipets provided, add 1.0 mL of source culture to a Tryptic Soy Broth Tube.

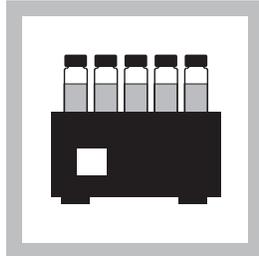


**2.** Incubate at 37 °C until the vial contents are visibly turbid (turbidity indicates bacterial growth).

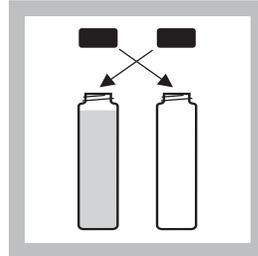
### Inoculum Development Using Aqua QC-Stiks



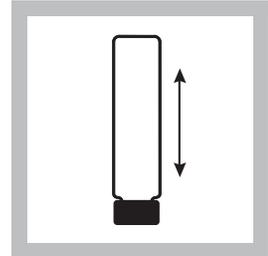
**1.** Inoculate a Lauryl Tryptose broth tube with an *E. coli* Aqua QC-Stik™ according to the instructions that come with the stick.



**2.** Incubate the Lauryl Tryptose Broth Tube at 35°C (95°F) until the medium is visibly turbid (approximately 12 hours). Turbidity develops much faster in an incubator than at room temperature.



**3.** Inoculate a new Lauryl Tryptose Broth Tube by first inverting the tube and switching the caps of the two tubes. In this way, several medium vials can be inoculated from one Aqua-QC Stick™.



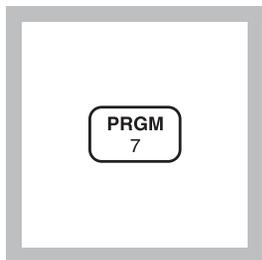
**4.** Invert the new tube. After incubation, this new vial may be used in subsequent tests. If toxicity tests will be run on consecutive days, inoculum may be kept several days at room temperature. Cultures 10 to 72 hours old give best results.

\* U.S. Patent number 5,413,916.

\*\* Liu, D., *Bull. Environ. Contam. Toxicol.* 26, 145-149 (1981).

## TOXTRAK TOXICITY TEST, continued

### Colorimetric Reaction

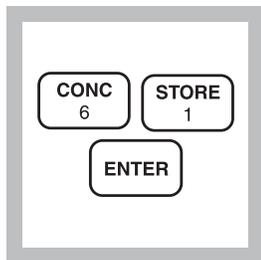


**1.** Enter the stored program number for toxicity.

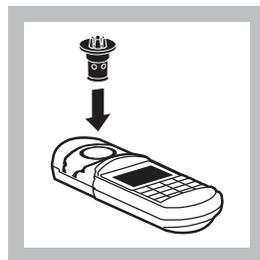
Press: **PRGM**

The display will show:

**PRGM ?**

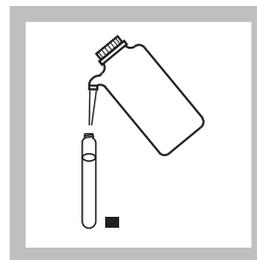


**2.** Press: **61 ENTER**  
The display will show:  
**ABS 610 nm**  
and the zero icon.

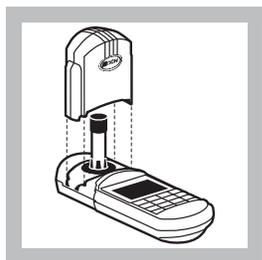


**3.** Insert the TNT/COD Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

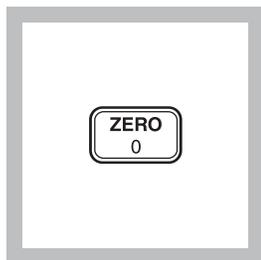
*Note: A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



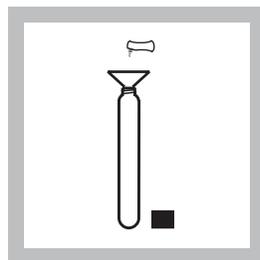
**4.** Fill a Test 'N Tube vial with deionized water. Label this vial as the "blank". Wipe the outside of all the vials with a tissue to remove fingerprints and smudges.



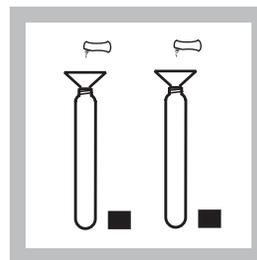
**5.** Place the blank in the adapter. Tightly cover the vial with the instrument cap.



**6.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.000 ABS**

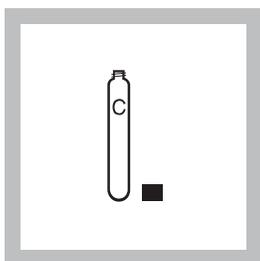


**7.** Label a vial "control." Open one ToxTrak Reagent Powder Pillow and add the contents to the empty reaction vial.

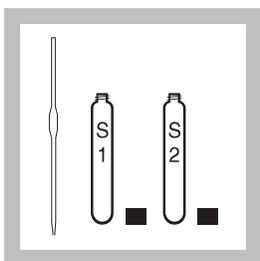


**8.** Label each sample or dilution vial clearly. Add the contents of one ToxTrak Reagent Powder Pillow to each labeled vial.

## TOXTRAK TOXICITY TEST, continued

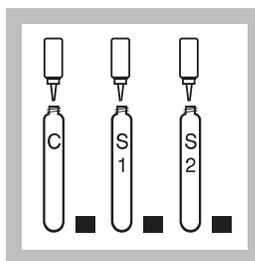


**9.** Add 5.0 mL of deionized water to the control tube.

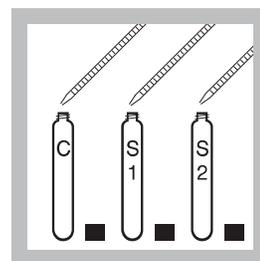


**10.** Add 5.0 mL of the sample (or dilution) to each sample vial.

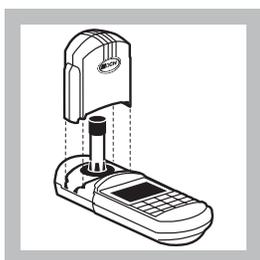
*Note: To determine the approximate threshold level of toxicity for a sample, dilute 1 mL of sample to 10 mL of deionized water and run the test. Continue to make serial 1:10 dilutions until a level is reached which gives a 0% Inhibition in Step 18.*



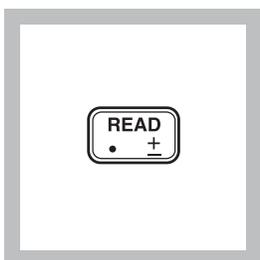
**11.** Add 2 drops of Accelerator Solution to each vial. Cap and invert to mix.



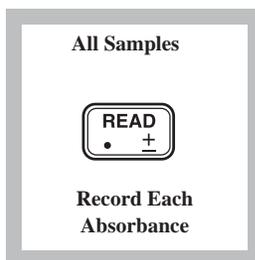
**12.** Add 0.5 mL of inoculum (previously prepared) to each vial. Cap and invert to mix.



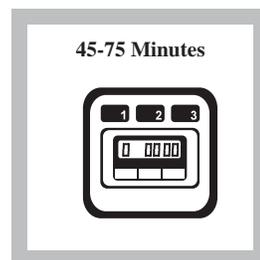
**13.** Place the control vial in the cell holder. Tightly cover the vial with the instrument cap.



**14.** Press: **READ**  
The cursor will move to the right, then the result in ABS will be displayed. Record the absorbance of the “control” vial.

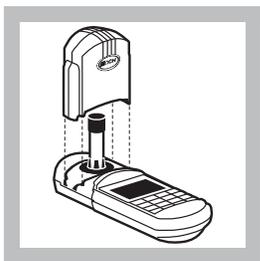


**15.** Repeat Steps 13-14 for all samples and dilutions. Be sure to record each absorbance.

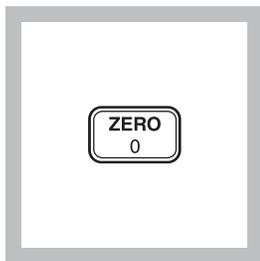


**16.** Allow the solutions in the tubes to react until the absorbance of the **control tube** decreases  $0.60 \pm 0.10$ . This should take about 45-75 minutes.

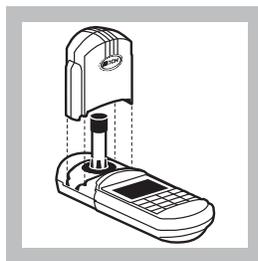
## TOXTRAK TOXICITY TEST, continued



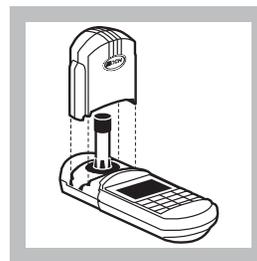
**17.** After the absorbance of the “control” vial has decreased  $0.60 \pm 0.10$  absorbance units, place the blank in the adapter. Tightly cover the vial with the instrument cap.



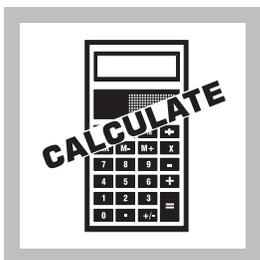
**18.** Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.000 ABS**



**19.** Place the “control” vial in the cell holder. Tightly cover the vial with the instrument cap. Record the absorbance value of the control.



**20.** Place each sample or dilution vial in the cell holder. Tightly cover the vial with the instrument cap. Record each absorbance value.



**21.** Calculate the % Inhibition as follows:

$$\%I = \left[ 1 - \left( \frac{\Delta\text{Abs sample}}{\Delta\text{Abs control}} \right) \right] \times 100$$
  
 $\Delta A$  is the initial absorbance value minus the final absorbance value.

See the example following this step.

*Note: Some toxins increase respiration and will give a negative % inhibition on all respiration-based toxicity tests. After repeated testing, samples which always give a % inhibition in Step 21 that is more negative than -10% should be considered toxic.*

### Example

The control tube (C) has an initial absorbance of 1.6 and decreases to 1.0 Abs. The sample tube has an initial absorbance of 1.7 and decreases to 1.3 Abs.

$$\Delta\text{Abs. Sample} = 1.7 - 1.3 = 0.4 \qquad \Delta\text{Abs. Control} = 1.6 - 1.0 = 0.6$$

$$\%I = \left( 1 - \left( \frac{0.4}{0.6} \right) \right) \times 100 \qquad \%I = 33.3$$

## TOXTRAK TOXICITY TEST, continued

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### Interpreting Results

The Percent Inhibition results obtained are only a relative measurement. They do not represent a true quantitative measurement of toxic concentration. The Percent Inhibition does not necessarily increase in direct proportion to the concentration of toxins. To determine the minimum inhibition concentration of a toxin, it is possible to make tenfold dilutions of the sample and determine the Percent Inhibition for the dilutions. When the sample is diluted so that no inhibition is observed, this is the No Observed Effect Concentration (NOEC).

Due to the many variables involved in the test, the limits of detection are on the order of 10% Inhibition. This would correlate to the Lowest Observable Effect Concentration (LOEC). If a sample shows less than 10% Inhibition, repeat the test. After several repetitions, look at the series of data to determine the likelihood of toxicity. Results below 10% are not reliable, but can be used to surmise some presence of toxicity if they are consistent. See the table below.

**Toxicity Results**

<b>Data Points: Percent Inhibition</b>	<b>Conclusion</b>
7%, 9%, 5%, 8%, 5%	May be slightly toxic
7%, -4%, 5%, 5%, 1%	Most likely not toxic
-7%, -9%, 5%, -8%, -5%	May be slightly toxic

Some toxins will increase respiration and will give a negative Percent Inhibition on this and all other respiration-based toxicity tests. After repeated testing, samples that always give a Percent Inhibition that is more negative than -10% should be considered toxic.

### Disposal of Test Cultures

Dispose of active bacterial cultures by using one of these methods:

1. Autoclave used test containers at 121 °C for 15 minutes at 15 pounds of pressure. Once the containers are sterile, pour the contents down the drain with running water. The reaction tubes may be washed and re-used.
2. Sterilize test containers by using a 1:10 dilution of commercial

## TOXTRAK TOXICITY TEST, continued

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laundry bleach. Pour the test container contents and test containers into the bleach solution. Allow 10-15 minutes of contact time with the bleach solution. Then pour the liquid down the drain and wash the reaction tubes for re-use.

### Summary of Method

Resazurin is a redox-active dye, which changes from pink to blue when it is reduced. Bacterial respiration occurring in the sample reduces resazurin. If toxic substances are present, they inhibit the rate of resazurin reduction. The sample color is compared to a toxin-free control tube to determine how toxic the sample is to an indigenous culture or a culture of *E. coli*. A chemical accelerant reduces the reaction time of the procedure.

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### REQUIRED REAGENTS

Description	Cat. No.
ToxTrak Reagent Set (25 tests).....	25972-00
Includes: (1) 25607-66, (1) 25608-36, (1) 22777-00, (1) 24092-32	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Aqua QC–Sticks, Escherichia Coli.....	1	3 cultures	27063-03
Sodium Thiosulfate Standard Solution .....	varies	100 mL	24092-32
ToxTrak Reagent Powder Pillows .....	1 pillow	50/pkg	25607-66
ToxTrak Accelerator Solution.....	2 drops	15 mL SCDB	25608-36
Tryptic Soy Broth Tubes.....	1	15/pkg	22777-00
Tube, culture, with cap.....	1	10/pkg	20962-08
Water, deionized.....	varies	200 mL	272-29

### REQUIRED APPARATUS

Cap, White .....	1	6/pkg	22411-06
Clippers, to open powder pillows .....	1	each	936-00
COD/TNT Adapter .....	1	each	48464-00
Dropper Pipet, 1 mL .....	varies	10/pkg	21247-20
Forceps, flat square tip.....	1	each	14537-00
Pipet, volumetric, 5.00 mL, Class A.....	1	each	14515-37
Pipet Filler, Safety Bulb.....	1	each	14651-00
Vial, Test ‘N Tube.....	1	6/pkg	25831-25

## TOXTRAK TOXICITY TEST, continued

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### OPTIONAL APPARATUS

Description	Unit	Cat. No.
Burner, Alcohol, 60 mL .....	each .....	20877-42
Burner, Bunsen .....	each .....	21627-00
Germicidal Cloth .....	50/pkg .....	24632-00
Incubator, Dri Bath, 25 well, 120/230 V .....	each .....	45900-00
Incubator, Dri Bath, 25 well, 120/230 V, with European power cord .....	each .....	45900-02
Pipet, Sterile Transfer .....	15/pkg .....	22325-12
Timer .....	each .....	26305-00

### *For Technical Assistance, Price and Ordering*

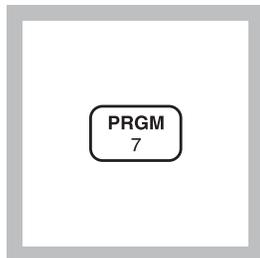
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**TURBIDITY (0 to 1000 FAU)**

For water, wastewater, and seawater

**Absorptometric Method\***

1. Enter the stored program number for turbidity.

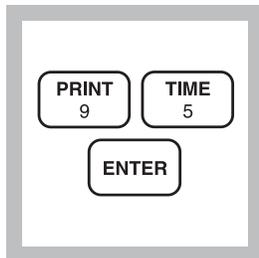
Press: **PRGM**

The display will show:

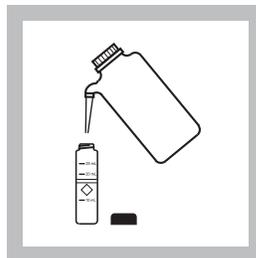
**PRGM ?**

*Note:*

*1 FAU=1 NTU=1 FTU when measuring formazin. These are not equivalent when measuring other types of standards or samples.*



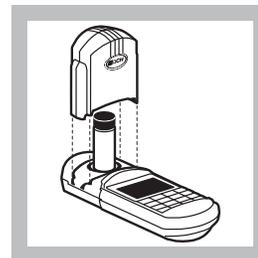
2. Press: **95 ENTER**  
The display will show **FAU** and the **ZERO** icon.



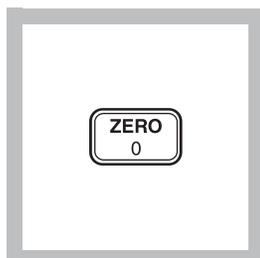
3. Fill a sample cell with 10 mL of deionized water (the blank).

*Note: Wipe the surface of the cell with a soft cloth.*

*Note: For highly colored samples, use a filtered portion of sample in place of the deionized water.*

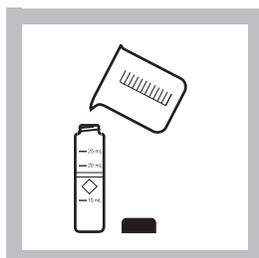


4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



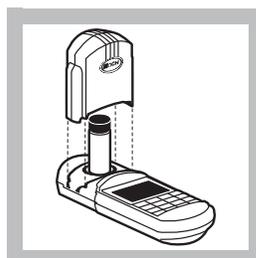
5. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0 FAU**

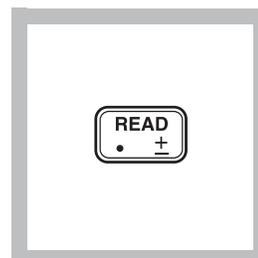


6. Fill another sample cell with 10 mL of sample.  
*Note: Mix the sample well before transferring it to the sample cell.*

*Note: Wipe the surface of the cell with a soft cloth.*



7. Place the sample cell into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **READ**  
The cursor will move to the right, then the result in Formazin Attenuation Units (FAU) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section I).*

\* Adapted from FWPCA *Methods for Chemical Analysis of Water and Wastes*, 275 (1969)

## TURBIDITY, continued

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### Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible. Store samples up to 48 hours by cooling to 4 °C (39 °F). Analyze the sample at the same temperature as it was collected.

### Accuracy Check

#### Standard Solution Method

The stored program has been calibrated using formazin, the primary standard for turbidity. A 200 FAU formazin solution for checking the accuracy of the test can be prepared using the following procedure.

1. Pipet 5.00 mL of a 4000 NTU Formazin stock solution into a 100-mL volumetric flask.
2. Dilute to the mark with deionized water. Prepare this daily.

Convenient stabilized turbidity stock solution (200 NTU StablCal™ Standard) is available from Hach.

#### Standard Adjust

To adjust the calibration curve using the reading obtained with the 200 FAU formazin standard, press the **SETUP** key and scroll (using the arrow keys) to the **STD** setup option. Press **ENTER** to activate the standard adjust option. Then enter **200** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

### Method Precision

#### Precision

In a single laboratory, using a turbidity standard solution of 200 FAU with the instrument, a single operator obtained a standard deviation of  $\pm 2$  FAU.

#### Estimated Detection Limit

The estimated detection limit for program 95 is 21 FAU. For more information on the estimated detection limit, see *Section 1*.

## TURBIDITY, continued

### Interferences

Interfering Substance	Interference Levels and Treatments
Air Bubbles	Interfere at all levels. Degass samples using the Degassing Kit or an ultrasonic bath.
Color	Interferes if the color absorbs light at 520 nm.
Temperature extremes	May interfere by changing the turbidity of the sample. Analyze samples as soon as possible after collection. Analyze at the same temperature as the original sample.

### Summary of Method

This turbidity test measures an optical property of the sample which results from scattering and absorption of light by particles in the sample. The amount of turbidity measured depends on variables such as the size, shape, color, and refractive properties of the particles.

This procedure is calibrated using formazin turbidity standards and the readings are in terms of Formazin Attenuation Units (FAU). This test cannot be used for USEPA reporting purposes, but it may be used for daily in-plant monitoring. One FAU is equivalent to one Nephelometric Turbidity Unit (NTU) of Formazin. However, the optical method of measurement for FAU is very different than the NTU method (1 NTU = 1 FTU = 1 FAU when traced to formazin primary standards.)

### REQUIRED APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06

### REQUIRED REAGENTS

Description	Units	Cat. No.
Formazin Stock Solution, 4000 NTU .....	500 mL .....	2461-49
Silicone Oil.....	15 mL DB .....	1269-36
StablCal Stabilized Turbidity Standard, 200 NTU .....	500 mL .....	26604-49
Water, deionized .....	4 L .....	272-56

## TURBIDITY, continued

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### OPTIONAL APPARATUS

Description	Units	Cat. No.
Bath, ultrasonic .....	each.....	24895-00
Bottle, wash, 250 mL.....	each.....	620-31
Flask, volumetric, Class A, 100 mL .....	each.....	14574-42
Flask, filter, 500 mL.....	each.....	546-49
Filter Holder.....	each.....	13529-00
Filter Pump, aspirator .....	each.....	2131-00
Oiling cloth, for applying silicone oil.....	each.....	26873-00
Pipet Filler, safety bulb .....	each.....	14651-00
Pipet, volumetric, Class A, 5.0 mL.....	each.....	14515-37
Sample Degassing Kit.....	each.....	43975-00
Stopper, rubber, one-hole, No. 7 .....	6/pkg.....	2119-07
Tubing, rubber, 5/16" I.D.....	12 feet.....	560-19
Tweezers, plastic .....	each.....	14282-00

### *For Technical Assistance, Price and Ordering*

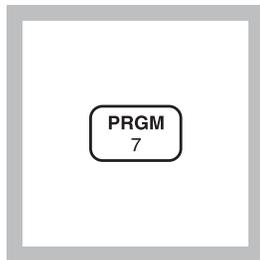
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# VOLATILE ACIDS (0 to 2800 as mg/L HOAc)

Method 8196  
For digester sludges

## Esterification Method\*



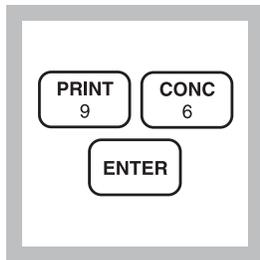
1. Enter the stored program number for Volatile Acids as acetic acid (HOAc).

Press: **PRGM**

The display will show:

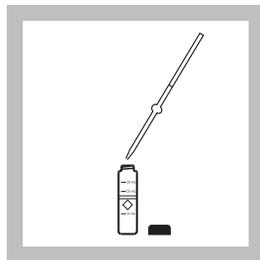
**PRGM ?**

*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **96 ENTER**  
The display will show **mg/L, HOAc** and the **ZERO** icon.

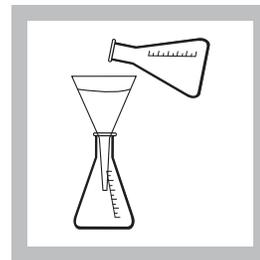
*Note: If high levels of dissolved solids or mineral acids are present, distill as described in the Hach Distillation Apparatus manual.*



3. Pipet 0.5 mL of deionized water into a dry 25-mL sample cell (the blank).

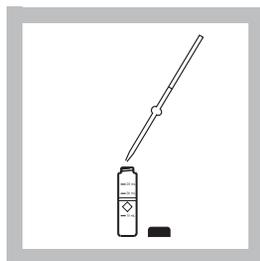
*Note: Use a Class A or TenSette Pipet.*

*Note: Adjust the pH of stored samples before analysis.*



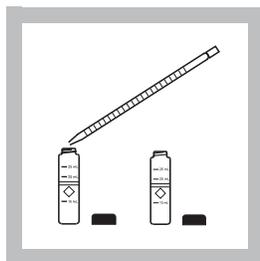
4. Filter or centrifuge 25 mL of the sample.

*Note: Centrifugation is faster than filtration.*

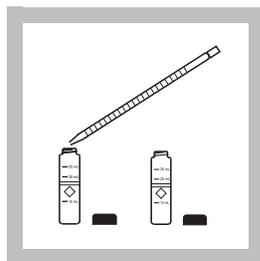


5. Pipet 0.5 mL of the filtrate or supernatant into another dry 25-mL sample cell (the prepared sample).

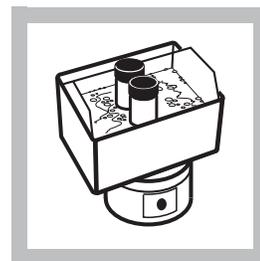
*Note: Use a Class A or TenSette Pipet.*



6. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



7. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.



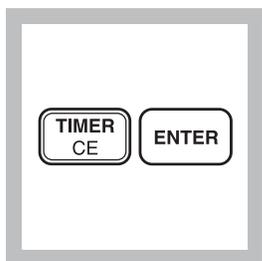
8. Place both cells into a boiling water bath.

*Note: Samples may be boiled in a 600-mL beaker.*

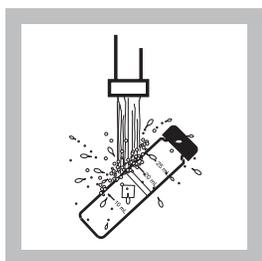


\* Adapted from *The Analyst*, 87, 949 (1962)

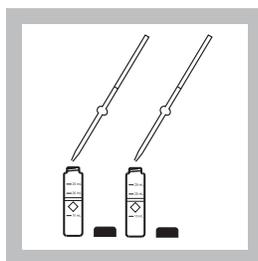
## VOLATILE ACIDS, continued



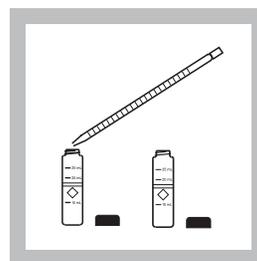
**9.** Press: **TIMER**  
**ENTER**  
A 3-minute reaction period will begin.



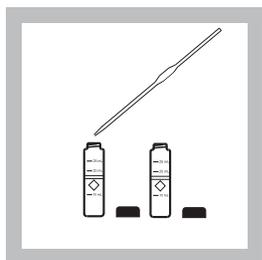
**10.** When the timer beeps, cool solutions to 25 °C (until cells feel cool) with running tap water. Then dry the cells with a soft cloth.



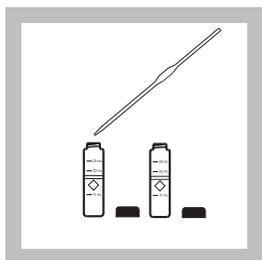
**11.** Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



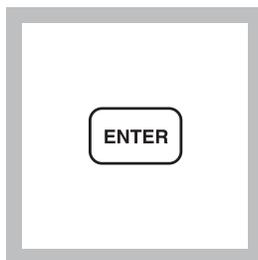
**12.** Pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Cap and invert to mix.



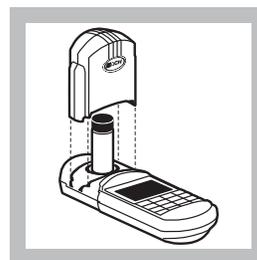
**13.** Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Cap and invert to mix.



**14.** Add 10 mL of deionized water to each cell. Cap and invert to mix.



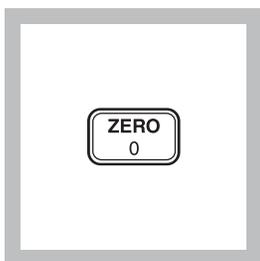
**15.** The display will show: **3:00 TIMER 2**  
Press: **ENTER**  
A 3-minute reaction period will begin.  
*Note: After this three-minute reaction period, proceed immediately through steps 16-19.*



**16.** When the timer beeps, immediately place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

## VOLATILE ACIDS, continued

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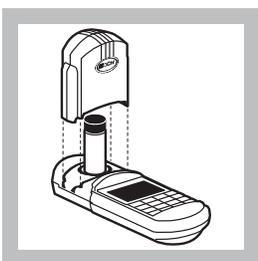


**17. Press: ZERO**

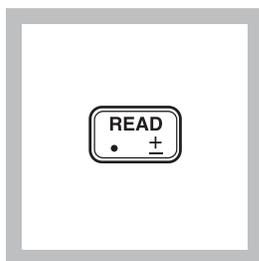
The cursor will move to the right, then the display will show:

**0 mg/L HOAc**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



**18. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.**



**19. Press: READ**

The cursor will move to the right, then the result in mg/L Volatile Acids as acetic acid will be displayed.

---

### Sampling and Storage

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before testing.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Volatile Acids PourRite Ampule Standard Solution, 62,500 mg/L as acetic acid.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL graduated mixing cylinders, each containing 25 mL of filtered sample. Stopper. Shake well to mix.
- c) Remove a 0.5 mL aliquot of sample from each cylinder; add to three dry sample cells. Analyze all three samples along with the original test sample beginning with Step 5 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions in Section 1*.

## VOLATILE ACIDS, continued

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### Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids PourRite Ampule Standard Solution (62,500 mg/L as acetic acid) to a 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 500 mg/L volatile acids as acetic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 8$  mg/L.

#### Estimated Detection Limit

The estimated detection limit for program 96 is 17 mg/L HOAc. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The volatile acids test is designed specifically for the determination of volatile acids in digester sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

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## REQUIRED REAGENTS

	Cat. No.
Volatile Acids Reagent Set (90 tests).....	22447-00
Includes: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32	

Description	Quantity Required		Cat. No.
	Per Test	Units	
Ethylene Glycol .....	3 mL.....	1000 mL.....	2039-53
Ferric Chloride-Sulfuric Acid Solution .....	20 mL.....	1000 mL.....	2042-53
Hydroxylamine Hydrochloride Solution, 100 g/L.....	1 mL.....	100 mL.....	818-42
Sodium Hydroxide Standard Solution, 4.5 N .....	4 mL.....	1000 mL.....	2040-53
Sulfuric Acid Standard Solution, 19.2 N .....	0.4 mL.....	100 mL.....	2038-32
Water, deionized.....	20.5 mL.....	4 L.....	272-56

## VOLATILE ACIDS, continued

---

### REQUIRED APPARATUS

Description	Quantity Required		Cat. No.
	Per Test	Units	
Cots, finger .....	2 .....	2/pkg .....	14647-02
Cylinder, graduated, 10 mL.....	1 .....	each .....	508-38
Filter Paper, folded, 12.5 cm .....	1 .....	100/pkg .....	1894-57
Flask, erlenmeyer, 50 mL.....	1 .....	each .....	505-41
Funnel, poly, 65 mm.....	1 .....	each .....	1083-67
Hot Plate, circular, 3.5-inch diameter.....	1 .....	each .....	12067-01
Pipet Filler, safety bulb .....	1 .....	each .....	14651-00
Pipet, serological, 2 mL.....	2 .....	each .....	532-36
Pipet, volumetric, Class A, 0.5 mL .....	3 .....	each .....	14515-34
Pipet, volumetric, Class A, 10.00 mL .....	3 .....	each .....	14515-38
Sample Cell, 10-20-25 mL, w/cap .....	2 .....	6/pkg .....	24019-06
Water Bath and Rack.....	1 .....	each .....	1955-55

### OPTIONAL REAGENTS

Volatile Acids Standard Solution, PourRite ampule, 62,500 mg/L as acetic acid, 10 mL .....	16/pkg .....	14270-10
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### OPTIONAL APPARATUS

Ampule Breaker, PourRite .....	each .....	24846-00
Beaker, 600 mL .....	each .....	500-52
Bottle, wash, 500 mL .....	each .....	620-11
Centrifuge, laboratory, 115 Vac.....	each .....	26765-00
Centrifuge, laboratory, 230 Vac.....	each .....	26765-02
Centrifuge Tubes, 15 mL.....	10/pkg .....	22787-39
Centrifuge Tube Caps.....	20/pkg .....	25852-20
Cylinder, graduated, mixing, 25 mL .....	each .....	1896-40
Cylinder, graduated, plastic, 250 mL .....	each .....	1081-46
Distillation Apparatus .....	each .....	22653-00
Distillation Heater and Support Apparatus .....	each .....	22744-00
Flask, volumetric, Class A, 100 mL.....	each .....	14574-42
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, TenSette, 1.0 to 10.0 mL .....	each .....	19700-10
Pipet Tips, for 19700-10.....	50/pkg .....	21997-96

### *For Technical Assistance, Price and Ordering*

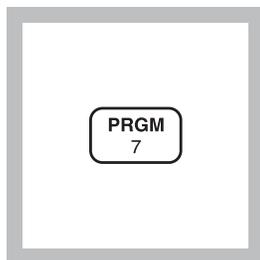
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**ZINC (0 to 3.00 mg/L Zn)**

For water and wastewater

**Zincon Method\*** USEPA approved for wastewater analysis\*\* (digestion needed; see Section 2)

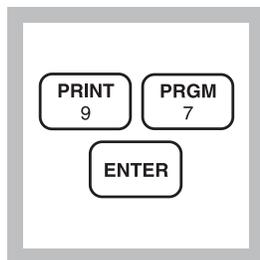
**1.** Enter the stored program number for zinc (Zn).

Press: **PRGM**

The display will show:

**PRGM ?**

*Note:* For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).

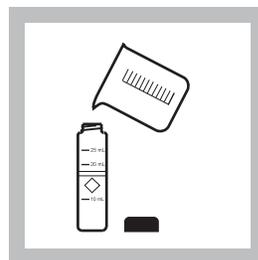


**2.** Press: **97 ENTER**

The display will show **mg/L, Zn** and the **ZERO** icon.

*Note:* Total zinc requires a prior digestion; use either the Digesdahl or mild digestion (Section 2).

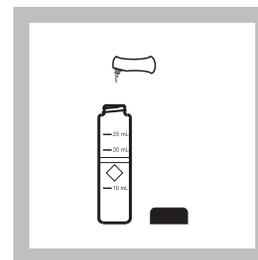
*Note:* Adjust the sample to pH 4-5; see Sampling and Storage following these steps.



**3.** Fill a 25-mL sample cell with 20 mL of sample.

*Note:* Rinse glassware with 1:1 hydrochloric acid and deionized water before use.

*Note:* If samples cannot be analyzed immediately, see Sampling and Storage.



**4.** Add the contents of one ZincoVer 5 Reagent Powder Pillow. Cap. Invert several times to completely dissolve the powder. If the sample does not turn orange, see the note below.

*Note:* Powder must be completely dissolved or inconsistent results may occur.

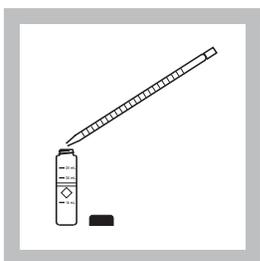
*Note:* The sample should be orange. If it is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high or an interference is present.

**Caution: ZincoVer 5 contains cyanide and is very poisonous if taken internally or inhaled. Do not add to an acidic sample. Store away from water and acids.**

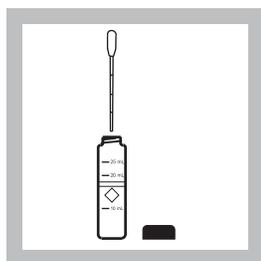
\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

\*\* *Federal Register*, 45 (105) 36166 (May 29, 1980).

## ZINC, continued

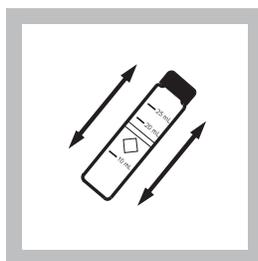


5. Measure 10 mL of the orange solution into another sample cell (the blank).



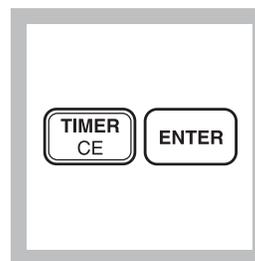
6. Add 0.5 mL of cyclohexanone to the remaining orange solution in the first sample cell (the prepared sample).

*Note: Use a plastic squeezer. Rubber bulbs may contaminate the cyclohexanone.*



7. Tightly cap the cell. Shake vigorously for 30 seconds (the prepared sample).

*Note: The sample will be red-orange, brown or blue, depending on the zinc concentration.*

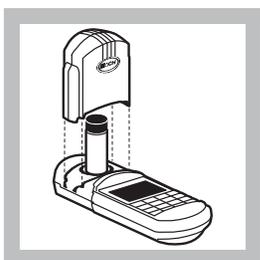


8. Press:

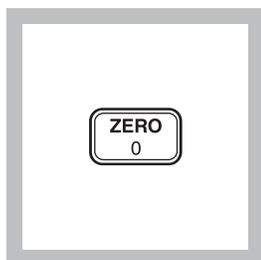
**TIMER ENTER**

A 3-minute reaction period will begin.

*Note: Steps 9-11 must be completed within 10 minutes after the timer beeps.*



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

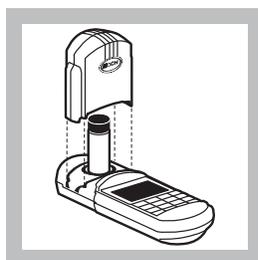


10. Press: **ZERO**

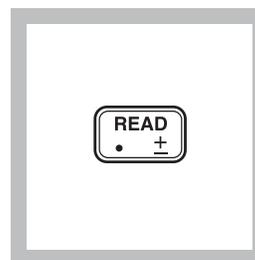
The cursor will move to the right, then the display will show:

**0.00 mg/L Zn**

*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L Zn will be displayed.

*Note: Standard Adjust may be performed using a prepared 0.50 mg/L standard. See Section 1.*

## ZINC, continued

---

### Sampling and Storage

Collect samples in acid-washed plastic bottles. For storage, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). The preserved samples can be stored up to six months at room temperature.

Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as zinc may be lost as a precipitate. Correct the test result for volume additions; see *Sampling and Storage, Volume Additions*, in *Section 1* for more information.

If only dissolved zinc is to be determined, filter the sample before the acid addition.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a Zinc PourRite Ampule Standard, 25 mg/L Zn.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.
- c) Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

#### Standard Solution Method

Prepare a 0.50 mg/L zinc standard solution by diluting 5.00 mL of Zinc Standard Solution, 100 mg/L as Zn, to 1000 mL with deionized water in a Class A 1000-mL volumetric flask. Prepare this solution daily. Use this solution as the sample and perform the zinc procedure as described above.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.50 mg/L Zn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Zn.

#### Estimated Detection Limit (EDL)

The EDL for program 97 is 0.02 mg/L Zn. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## ZINC, continued

---

### Interferences

The following may interfere when present in concentrations exceeding those listed below.

Interfering Substance	Interference Level and Treatments
Aluminum	6 mg/L
Cadmium	0.5 mg/L
Copper	5 mg/L
Iron (ferric)	7 mg/L
Manganese	5 mg/L
Nickel	5 mg/L
Organic material	Large amounts may interfere. Perform the mild digestion (Section 2) to eliminate this interference.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment (see pH Interference in Section 1). Adjust pH to 4-5.

### Pollution Prevention and Waste Management

ZincoVer 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent the release of hydrogen cyanide gas.

In the event of a spill or release, clean up the area by following these steps:

- a) Use a fume hood or supplied-air or self-contained breathing apparatus.
- b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
- c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
- d) Neutralize and flush the solution down the drain with a large excess of water.

## ZINC, continued

### Summary of Method

Zinc and other metals in the sample complex with cyanide. Adding cyclohexanone selectively releases zinc. The zinc then reacts with the 2-carboxy-2'-hydroxy-5'-sulfoformazyl benzene (zincon) indicator and forms a blue color that is proportional to the zinc concentration.

### REQUIRED REAGENTS

Zinc Reagent Set, 20 mL size (100 tests).....24293-00  
Includes: (1) 14033-32, (1) 21066-69

Description	Quantity Required		Units	Cat. No.	
	Per Test				
Cyclohexanone .....	0.5 mL	.....	100 mL	MDB .....	14033-32
ZincoVer 5 Reagent Powder Pillows.....	1 pillow.....		100/pkg		21066-69

### REQUIRED APPARATUS

Pipet, serological, 10 mL..... 1 .....each .....532-38  
Pipet Filler, safety bulb ..... 1 .....each .....14651-00  
Sample Cell, 10-20-25 mL, w/cap ..... 2 .....6/pkg .....24019-06  
Squeezers, plastic dropper..... 1 .....20/pkg .....21247-20

### OPTIONAL REAGENTS

Bleach, household ..... 1 gal ..... buy locally  
Cylinder, graduated, mixing, 25mL .....each .....20886-40  
Hydrochloric Acid Standard Solution, 6 N ..... 500 mL .....884-49  
Nitric Acid, ACS ..... 500 mL .....152-49  
Nitric Acid 1:1..... 500 mL .....2540-49  
Sodium Hydroxide Standard Solution, 5.0 N..... 50 mL SCDB .....2450-26  
Water, deionized ..... 4 L .....272-56  
Zinc Standard Solution, 100 mg/L Zn..... 100 mL .....2378-42  
Zinc Standard Solution, PourRite ampule, 25 mg/L as Zn, 2mL..... 20/pkg .....14246-20

### OPTIONAL APPARATUS

Ampule Breaker, PourRite ampules .....each .....24846-00  
Aspirator, vacuum .....each .....2131-00  
Beaker, glass, 1000 mL .....each .....500-53  
Cylinder, graduated, 100 mL.....each .....508-42  
Cylinder, graduated, mixing, 250 mL .....each .....26362-46  
Filter discs, glass, 47 mm ..... 100/pkg .....2530-00  
Filter holder, 47 mm .....each .....2340-00  
Flask, erlenmeyer, 250 mL.....each .....505-46  
Flask, filtering, 500 mL.....each .....546-19

## ZINC, continued

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### OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Flask, volumetric, Class A, 100 mL .....	each.....	14574-42
Flask, volumetric, Class A, 1000 mL .....	each.....	14574-53
Hot plate, micro 115 V.....	each.....	12067-01
Hot plate, micro 230 V .....	each.....	12067-02
pH paper, 1 to 11 pH.....	5 rolls/pkg.....	391-33
pH meter, <i>Sensio</i> <sup>TM</sup> <i>I</i> , portable with electrode .....	each.....	51700-10
Pipet filler, safety bulb.....	each.....	14651-00
Pipet, serological, 2 mL .....	each.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet, TenSette, tips for 19700-01 .....	50/pkg.....	21856-96
Pipet, TenSette, 1.0 to 10.0 mL.....	each.....	19700-10
Pipet, TenSette, tips for 19700-01 .....	1000/pkg.....	21856-28
Pipet, TenSette, tips for 19700-10 .....	50/pkg.....	21997-96
Pipet, TenSette, tips for 19700-10 .....	250/pkg.....	21997-25
Pipet, volumetric, Class A, 5.00 mL.....	each.....	14515-37
Pipet, volumetric, Class A, 0.5 mL.....	each.....	14515-34

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## HOW TO ORDER

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**By Phone:**

6:30 a.m. to 5:00 p.m. MST  
Monday through Friday  
800-227-HACH (800-227-4224)

**By Mail:**

Hach Company  
P. O. Box 389  
Loveland, Colorado 80539-0389 U.S.A.

**By FAX:**

970-669-2932 (Hach Loveland)

**Information Required:**

- Hach account number (if available)
- Billing address
- Shipping address
- Your name and phone number
- Purchase order number
- Catalog number
- Brief description or model number
- Quantity

**Technical and Customer Service**

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use and to take your orders. Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224**.

## **HOW TO ORDER, continued**

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### **International Customers**

Hach maintains a network of dealers and distributors throughout the world.

#### **In Canada**

Hach Sales and Service Canada Ltd.  
1313 Border Street, Unit 34  
Winnipeg, Manitoba R3H 0X4  
Telephone: (204) 632-5598  
FAX: (204) 694-5134

#### **In other countries, contact:**

Hach Company World Headquarters  
P. O. Box 389  
Loveland, Colorado, U.S.A. 80539-0389  
Telephone: (1) (970) 669-3050  
FAX: (1) (970) 669-2932

Information presented on these pages applies only to Hach products manufactured for use within the United States. Exportation of these products renders these terms void.

### **Prices and Terms**

Prices are subject to change without notice. All prices are FOB from the shipping point (usually Ames, Iowa). Hach offers instant credit up to \$200 on Net 30 Day terms. Larger orders are subject to credit review. Customers may send remittance with orders or we can ship C.O.D. if you prefer.

### **Warranty**

Hach warrants its products to be of high quality, to be free of material defects on the date of shipment and to be as specified.

### **Limits of Usage**

Our chemicals and reagents are offered for laboratory and manufacturing use ONLY. They may not be used as drugs, cosmetics or food additives.

### **MSDS**

Hach Material Safety Data Sheets, among the most complete and informative in the industry, provide comprehensive safety data essential for day-to-day operations and safety training.

An MSDS accompanies all Hach chemical products including test kits. For an additional cost, we will print MSDSs on your own forms.

## ADDITIONAL INFORMATION

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### Label Information

Labels on Hach chemicals and reagents supply the following:

- Product Name -- In French, German, Italian and Spanish as well as English is printed on all but the smallest-size labels.
- Hach Catalog Number -- Makes reordering easy and helps match the appropriate MSDS.
- Storage Information and Lot Numbers -- Lot numbers made up of letters and numbers indicate an extended shelf life; a four-digit number indicates items should be rotated and checked with a standard to confirm performance. The lot number is essential if you call for technical assistance or with questions about reagent performance.

### Shipping

Our experienced warehouse staff packages your orders for safe arrival. Unless we are instructed otherwise, the best and most efficient mode of transportation is selected. Motor freight shipments will be sent freight collect unless you specify otherwise at the time you order.

If you have questions about methods for shipment and availability of special packaging, please ask when you place your order.

### Claims and Returns

We take extreme care to fill, check, re-check and pack orders properly. If errors or damages should occur, please report details to our Loveland Customer Service Department and to the carrier immediately. Be sure to keep all containers and packing materials.

AUTHORIZATION MUST BE OBTAINED from Hach when returning items for any reason. Call 1-800-227-4224 toll free. **ALL "FREIGHT COLLECT" SHIPMENTS OR MERCHANDISE RETURNED WITHOUT PROPER AUTHORIZATION FROM HACH WILL BE REFUSED.**





DOC022.53.80451

## **Pocket Colorimeter II**

User Manual

04/2014, Edition 1

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## Section 1 Specifications

Specifications are subject to change without notice.

Specification	Details
Dimensions (W x D x H)	6.1 x 3.2 x 15.2 cm (2.4 x 1.25 x 6 in.)
Enclosure	IP67, waterproof at 1 m (3.3 ft) for 30 minutes (battery compartment not included). Keep out of direct sunlight.
Light source	Light emitting diode (LED)
Detector	Silicon photodiode
Display	LCD with backlight
Weight	0.2 kg (0.43 lb)
Pollution degree	2
Installation category	I
Protection class	3
Power requirements	4 AAA batteries; approximate life of 2000 tests (use of backlight decreases this number) Rechargeable batteries are not recommended.
Operating environment	0 to 50 °C (32 to 122 °F), 0 to 90% relative humidity non-condensing
Storage temperature	-20 to 55 °C (-7.6 to 131 °F)
Photometric precision	± 0.0015 Abs
Wavelength	Fixed wavelength ±2 nm, different for each model
Filter bandwidth	15 nm
Absorbance range	0 to 2.5 Abs
Sample cell path length	1 cm (5–10 mL), 25 mm (10 mL)
Data storage	Last 10 measurements
Certifications	CE mark
Warranty	2 years

**Specifications**

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## Section 2 General information

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In no event will the manufacturer be liable for direct, indirect, special, incidental or consequential damages resulting from any defect or omission in this manual. The manufacturer reserves the right to make changes in this manual and the products it describes at any time, without notice or obligation. Revised editions are found on the manufacturer's website.

### 2.1 Safety information

#### **NOTICE**

The manufacturer is not responsible for any damages due to misapplication or misuse of this product including, without limitation, direct, incidental and consequential damages, and disclaims such damages to the full extent permitted under applicable law. The user is solely responsible to identify critical application risks and install appropriate mechanisms to protect processes during a possible equipment malfunction.

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

Make sure that the protection provided by this equipment is not impaired. Do not use or install this equipment in any manner other than that specified in this manual.

#### 2.1.1 Use of hazard information

##### **▲ DANGER**

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

##### **▲ WARNING**

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

##### **▲ CAUTION**

Indicates a potentially hazardous situation that may result in minor or moderate injury.

## General information

---

### NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.

#### 2.1.2 Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.

	This symbol, if noted on the instrument, references the instruction manual for operation and/or safety information.
	Electrical equipment marked with this symbol may not be disposed of in European domestic or public disposal systems. Return old or end-of-life equipment to the manufacturer for disposal at no charge to the user.

#### 2.1.3 Certification

##### Canadian Radio Interference-Causing Equipment Regulation, IECS-003, Class A:

Supporting test records reside with the manufacturer.

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de classe A répond à toutes les exigences de la réglementation canadienne sur les équipements provoquant des interférences.

##### FCC Part 15, Class "A" Limits

Supporting test records reside with the manufacturer. The device complies with Part 15 of the FCC Rules. Operation is subject to the following conditions:

1. The equipment may not cause harmful interference.
2. The equipment must accept any interference received, including interference that may cause undesired operation.

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## General information

Changes or modifications to this equipment not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment. This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be required to correct the interference at their expense. The following techniques can be used to reduce interference problems:

1. Move the equipment away from the device receiving the interference.
2. Reposition the receiving antenna for the device receiving the interference.
3. Try combinations of the above.

### 2.2 Product overview

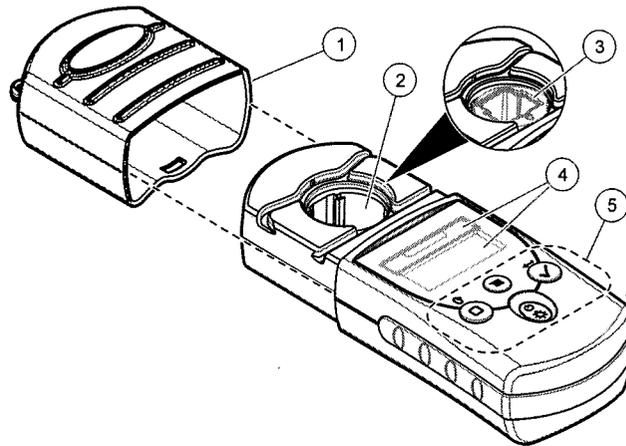
This instrument is a portable filter photometer used for testing water. Refer to Figure 1. This instrument is configured at the factory to measure one or two specific parameters. This instrument is calibrated at the factory. No user calibration is necessary.

*Note: This instrument has not been evaluated to measure chlorine and chloramines in medical applications in the United States.*

## General information

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Figure 1 Instrument overview



1 Instrument cap	3 Cell holder with 1-cm cell adapter <sup>1</sup>	5 Keypad
2 Cell holder	4 Display	

<sup>1</sup> Factory installed in some models

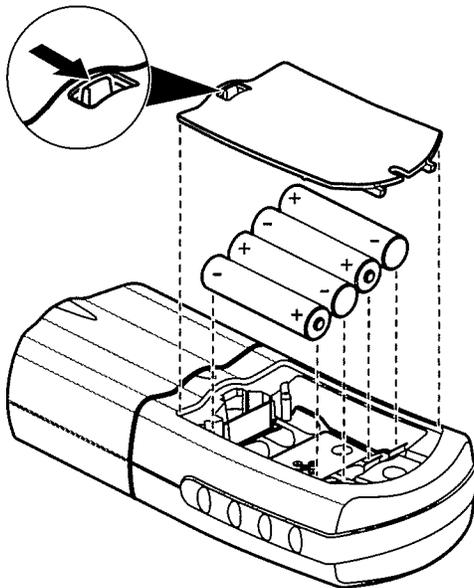
## Section 3 Startup

### 3.1 Install the batteries

▲ WARNING	
	Explosion hazard. Incorrect battery installation can cause the release of explosive gases. Be sure that the batteries are of the same approved chemical type and are inserted in the correct orientation. Do not mix new and used batteries.

Install the batteries as shown in Figure 2.

Figure 2 Install the batteries



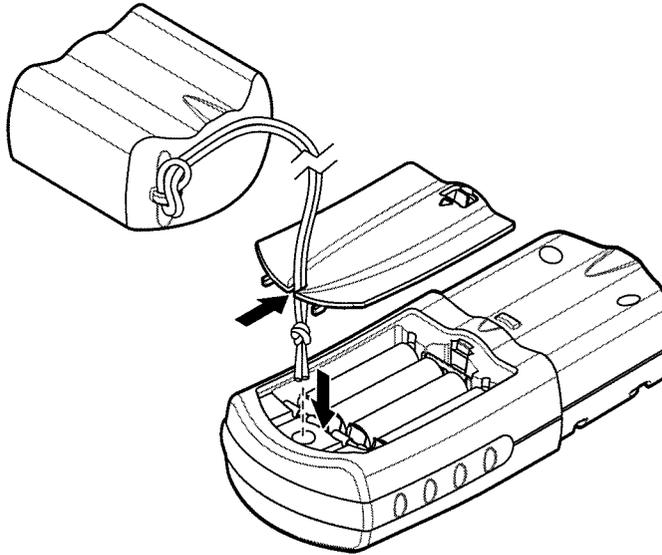
## Startup

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### 3.2 Install the cap cord

Attach the cap cord to prevent loss of the instrument cap. Refer to Figure 3.

Figure 3 Install the cap cord

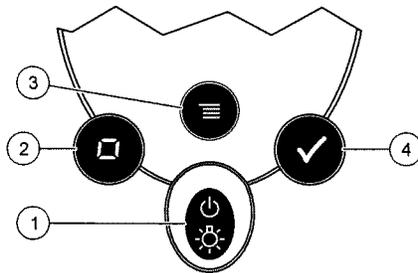


## Section 4 User interface and navigation

### 4.1 Keypad description

Figure 4 shows the keypad and gives the key functions.

Figure 4 Keypad



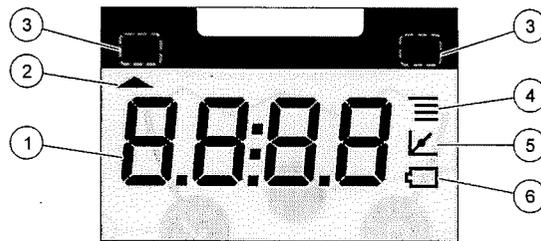
1 <b>Power/Backlight key:</b> Sets the power to on and off. Push and hold for 1 second to set the backlight to on or off.	3 <b>Menu key:</b> Enters and goes out of menu mode.
2 <b>Zero/Scroll key:</b> Sets the instrument to zero, scrolls through menu options and numbers	4 <b>Read/Enter key:</b> Starts a sample measurement, selects a menu option, moves the cursor to the next digit

### 4.2 Display description

Figure 5 shows the values and icons shown on the display.

## User interface and navigation

Figure 5 Display



1 <b>Numeric display:</b> Measured value or menu options	4 <b>Menu icon:</b> The instrument is in menu mode.
2 <b>Range icon:</b> Selected range or parameter	5 <b>Calibration adjusted icon:</b> The factory default calibration was adjusted or a user-entered calibration curve was entered. Refer to the expanded user manual on the manufacturer's website.
3 <b>Range value:</b> Range(s) or parameters	6 <b>Low battery icon:</b> Battery level is 10%. Flashes when the battery level is too low to complete measurements.

## Section 5 Operation

### 5.1 Configure the instrument

1. Push .
2. Push  to scroll through the menu options. Push  to select an option.

Option	Description
SEL	Sets the measurement range or parameter. Push  to toggle between the measurement ranges or parameters.
00:00	Sets the time in 24-hour format (hh:mm). Push  to change the time. Push  to change the first digit, then  to go to the next digit.
rCL	Shows the last 10 measurements recorded. Push  to show the recorded measurements (01—most recent measurement, 10—oldest measurement). Push  to scroll through the measurements. To select a measurement by number, push  to select the number and then  . Push  to go out of this option.
SCA	Refer to Standard calibration adjust on page 17.

3. Push  to go back to measurement mode.

### 5.2 Run a test

<b>▲ WARNING</b>	
	Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.
	

The basic measurement steps necessary to run a test follow. Refer to the applicable method to run a specific test.

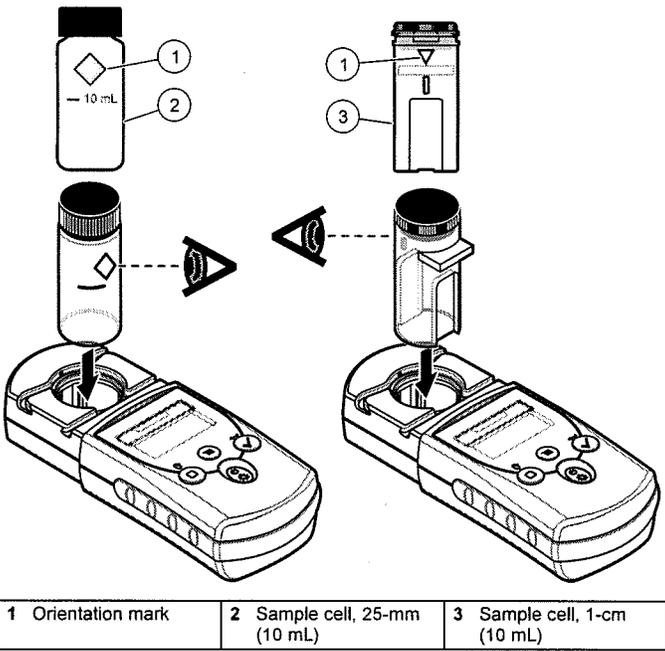
1. Select the applicable measurement range or parameter. Refer to Configure the instrument on page 13.
2. Prepare the blank according to the method document. Make sure to use the correct sample cell size. Rinse the sample cell and cap with the blank before the sample cell is filled.

## Operation

---

3. Close the sample cell and clean the optical faces of the sample cell with a lint-free cloth.
4. Insert the blank sample cell into the cell holder. Make sure to install the blank sample cell in the correct and consistent orientation so that the results are more repeatable and precise. Refer to Figure 6.
5. Install the instrument cap over the cell holder. Refer to Figure 7.
6. Push  to set the instrument zero. The display shows "0.000", or the degree of resolution that was previously selected.
7. Prepare the sample. Rinse the sample cell and cap with the sample three times before the sample cell is filled. Add reagents as specified by the method document.
8. Close the sample cell and clean the optical surfaces of the cell with a lint-free cloth.
9. Insert the sample into the cell holder. Make sure to install the sample cell in the correct and consistent orientation so that the results are more repeatable and precise. Refer to Figure 6.
10. Install the instrument cap over the cell holder. Refer to Figure 7.
11. Push . The display shows the results in concentration units or absorbance.  
*Note: The result flashes if the result is less or more than the instrument range.*
12. Remove the prepared sample from the cell holder.
13. Immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.

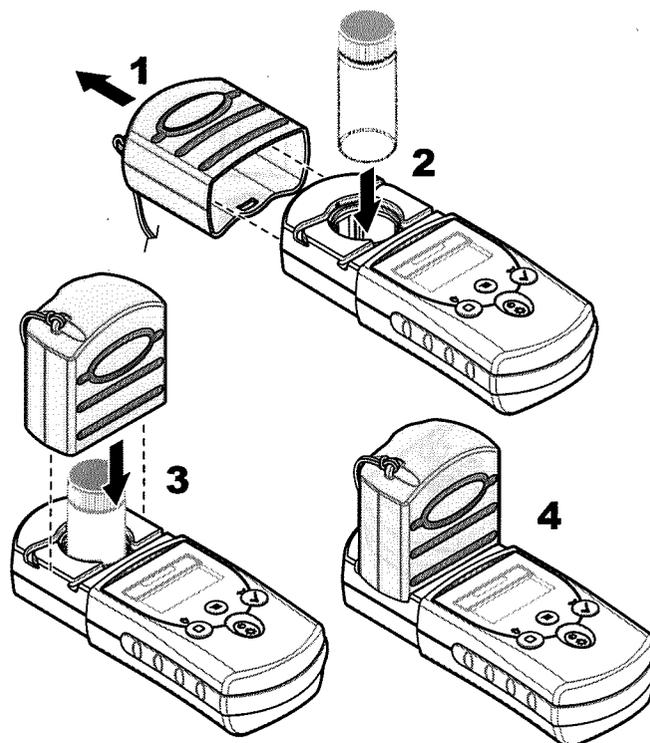
Figure 6 Sample cell orientation



## Operation

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Figure 7 Install the instrument cap over the cell holder



### 5.3 Show the recorded measurements

Refer to the "rCL" option in Configure the instrument on page 13.

## 5.4 Standard calibration adjust

Use the standard calibration adjust (SCA) option when a calibration must be adjusted to meet regulatory requirements. The factory calibration is adjusted slightly with the standard calibration adjust (SCA) option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.

*Note: For instruments with factory-calibrated ranges or methods, the standard calibration adjust (SCA) feature is disabled when a user-entered method is entered into the instrument. To set SCA back to on, set the instrument to the factory default calibration. Refer to Set to the factory default calibration on page 22.*

### 5.4.1 Adjust the factory calibration with a standard

1. Complete the test procedure for the range to calibrate. For the sample, use the standard solution concentration given in the test procedure documentation.

*Note: If a standard solution concentration is not given in the test procedure documentation, a different known standard can be used.*

2. When the test procedure is completed, push .
3. Push  until "SCA" shows, then push .
4. If a different known standard is used, enter the value of the standard:
  - a. Push  until "Edit" shows, then push .
  - b. Push  to enter the value of the standard. Push  to go to the next digit.
5. Push  to add the standard calibration adjust value to the factory calibration curve.

The calibration adjusted icon shows on the display. Refer to Figure 5 on page 12.

## Operation

---

### 5.4.2 Set the standard calibration adjust to off

To use the factory default calibration again, set standard calibration adjust (SCA) to off.

1. Push .
2. Push  until "SCA" shows, then push .
3. Push  until "OFF" shows, then push .

*Note: To set the SCA function to on again, calibrate with a standard.*

### 5.5 User-entered calibration

This instrument accepts a user-prepared calibration curve. The calibration curve can be from 0 to 2.5 absorbance. Make sure that the calibration curve includes standard values that are less and more than the range of interest.

The instrument range will be the same as the calibration range. For example, when the standards that are used are 1.00, 2.00 and 4.00. The instrument range is 1.00 to 4.00.

There are two options to enter a user calibration curve:

- **Enter a calibration curve with standards**—The standard solution values are entered with the keypad and the absorbance values are measured.
- **Enter a calibration curve with the keypad**—The standard solution values and absorbance values are entered with the keypad.

*Note: If the instrument is set to off or the instrument power is removed before a user-entered calibration curve is completed, the calibration curve is not saved. The instrument automatically switches off in user-entered calibration entry mode after 60 minutes of no activity. User-entered calibrations are completed when the user goes out of calibration (cal) mode or edit mode.*

#### 5.5.1 Channel restrictions

A user-entered calibration curve can be entered into any channel that does not contain a factory-programmed curve. These channels have the label:

- "abs" on the instruments that have a single factory calibration

---

## Operation

- "1" and "2" on the single wavelength instruments that are not calibrated

Any chemistry that can be done at the instrument wavelength can contain a user-entered calibration in these channels.

### 5.5.2 Enter a calibration curve with standards

*Note: Deionized water can be used for the blank unless the sample is significantly more turbid or has more color than deionized water.*

1. Set the instrument to the range to calibrate. Refer to Configure the instrument on page 13.
2. Prepare the blank and the reacted standard solution. Refer to the test procedure. Let the color fully develop.
3. Set the instrument to zero.
  - a. Insert the blank sample cell in the cell holder.
  - b. Install the instrument cap over the cell holder.
  - c. Push . The display shows "---", then "0.000".
  - d. Remove the instrument cap.
  - e. Remove the sample cell from the cell holder.
4. Push and hold  until "USER" and then "CAL" shows, then push .

*Note: If "USER" and "CAL" do not show, the factory calibration cannot be changed on the selected range.*

5. If "RES" shows on the display, set the resolution.
  - a. Push . The resolution setting (decimal placement) shows.
  - b. To change the resolution, push , then . Push  to save the change.
  - c. To not change the resolution, push .

*Note: "RES" does not show on the display of factory-calibrated instruments because the resolution cannot be changed. Only instruments that are not factory calibrated or have "abs" as one of the two ranges show "RES" on the display.*

## Operation

---

6. When "S0" shows on the display, push ✓. Push  to enter the blank value, then push ✓.  
*Note: Push ✓ to go to the next digit.*
7. When "A0" shows on the display, measure the absorbance of the blank.
  - a. Insert the blank sample cell in the cell holder.
  - b. Install the instrument cap over the cell holder.
  - c. Push ✓. The display shows the absorbance value for "S0".
  - d. Remove the sample cell from the cell holder.
8. Push  to show "S1".
9. When "S1" shows on the display, push ✓. Push  to enter the first standard value, then push ✓.  
*Note: Push ✓ to enter the next digit.*
10. When "A1" shows on the display, measure the absorbance of the reacted standard solution.
  - a. Insert the reacted standard sample cell in the cell holder.
  - b. Install the instrument cap over the cell holder.
  - c. Push ✓. The display shows the absorbance value for "S1".
  - d. Remove the sample cell from the cell holder.
11. The calibration is completed with two calibration points. If additional standards are necessary for calibration:
  - a. Push  until "Add" shows, then push ✓.
  - b. Do steps 9–10 again to enter more standards.
12. Push  two times to go back to measurement mode.

### 5.5.3 Enter a calibration curve with the keypad

At least two data pairs are necessary to enter a user-prepared calibration curve. A concentration value and the absorbance value for the given concentration is necessary for each data pair. A maximum of 10 data pairs can be entered.

*Note: This procedure can also be used to change the data pairs in a user-entered calibration curve or factory calibration curve.*

---

## Operation

1. Set the instrument to the range to calibrate. Refer to Configure the instrument on page 13.
2. Push and hold  until "USER" and then "CAL" shows, then push .  
*Note: If "USER" and "CAL" do not show, the factory calibration cannot be changed on the selected range.*
3. Push  until "EDIT" shows, then push .
4. If "RES" shows on the display, set the resolution.
  - a. Push . The resolution setting (decimal placement) shows.
  - b. To change the resolution, push , then . Push  to save the change.
  - c. To not change the resolution, push .*Note: "RES" does not show on the display of factory-calibrated instruments because the resolution cannot be changed. Only instruments that are not factory calibrated or have "abs" as one of the two ranges show "RES" on the display.*
5. When "S0" shows on the display, push . Push  to enter the concentration value of the first data pair, then push .
- Note: Push  to go to the next digit.*
6. When "A0" shows on the display, push . Push  to enter the absorbance value of the first data pair, then push . "S1" shows on the display.
7. Do steps 5–6 again to enter the second data pair (S1 and A1).
8. The calibration is completed with two data pairs. If additional data pairs are necessary for calibration:
  - a. When "Add" shows, push .
  - b. Do steps 5–6 again to enter more data pairs.
9. Push  two times to go back to measurement mode.

### 5.5.4 Remove a calibration point

To remove a calibration point from a user-entered calibration curve:

## Section 6 Maintenance

### ▲ CAUTION



Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

### NOTICE

Do not disassemble the instrument for maintenance. If the internal components must be cleaned or repaired, contact the manufacturer.

#### 6.1 Clean the instrument

Clean the exterior of the instrument with a moist cloth and a mild soap solution and then wipe the instrument dry.

#### 6.2 Clean the sample cells

### ▲ CAUTION



Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.



### ▲ CAUTION



Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

Most laboratory detergents are used at recommended concentrations. Neutral detergents, such as Liquinox, are safer to use when regular cleaning is necessary. To decrease the cleaning times, increase the temperature or use an ultrasonic bath. To complete the cleaning, rinse a few times with deionized water and then let the sample cell air dry. Sample cells may also be cleaned with acid, followed by a thorough rinse with deionized water.

*Note: Always use acid to clean sample cells that were used for low-level metal tests.*

## **Maintenance**

---

Special cleaning methods are necessary for individual procedures. When a brush is used to clean sample cells, take extra care to avoid scratches on the interior surfaces of the sample cells.

### **6.3 Replace the batteries**

Replace the batteries when the battery power level is low. Refer to Install the batteries on page 9.

## Section 7 Troubleshooting

Error	Description	Solution
E-0	No zero	In user calibration mode, a standard solution was measured before the instrument zero was set. Measure a blank solution to set the instrument to zero.
E-1	Ambient light error <sup>1</sup>	There is ambient light in the cell holder. Make sure that the instrument cap is fully installed over the cell holder.
E-2	LED error <sup>1</sup>	The LED (light source) is out of regulation. Replace the batteries. Make sure that the LED in the cell holder comes on when ✓ or  is pushed.
E-3	Standard adjust error	<ul style="list-style-type: none"> <li>• The measured value of the standard solution is more than the adjustment limits. Prepare a fresh standard.</li> <li>• The standard solution is not within the concentration range that can be used for standard calibration adjust. Prepare a standard with a value at or near the recommended concentrations given in the procedure.</li> <li>• Make sure that the concentration of the standard solution is entered correctly.</li> </ul>
E-6	Abs error	The absorbance value is not correct or the user-entered calibration curve has fewer than two points. Enter or measure the absorbance value again.
E-7	Standard value error	The standard solution concentration is equal to another standard solution concentration that is already entered in the user-entered calibration curve. Enter the correct standard concentration.
E-9	Flash error	The instrument is not able to save data.

## Troubleshooting

---

Error	Description	Solution
Reading flashes	The reading is more or less than the instrument range. <sup>2</sup>	If the reading is less than the instrument range, make sure that the instrument cap is fully installed over the cell holder. Measure a blank. If the blank reading is not zero, set the instrument to zero again.
		If the reading is more than the instrument range, identify if there is a light blockage in the cell holder. Dilute the sample. Do the test again.
		For factory-calibrated programs, the maximum and minimum values always equal the factory-calibrated values and cannot be changed.

<sup>1</sup> When an E-1 or E-2 error occurs on a measurement, the display shows ".\_.". The decimal place depends on the chemistry. If the E-1 or E-2 error occurs while the instrument is set to zero, set the instrument to zero again.

<sup>2</sup> The flashing value will be 10% over the upper test range limit.

# Detergents SAM

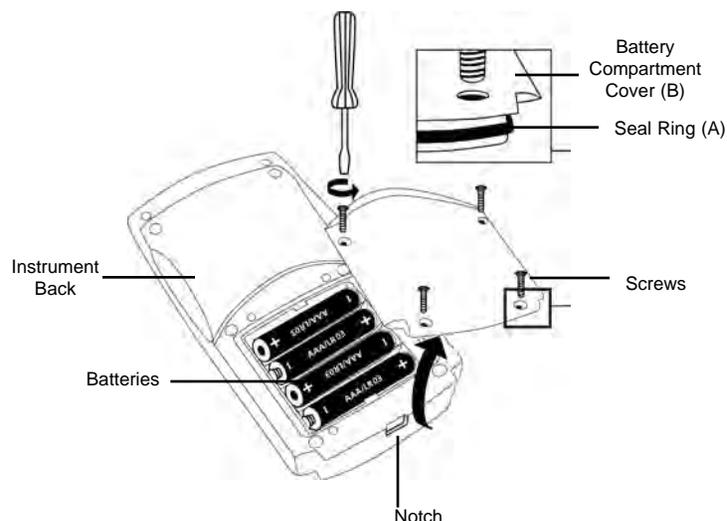
I-2017

0 to 2.50  
PPM (mg/Liter)



Simplicity in Water Analysis

## Battery Replacement



To ensure that the instrument is waterproof:

- seal ring (A) must be in position
- battery compartment cover (B) must be fixed with the four screws

### To Set Zero

1. Press the Power key.
2. The display will show “**det**”.
3. Insert the ZERO test tube (supplied in detergents test kit) filled with distilled or deionized water into the sample cell compartment (with mild downward pressure), making sure that it is fully seated.
4. Place the light shield over the ZERO test tube.
5. Press the Zero/Test key. The “**det**” symbol will flash for approximately 8 seconds, then the display will show “0.0.0”.

### To Make a Measurement

1. Follow the Test Procedure in the Instrumental Detergents Test (Cat. # R-9423).
2. Insert the resulting Detergents test tube into the sample cell compartment (with mild downward pressure), making sure that it is fully seated.
3. Place the light shield over the test tube.
4. Press the Zero/Test key. The “**det**” symbol will flash for approximately 3 seconds, then the sample test result will appear in the display as ppm (mg/Liter).

## Operating Tips

- Upon startup, the photometer automatically proceeds to the zeroing process. Every time the photometer powers on, it must be re-zeroed.
- To re-zero the photometer, it must be turned off and back on again.
- A series of readings can be taken without re-zeroing, as long as the photometer stays on during the series.
- Protect photometer from extreme humidity, corrosive fumes and dusty areas. Store in a cool, dry place.
- Remove the batteries when photometer is not in use.
- Press the ! key to turn the display back light on or off.
- When moving the photometer from one temperature extreme to another, wait at least 10 minutes before use to allow photometer to come to temperature equilibrium.
- Contamination of the optics in the sample chamber will result in incorrect measurements. The windows in the sample chamber should be checked at regular intervals and cleaned as necessary. Use a soft moist cloth or cotton swab for cleaning purposes.
- If the sample cell adapter has been removed, it must be replaced with proper orientation, aligning the triangle on the adapter with the triangle on the photometer.

## Displays and Troubleshooting

**E01:** Light absorption too great (dirty optics)

**E20/E21:** Too much light reaching detector

**E22 or Battery Icon:** Battery should be replaced

**E27/E28/E29:** Instrument zeroed incorrectly, misaligned adapter, test tube not properly seated, dirty optics or failing light source.

**Hi/E03:** Measuring range exceeded or excessive turbidity

**Lo:** Test result has a negative value (less than 0 ppm)

## Specifications

**Auto Shutoff:** After 15 minutes of non-use

**Optics:** 660 nm LED/interference filter and photosensor in transparent sample chamber

**Operating Temp.:** 5 to 40°C (41 to 104°F)

**Battery:** 4 AAA batteries (approx. 5,000 tests or 17 hours)

**Waterproof:** Floating, IP68 (1 hour at 0.1 meter)

**Wavelength Accuracy:** ± 1 nm

**Photometric Accuracy:** 3% full scale (T = 20 - 25° C / 68 - 77° F)

**Photometric Resolution:** 0.01 A

**Ambient Conditions:** Temperature 5 - 40° C / 41 - 104° F

Rel. humidity 30 - 90 % (non-condensing)

**CE:** Certificate of Declaration of CE-Conformity available upon request.

## Menu Selection

### Setting Date and Time

Upon initial start-up, the SAM will display "Set", "dAtE", and "YYYY", then a 4 digit number. Proceed to Step 4 in the procedure below to set the date and time, or power the instrument off and on again to bypass this process. At any time that the time and/or data need to be reset, follow steps 1-6 of the procedure below.

1. Press the Mode key and hold. Turn the instrument on by pressing and releasing the Power key. Once three decimal points appear in the display, release the Mode key. The display will show "di 5".
2. Press and release the ! key until the display shows arrows in the upper right and lower left corners of the display, pointing to "Time" and "Date".
3. Press the Mode key. "Set", "dAtE" will briefly appear in the display.
4. Date and time settings are displayed in the following order: Year ("YYYY"), Month ("MM"), Day ("dd"), Hour ("hh"), Minutes ("mm"). Increase the displayed value for each setting by pressing the Mode key or decrease the value by pressing the Zero/Test key until the desired value is displayed.
5. Press the ! key to save the displayed value and to proceed to the next setting.
6. After setting the minutes, press the ! key. The display will flash "iS" "SEt" and then will return to the measurement mode.

### Recall of Stored Data

The SAM photometer automatically stores the last 15 data sets. To recall stored data:

1. Press the Mode key and hold. Turn the instrument on by pressing and releasing the Power key. Once three decimal points appear in the display, release the Mode key. The display will show "di 5".  
**Note:** If the instrument is already on, press and hold the ! key for at least 4 seconds and release to access the stored data.
2. Press the Mode key. The photometer will display the stored data sets in the following format:
  - a. Sample Number: nXX (e.g. n15, n14, ... n1)
  - b. Year: XXXX (e.g. 2017)
  - c. Date: mm.dd (e.g. 03.15)
  - d. Time: hh.mm (e.g. 12:05)
  - e. Analyte
  - f. Result
3. Press the Zero/Test key to repeat the current data set.
4. Press the Mode key to proceed to the next data set.
5. Press the ! key to return to the measurement mode.

*www.chemetrics.com*

*4295 Catlett Road, Midland, VA 22728 U.S.A.*

*Phone: (800) 356-3072; Fax: (540) 788-4856*

*E-Mail: orders@chemetrics.com*

*Feb. 18, Rev. 11*

# Chlorine Vacu-vials® Kit

**K-2513:** 0 - 5.00 ppm (Prog. # 32)

**K-2523:** 0 - 5.00 ppm (Prog. # 32 or 33)

## Instrument Set-up

For CHEMetrics photometers, follow the **Setup and Measurement Procedures** in the operator's manual.

For spectrophotometers, follow the manufacturer's instructions to set the wavelength to 515 nm and to zero the instrument using the ZERO ampoule supplied.

## Safety Information

Read SDS (available at [www.chemetrics.com](http://www.chemetrics.com)) before performing this test procedure. Wear safety glasses and protective gloves.

## Free Chlorine Procedure

1. Fill the sample cup to the 25 mL mark with the sample to be tested (fig 1).
2. Place the Vacu-vial ampoule, tip first, into the sample cup. Snap the tip. The ampoule will fill leaving a bubble for mixing (fig 2).
3. To mix the ampoule, invert it several times, allowing the bubble to travel from end to end. Tap the bottom of the ampoule on a hard surface to cause any tiny bubbles that have collected on the ampoule wall to rise to the top of the liquid in the ampoule.
4. Dry the ampoule and wait **1 minute** for color development.
5. Insert the Vacu-vial ampoule into the photometer, flat end first, and obtain a reading in ppm (mg/Liter) chlorine (Cl<sub>2</sub>).

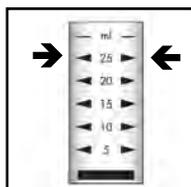


Figure 1

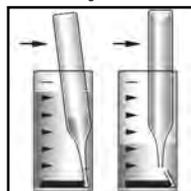


Figure 2

**NOTE:** If using a spectrophotometer that is not pre-calibrated for CHEMetrics products, then use the **equation below** or the **Concentration Calculator** found under the Support tab at [www.chemetrics.com](http://www.chemetrics.com).

$$\text{ppm} = 0.50 (\text{abs})^2 + 3.54 (\text{abs}) - 0.02$$

## Total Chlorine Procedure (K-2513 only)

1. Add 5 drops of A-2500 Activator Solution to the empty sample cup.
2. Fill the sample cup to the 25 mL mark with the sample to be tested.
3. Immediately perform the **Free Chlorine Procedure** starting with Step 2.

## Test Method

The Chlorine Vacu-vials®<sup>1</sup> test kit employs the DPD chemistry.<sup>2,3</sup> Free chlorine oxidizes DPD (N,N-diethyl-p-phenylenediamine) to form a pink colored species in direct proportion to the chlorine concentration. Other halogens, ozone and halogenating agents will produce high test results. Chlorine at concentrations significantly above the test range may prevent proper color development causing low test results.

**K-2513 only:** Total chlorine, the sum of free and combined chlorine, is determined by adding an excess of potassium iodide to the sample. Chloramines (combined chlorine) oxidize the iodide to iodine. The iodine then oxidizes DPD to the pink colored species.

1. Vacu-vials is a registered trademark of CHEMetrics, Inc. U.S. Patent No. 3,634,038
2. APHA Standard Methods, 22nd ed., Method 4500-Cl G - 2000
3. EPA Methods for Chemical Analysis of Water and Wastes, Method 330.5 (1983)

Visit [www.chemetrics.com](http://www.chemetrics.com) to view product demonstration videos.

Always follow the test procedure above to perform a test.



[www.chemetrics.com](http://www.chemetrics.com)

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Feb. 18, Rev. 23



**INSTRUCTION MANUAL**

**EC/TDS/SALT Testr**

Large Screen Waterproof Multi Range  
Conductivity/TDS/Salt Tester  
with Temperature Display

**Introduction**

Thank you for selecting microprocessor-based waterproof EC / TDS / SALT tester with large dual line display. You have one of the following models:

- ECTestr11 • ECTestr11+ • TDSTestr11 • TDSTestr11+ • SALTTestr11

Non-plus models (ECTestr11, TDSTestr11 & SALTTestr11) come with user-replaceable two-pin type sensor and have many user friendly features such as Dual-range measurement, the Hold function, Automatic Temperature Compensation (ATC) and Self-Diagnostic Messaging capabilities.

Plus models (ECTestr11+ & TDSTestr11+) come with the user-replaceable cup type sensor and have additional features such as Multi-range measurement, up to 3-point calibration and higher resolution measurement.

**Before You Begin**

Remove the electrode's protective cap. Soak the electrode for a few minutes in alcohol to remove any oil stains on the electrodes which will affect the accuracy of the tester. Rinse thoroughly with de-ionized water and shake off dry.

**Key Functions**

Key	Function
	- Power on and off the tester (The tester automatically switches off, if no button is pressed for 8.5 seconds)
	- In measurement mode, temperature reading switches between Celsius & Fahrenheit - In calibration mode, switches the tester to temperature calibration mode - In temperature calibration mode, exits calibration mode without confirming calibrated values
	- In measurement mode, switches to hold mode freezing the display. - In hold mode, switches back to measurement mode - In manual calibration and temperature calibration modes, exits calibration mode without confirming calibrated values - In range selection mode, selects a range
	- In measurement mode, enters calibration mode - In calibration mode, adjusts the calibration values - In hold mode, enters TDS factor setting mode - In TDS factor setting mode, adjusts TDS factor

Note: INC & DEC keys are located inside the battery compartment. Refer figure 1.

Note: For ECTestr11 & ECTestr11+ models, the caption of HOLD key is 'HOLD/ENT'

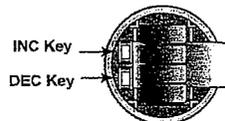


Figure 1: Battery compartment

**Switching On**

Press ON/OFF key to switch on the tester. The LCD shows the power-up sequence as illustrated in Figure 2. When the tester is on, if you do not press a key for 8.5 minutes, the tester automatically switches off to conserve batteries.

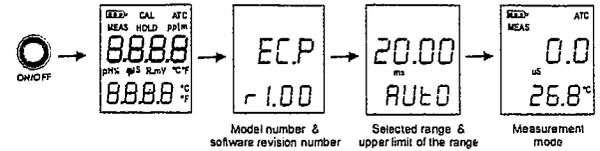


Figure 2: Power Up Sequence

**Range Selection**

Depending on the selected model, you can set the tester to limit its reading to a particular measuring range (PU, LO or HI) or full scale (AUTO). The default setting is AUTO. When you select a range other than AUTO, the tester can be calibrated only for that particular range. If you try to measure a sample which has a higher conductivity/TDS value than that of the selected measuring range, the LCD shows 'OR' error message. Refer Specifications section for available ranges of the selected model.

To select a range:

1. Switch off the tester. Press and hold °C/°F key and then switch on the tester using ON/OFF key. Release °C/°F key.
2. The tester goes to range selection mode. The LCD shows the currently selected Range (the default is AUTO) in the lower display. The upper display shows the maximum possible reading for the selected range. Press HOLD key repeatedly until you see the required range (PU, LO or HI).

Note: If no key is pressed within 5 seconds, LCD shows power-up sequence and tester goes to measurement mode.

3. The tester automatically confirms the last selection if no key is pressed for 5 seconds. Upper display momentarily shows 'CO'. The LCD shows power-up sequence and the tester goes to measurement mode.

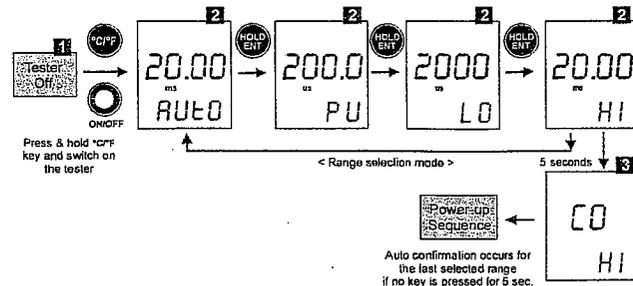


Figure 3: Range selection sequence from AUTO to HI for ECTestr11+

## Measurement

1. Press the ON/OFF key to switch on the tester. The 'MEAS' indicators appears when the tester is in measurement mode.
2. Dip the electrode into the test solution making sure that it is fully immersed. Stir to clear any trapped air bubbles from the electrode and let the reading stabilize. For plus models, you can opt for the cup style measurement by filling the electrode cup with sample of test solution.

*Note: The LCD indicates 'Or' (over range) if the reading is outside the selected range. If this occurs, select an appropriate range to suit the reading.*

3. The upper display shows the main reading (conductivity/TDS/Salt) of the solution, automatically temperature compensated (ATC) to normalized temperature of 25°C. The lower display shows the temperature of the solution.

## HOLD Function

This feature lets you freeze the display for a delayed observation.

1. Press HOLD key to freeze the measurement. The tester goes to hold mode and 'HOLD' indicator is displayed in LCD. The measurements are frozen and the 'MEAS' indicator disappears.
2. Press HOLD key again to release the measurement. The 'HOLD' indicator is no longer displayed. The tester goes back to measurement mode.

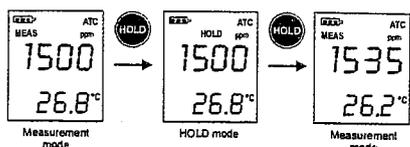


Figure 4: HOLD Function

## Temperature Unit of Measurement Selection

This feature lets you set the unit of measurement of temperature to either Celsius (°C) or Fahrenheit (°F).

When the tester is in the measurement mode, press °C/°F button. The temperature display toggles between the Celsius and Fahrenheit reading.

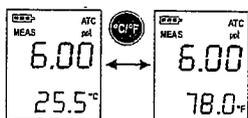


Figure 5: Temperature unit of measurement selection

## About Calibration

To ensure higher accuracy, the tester must be calibrated on a regular basis. Calibration can be manual or automatic (only for ECTestr11 & ECTestr11+); calibration can be 1-point or multi-point. You can choose any combination of the above two options for calibration. If you calibrate the tester for 1-point, the calibration is applied for all the measuring ranges. In applications where you need higher accuracy, and when you intend to measure values in more than one range, it is recommended to select multi-point calibration.

## Selection of Automatic or Manual Calibration

ECTestr11 & ECTestr11+ models support both automatic & manual calibration while all other models have to be calibrated manually. In automatic calibration, the tester automatically detects and verifies known conductivity standard solutions (84uS, 1413uS & 12.88mS). In manual calibration, you can use non-standard solutions which may be specific for your application.

The factory default is Automatic calibration (Auto). You can enable or disable automatic calibration as described below (for only ECTestr11 & ECTestr11+ models).

## Selection of 1-point or Multi-point Calibration

The factory default is 1-point Calibration. For higher accuracy, it is recommended that you calibrate the tester for multiple ranges if you intend to measure values in multiple ranges. You can enable or disable multi-point calibration as described below.

*Note: If you have selected a specific measuring range for the tester, selecting multi-point calibration has no meaning, as the tester can only be calibrated for 1-point for the selected range. Set the tester to 'AUTO' measuring range if you wish to calibrate multi-points.*

To enable/disable auto calibration and multi-point calibration:

1. Switch off the tester. Press and hold INC key and then switch on the tester using ON/OFF key.
2. The tester goes to auto calibration selection mode. The lower display shows 'A.CAL' and the upper display blinks the current choice ('Yes' or 'No').

*Note: This mode is available only for ECTestr11 & ECTestr11+ models. For other models, go directly to 1-point selection mode, described in step 4 below.*

Press INC or DEC key to select 'Yes' (to enable auto calibration) or 'No' (to disable auto calibration)

*Note: Press °C/°F key, if you wish to skip this setting without confirming changes.*

*Note: Press °C/°F key twice, if you wish to return to measurement mode without confirming changes.*

3. Press HOLD/ENT key to confirm the selection. The display shows 'CO'.
4. The tester goes to 1-point calibration selection mode. The lower display shows '1.Pnt' and the upper display blinks the current choice ('Yes' or 'No'). Press INC or DEC key to select 'Yes' (to enable 1-point calibration) or 'No' (to disable 1-point calibration, i.e. to enable multi-point calibration).

*Note: Press °C/°F key if you wish to skip this setting without confirming.*

5. Press HOLD/ENT key to confirm the selection. The display shows 'CO' for few seconds and then shows power-up sequence. The tester goes to measurement mode.

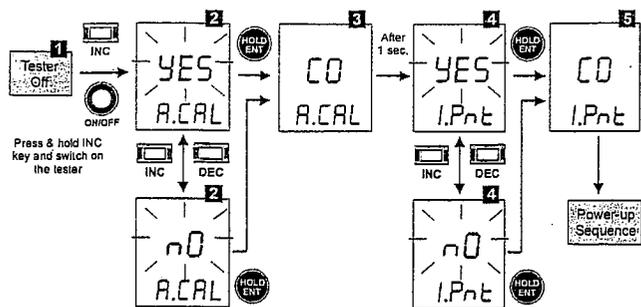


Figure 6: Selecting auto calibration & 1-point calibration for ECTestr11+

### Auto Calibration

Auto calibration feature is available only for conductivity in ECTestr11 & ECTestr11+ models. Make sure that 'auto calibration' is enabled as described in the previous section. Auto calibration is suitable if you use standard conductivity solutions for calibration process.

If you selected 1-point calibration, you need to choose a calibration standard corresponds to the selected measuring range of the tester listed below. If you have selected multi-point calibration & AUTO measuring range, you can choose any of the calibration standards listed below. During auto calibration, the tester recognizes the calibration standard if its value is within 50% tolerance. For multi-point calibration (with AUTO measuring range), the tester automatically scans through all possible calibration points until all of them are calibrated.

Selected Measuring Range	Calibration Standard
PU (0 to 200.0 uS/cm)	84 uS
LO (0 to 2000 uS/cm)	1413 uS
HI (0 to 20.00 mS/cm)	12.88 mS
AUTO	84 uS, 1413 uS, 12.88 mS

### To prepare calibration standards:

Use fresh calibration standard solutions listed in the above table. Prepare each solution in two beakers - one for rinsing and the other for calibration. Rinse the electrode in de-ionized water before calibration.

### To begin automatic calibration:

- Switch on the tester. Make sure the tester is in measuring mode. Press INC or DEC key to enter conductivity calibration mode.
- 'CAL' indicator appears in LCD. The display briefly shows 'CAL' and the number of points the tester will be calibrated.
- The upper display shows the conductivity reading and the lower display sequentially shows calibration standard values 1413 uS & 12.88 mS (for ECTestr11) or 84 uS, 1413 uS & 12.88 mS (for ECTestr11+) if the measuring range of the tester is set to AUTO.

Note: If you have selected a specific measuring range for the tester, the lower display shows the corresponding calibration standard value that matches the selected measuring range.

- Rinse the electrode with the calibration standard that you intend to calibrate and then dip the electrode in the other beaker with same calibration standard. Swirl gently to create a homogenous sample and allow time for the reading to stabilize.

Note: For multi-point calibration, the lower display automatically locks at the calibration standard value that closely matches. The tolerance range is  $\pm 50\%$  of the calibration standard. The tester shows error message 'Er.1' if you try to calibrate with a solution whose conductivity is outside the tolerance range.

Note: Press INC or DEC key if you wish to exit from auto calibration, during any of the above steps.

- Press HOLD/ENT key to confirm the calibration. LCD shows 'CO' for 2 seconds. The calibration is complete and the tester returns to measurement mode, if this is a 1-point calibration.
- For multi-point calibration, the tester goes to the next calibration point, lower display showing next calibration standard values. Rinse the electrode in de-ionized water and repeat step 4 & 5 to continue calibrating with next calibration standard solution.

Note: The tester shows error message 'Er.0' and returns to measuring mode if the temperature of the calibration solution is not within 0°C to 50°C.

Note: The tester shows error message 'Er.1' if you press HOLD/ENT key before the tester recognizes the calibration standard.

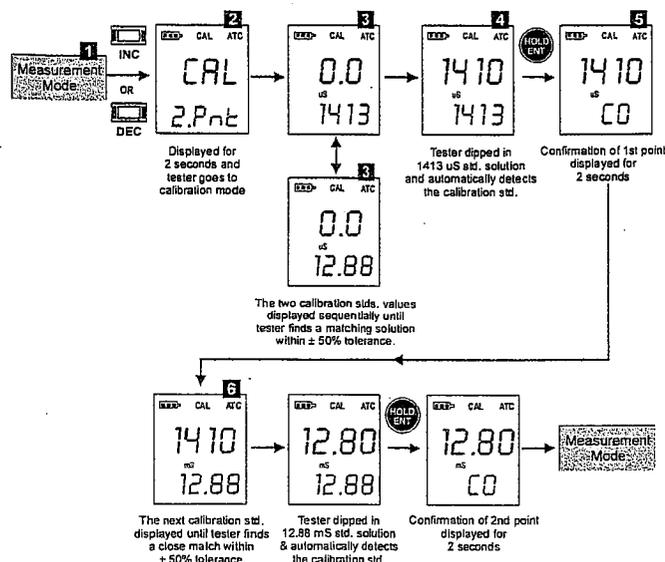


Figure 7: Two-point automatic calibration sequence for ECTestr11

## Manual Calibration

In manual calibration, the tester allows 1-point calibration for each measuring range. You can use customized calibration solutions with known conductivity/TDS values to calibrate the tester. The following table shows acceptable conductivity/TDS ranges of calibration solutions for each measuring range. Make sure your calibration solutions are within the given ranges.

Selected Measuring Range	Acceptable Calibration Standard Range	
	Conductivity	TDS/Salt
PU	2.0 - 200.0 uS/cm	2.0 - 200.0 ppm
LO	200 - 2000 uS/cm	200 - 2000 ppm
HI	2.00 - 20.00 mS/cm	1.00 - 10.00 ppt
AUTO	Select a calibration standard nearer to application sample	

To prepare calibration standards:

Use fresh calibration solutions. Measure conductivity/TDS values of the solution with a meter known to be accurate. Prepare each solution in two beakers - one for rinsing and the other for calibration. Rinse the electrode in de-ionized water before calibration.

To begin manual calibration:

1. Switch on the tester. Make sure the tester is in measuring mode. Rinse the electrode with the calibration standard that you intend to calibrate and then dip the electrode in the other beaker with same calibration standard. Swirl gently to create a homogenous sample and allow time for the reading to stabilize.
2. Press INC or DEC key to enter calibration mode. The 'CAL' indicator appears in LCD. The display briefly shows 'CAL' and the number of points the tester will be calibrated.
3. The upper display shows the measured conductivity/TDS reading of the solution based on previous calibration (if any) and the lower display shows the default (uncalibrated) conductivity/TDS reading.

**Note:** The tester shows error message 'Er.1':

- (a) If the reading is over range (Or) of selected measuring range of the tester, or
- (b) If the default (uncalibrated) reading is not within the acceptable calibration standard range.

Use INC and DEC keys to adjust the upper display to the correct conductivity/TDS value of the calibration solution.

**Note:** The calibration adjustment window is  $\pm 50\%$  from the default reading.

**Note:** If you do not press INC or DEC key within 5 seconds, the tester shows the confirmation 'CO' and returns to the measurement mode. However, the tester is not calibrated to new values yet. The old calibration is still active. If this happens, press INC or DEC key once again to enter calibration mode.

4. Wait for 5 seconds for the tester to automatically confirm the calibration by displaying 'CO' and return to the measurement mode.

**Note:** To exit calibration mode without confirming the calibration, press HOLD/ENT key before the automatic confirmation takes place.

**Note:** The tester shows error message 'Er.0' and returns to measuring mode if the temperature of the calibration solution is not within 0°C to 50°C.

5. For multi-point calibration rinse the electrode in de-ionized water and repeat step the above steps with another standard solution.

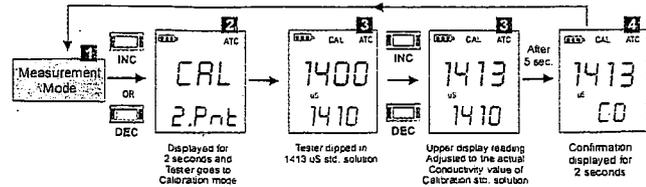


Figure 8: One-point manual calibration sequence for ECTestr11+

## TDS Factor Setting

TDS factor is only applicable for TDSTestr11 & TDSTestr11+ models. The factory default TDS factor is 0.71. You can adjust the TDS factor to suit different samples of your applications.

To change TDS factor:

1. Switch on the tester. Make sure the tester in measurement mode. Press HOLD key to bring the tester to the HOLD mode.
2. Press INC or DEC key to enter the TDS factor setting mode.
3. The upper & lower displays of LCD show the last configured TDS factor. The upper display is adjustable. Use the INC or DEC key to adjust the TDS factor. The adjustable range is 0.4 to 1.0.

**Note:** If you do not press INC or DEC key within 5 seconds, the tester shows the confirmation 'CO' and returns to measurement mode.

4. Wait for 5 seconds for the tester to automatically confirm the new setting by displaying 'CO' and return to the measurement mode.

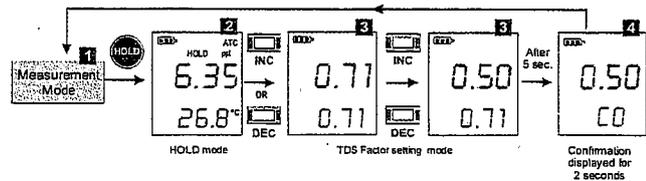


Figure 9: TDS Factor setting

## Temperature Calibration

Temperature calibration need not be performed every time, unless the temperature reading differs from that of an accurate thermometer. If temperature calibration is performed, Conductivity/TDS/Salt calibration is mandatory.

1. Switch on the tester. Make sure the tester is in measuring mode. If required, press °C/°F key to select the desired unit of measurement for temperature (Celsius or Fahrenheit). Dip the tester into a solution of known temperature and allow time for the temperature reading to stabilize.
2. Press INC or DEC key to bring the tester to the calibration mode. CAL indicator appears in LCD. Immediately press °C/°F key to switch to the temperature calibration mode.

*Note: When you enter calibration mode, if the conductivity/TDS/salt reading is outside the specified range (Or), the tester shows 'Er.1' error message. You can still proceed to the temperature calibration mode by pressing °C/°F key immediately. If the °C/°F key is not pressed within 2 seconds, the tester exits the calibration mode and returns to the measurement mode.*

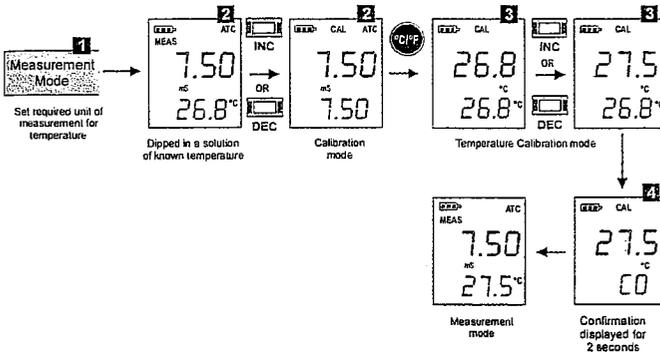


Figure 10: Temperature calibration sequence

3. The upper display shows the measured temperature reading based on the last set offset (if any) and the lower display shows the default (uncalibrated) temperature reading based on factory settings. Use INC and DEC keys to adjust the upper temperature reading to the known temperature value of the solution.

*Notes: The temperature adjustment window is  $\pm 5^{\circ}\text{C}$  ( $\pm 9^{\circ}\text{F}$ ) from the default reading.*

4. Wait for 5 seconds for the tester to automatically confirm the temperature calibration value by displaying 'CO' and return to the measurement mode.

*Note: To exit temperature calibration mode without confirming the calibration, press °C/°F key or HOLD/ENT key before the automatic confirmation takes place.*

*Note: The tester shows error message 'Er.0' and returns to measuring mode if the temperature of the solution is not within 0°C to 50°C.*

## Reset

Reset option allows you to restore the calibration and other parameters back to factory default settings.

1. Switch off the tester. Press and hold the HOLD key and then switch on the tester using ON/OFF key. Release HOLD key.
2. The lower display shows 'rSt' (reset) and the upper display blinks 'No'. Use INC or DEC key to select 'Yes' (to proceed with resetting) or 'No' (to quit without resetting).

*Note: Press °C/°F key if you wish to skip to measurement mode without making any selection.*

3. Press HOLD key to confirm your selection. LCD shows 'CO'. If 'Yes' is selected, the tester resets to its factory default values as listed below. LCD shows power-up sequence and tester goes to measurement mode.

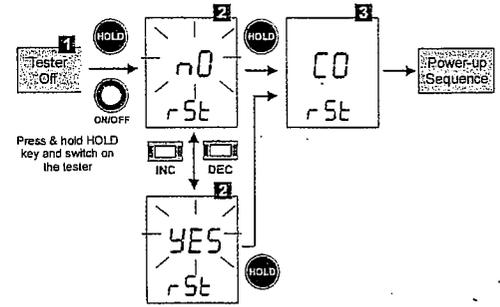


Figure 11: Resetting sequence

Parameter	Factory Default
User calibration (conductivity/TDS/salt)	(Reset)
Temperature unit of measurement	Celsius (°C)
Temperature offset	0
Auto calibration (for ECTestr11 & ECTester11+)	Enable
1-point calibration	Enable
Conductivity calibration factor (for ECTestr11 & ECTester11+)	1.0
TDS factor (for TDSTestr11 & TDSTester11+)	0.71

## Changing Batteries

Replace the batteries when the low battery indicator starts blinking.

1. Open the battery compartment lid (with attached lanyard loop).
2. Remove old batteries by pulling plastic ribbon. Replace with fresh ones. Note polarity as shown in figure 11.

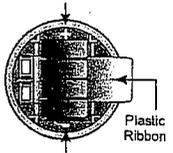


Figure 11: Battery compartment

## Electrode Maintenance

1. Always keep the sensor electrodes clean. Rinse the electrodes with de-ionized water and wipe them dry with clean cloth before storing with its protective cap. For cup type electrodes, remove the white plastic cup and insert to thoroughly clean viscous solutions. Never scratch electrodes with a hard substance.
2. For better performance, soak the electrode in alcohol for 10 to 15 minutes and rinse with de-ionized water before starting any measurement process. This is to remove dirt and oil stains on the electrode which may affect the accuracy of the measurements.

## Electrode Replacement

When the tester fails to calibrate or gives fluctuating readings in calibration standards, you need to change the electrode module. You can replace the electrode module at a fraction of the cost of a new tester.

1. With dry hands, grip the ribbed tester collar with electrode facing you. Twist the collar counter clockwise (see Figure 13-A). Save the ribbed tester collar and O-ring for later use.
2. Pull the old electrode module away from the tester.
3. Align the four tabs of the new electrode module so that they match the four slots on the tester (see Figure 13-B).
4. Gently push the module into the slots to sit it in position. Push the smaller O-ring fully onto the new electrode module. Push the collar over the module and thread it into place by firmly twisting clockwise.

*Note: It is necessary that you recalibrate the tester prior to measurement after an electrode replacement.*

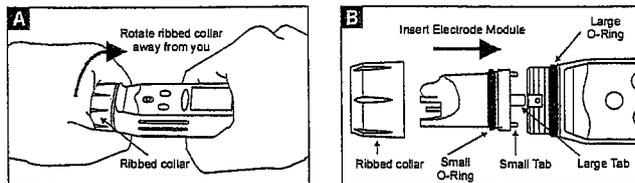


Figure 13: Removing collar & inserting electrode

## Self-Diagnostic Messages

Low battery indicator		3 Bars indicates Battery is full (100%)
		2 Bars indicates 50% of the battery life is left
		1 Bar indicates 25% of the battery life is left
Over range / Under range signal		Blinking battery casing indicates the need to replace batteries with fresh ones as specified by manufacturer
	Or / Ur (Still)	The sensor electrodes short circuited Replacement sensor is not connected properly to the tester during sensor replacement Measured value or temperature value exceeds the specified maximum or minimum value
	ATC / Or / Ur (Blinking)	Blinking 'ATC', 'Or' or 'Ur' indicates that there is a short or open circuit at the built in temperature sensor
Error Message	Er0	Calibration error due to temperature value not within the specified range
	Er1	Calibration error due to Conductivity/TDS/Salt value not within the specified calibration standard range

-11-

## Accessories

Item	Order Code
ECTESTR, TDSTESTR & SALTESTR replacement sensor	TDSENSOR
ECTESTR+ & TDSTESTR+ replacement sensor	TDSENSORPLUS

## Warranty

The waterproof testers are warranted to be free from manufacturing defects for 1 year and electrode module for 6 months; unless otherwise specified. If repair, adjustment or replacement is necessary and has not been the result of abuse or misuse within the time period specified, please return the tester - freight prepaid - and correction will be made without charge. Out of warranty products will be repaired on a charge basis.

## Return of Items

Authorization must be obtained from your distributor before returning items for any reason.

When applying for authorization, please include information regarding the reason the item(s) are to be returned.

*Note: Eutech Instruments/Oakton Instruments reserve the right to make improvements in design, construction and appearance of products without notice. Prices are subject to change without notice.*

## Specifications

Model	ECTestr11	ECTestr11+	TDSTestr11	TDSTestr11+	SALTestr11
Range:	PU LO 0 to 2000 uS/cm HI 0 to 20.00 mS/cm	0 to 200.0 uS/cm 0 to 2000 uS/cm 0 to 20.00 mS/cm	- 0 to 2000 ppm 0 to 10.00 ppt	0 to 200.0 ppm 0 to 2000 ppm 0 to 10.00 ppt	- - 0 to 10.00 ppt
Resolution:	PU LO 10 uS/cm HI 0.10 mS/cm	0.1 uS/cm 1 uS/cm 0.01 mS/cm	10 ppm 0.10 ppt	0.1 ppm 1 ppm 0.01 ppt	- - 0.10 ppt
Accuracy	± 1% of Full Scale				
Calibration Type	Auto or Manual	Auto or Manual	Manual	Manual	Manual
Calibration Points	1 or 2 points	1, 2 or 3 points	1 or 2 points	1, 2 or 3 points	1 point
Calibration Window	± 50% from each point				
Calibration PU Standard Range:	LO 200 - 2000 uS/cm HI 2.00 - 20.00 mS/cm	2.0 - 200.0 uS/cm 200 - 2000 uS/cm 2.00 - 20.00 mS/cm	200 - 2000 ppm 1.00 - 10.00 ppt	2.0 - 200.0 ppm 200 - 2000 ppm 1.00 - 10.00 ppt	- - 1.00 - 10.00 ppt
Sensor Type	Two-pin	Cup	Two-pin	Cup	Two-pin
TDS Factor	-	-	0.4 to 1.0 (Default 0.71)		-
Temperature	Range in °C 0.0 to 50.0°C Range in °F 32.0 to 122°F Resolution 0.1°C (0.1°F) Accuracy ±0.5°C (±0.9°F)				
Calibration point	1 point				
Calibration Window	± 5°C (± 9°F) from factory default				
ATC	0 to 50°C				
Temp Coefficient	2% per °C				
Normalization Temp	25.0°C				
Auto Off	8.5 minutes after last key press				
Operating Temp	0 to 50°C				
Power Battery	4 X 1.5V 'A76' micro alkaline battery				
Battery Life	> 150 hrs				
LCD Display	Custom Dual Display 27mm(H) X 21mm(W)				
Dimensions	Tester: 16.5 cm X 3.8 cm; 90g				
Weight	Boxed: 22cm X 6cm X 5cm; 170 g				

68X068056 Rev 2 May 06



# Pro30



## USER MANUAL

English

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## WARRANTY

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The YSI Professional 30 instrument (Pro30) is warranted for three (3) years from date of purchase by the end user against defects in materials and workmanship, exclusive of batteries and any damage caused by defective batteries. Pro30 cable/probe assemblies are warranted for two (2) years from date of purchase by the end user against defects in material and workmanship. Pro30 instruments & cables are warranted for 90 days from date of purchase by the end user against defects in material and workmanship when purchased by rental agencies for rental purposes. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio at +1 937 767-7241, 800-897-4151 or visit [www.YSI.com](http://www.YSI.com) for a Product Return Form. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

### LIMITATION OF WARRANTY

This Warranty does not apply to any YSI product damage or failure caused by:

1. Failure to install, operate or use the product in accordance with YSI's written instructions;
2. Abuse or misuse of the product;
3. Failure to maintain the product in accordance with YSI's written instructions or standard industry procedure;
4. Any improper repairs to the product;
5. Use by you of defective or improper components or parts in servicing or repairing the product;
6. Modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI'S LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

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## INTRODUCTION

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Thank you for purchasing the YSI Pro30, an instrument from the YSI *Professional Series* product family. The Pro30 measures conductivity and temperature in water. The Pro30 features an impact resistant and waterproof (IP-67) case, a rugged MS-8 (military-spec) cable connector, backlit display, user-selectable sensor options, 50 data set memory, internal barometer and a rubber over-mold case.

The Pro30 provides valuable instructions and prompts near the bottom of the display that will guide you through operation and use. However, reading the entire manual is recommended for a better understanding of the instrument's features.



*The Pro30 cannot communicate to a PC via a Pro Series communications saddle. Connecting the Pro30 to a communication saddle may cause erratic instrument behavior.*

## GETTING STARTED

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### INITIAL INSPECTION

---

Carefully unpack the instrument and accessories and inspect for damage. Compare received parts with items on the packing list. If any parts or materials are damaged or missing, contact YSI Customer Service at 800-897-4151 (+1 937 767-7241) or the authorized YSI distributor from whom the instrument was purchased.

### BATTERY INSTALLATION

---

The instrument requires 2 alkaline C-cell batteries. Under normal conditions, the average battery life is 425 hours at room temperature without using the back light. A battery symbol  will blink in the lower, left corner of the display to indicate low batteries when approximately 1 hour of battery life remains.

To install or replace the batteries:

1. Turn the instrument off and flip over to view the battery cover on the back.
2. Unscrew the four captive battery cover screws.
3. Remove the battery cover and remove the old batteries if necessary.
4. Install the new batteries, ensuring correct polarity alignment (figure 1).

- Place the battery cover on the back of the instrument and tighten the four screws. Do not over-tighten.

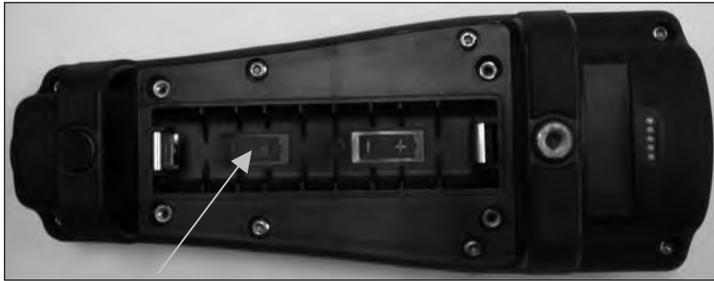


Figure 1. Pro30 with battery cover removed. Notice battery symbols indicating polarities.

**i** The waterproof instrument case is sealed at the factory and is not to be opened, except by authorized service technicians. Do not attempt to separate the two halves of the instrument case as this may damage the instrument, break the waterproof seal, and will void the warranty.

## KEY PAD

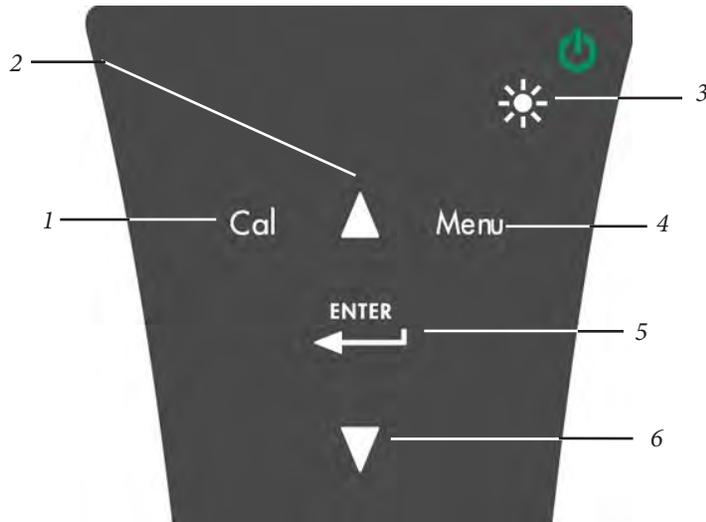


Figure 2, Keypad

Number	Key	Description
1		<b>Calibrate</b> Press and hold for 3 seconds to calibrate. Opens Calibrate menu from the Run screen.
2		<b>Up Arrow</b> Use to navigate through menus, to navigate through box options along the bottom of the Run screen and to increase numerical inputs.
3		<b>Power and Backlight</b> Press once to turn instrument on. Press a second time to turn backlight on. Press a third time to turn backlight off. Press and hold for 3 seconds to turn instrument off.
4		<b>Menu</b> Use to enter the System Setup menu from the Run screen.
5		<b>Enter</b> Press to confirm entries and selections.
6		<b>Down Arrow</b> Use to navigate through menus, to navigate through box options at the bottom of the Run screen and to decrease numerical inputs.

## CONNECTING THE PROBE/CABLE ASSEMBLY TO THE INSTRUMENT

The conductivity and temperature sensors are integral to the cable assembly; therefore, they cannot be removed from the cable.

To connect the cable, align the keys on the cable connector to the slots on the instrument connector. Push together firmly and then twist the outer ring until it locks into place (figure 3). This connection is water-proof.



Figure 3, Note the keyed connector.

## RUN SCREEN

Press the power/backlight key  to turn the instrument on. The instrument will run through a self test and briefly display a splash screen with system information before displaying the main Run screen (figure 4). The first time the Pro30 is turned on, it will prompt you to select a language; see the First Power On section of this manual for more information.

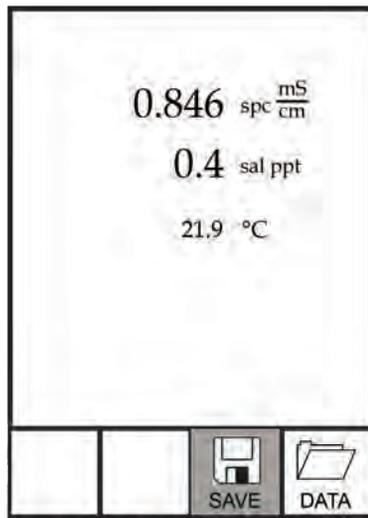


Figure 4, Run screen.

## BACKLIGHT

Once the instrument is powered on, pressing the power/backlight key  will turn on the display backlight. The backlight will remain on until the key is pressed again or after two minutes of not pressing any key on the keypad.

## POWERING OFF

To turn the instrument off, press and hold the power/backlight key  for three seconds.

## NAVIGATION

The up  and down  arrow keys allow you to navigate through the functions of the Pro30.

### NAVIGATING THE RUN SCREEN

When in the Run screen, the up  and down  arrow keys will move the highlighted box along the bottom options. Once a box is highlighted, press enter to access the highlighted option.

Description of Run screen box functions from left to right:

Option	Description
 SAVE	Highlight and press enter to save displayed data to memory.
 DATA	Highlight and press enter to view and/or erase saved data.

### NAVIGATING THE SYSTEM SETUP MENU

When in the System Setup menu, the up and down arrow keys will move the highlighted bar up and down the system setup options. See the System Setup menu section of this manual for more information about these options.

## FIRST POWER ON

The instrument will step through an initial language configuration when powered on for the first time. Use the up or down arrow keys to highlight the

appropriate language then press enter to confirm (figure 5). If an incorrect language is selected, it may be changed in the System Setup menu.

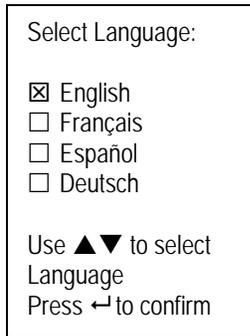


Figure 5, Select language.

After selecting a language, the Run screen will be displayed. The next time the instrument is powered up, the Run screen will display immediately after the splash screen.

## SYSTEM SETUP MENU

---

Press the menu  key to access the System Setup menu. The System Setup menu contains multiple screens that are notated as 'pages'. The current page is indicated near the bottom of the display (figure 6).

Use the up and down arrow keys to scroll through menu options and menu pages.

### EXITING THE SYSTEM SETUP MENU

---

To exit the System Setup menu, press the down arrow key until the ESC - Exit box is highlighted, then press enter to return to the Run screen.

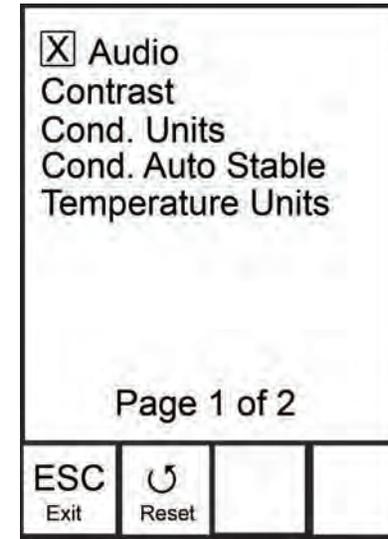


Figure 6, page 1 of System Setup menu. Audio is enabled.

## AUDIO

---

Audio can be enabled or disabled by using the up or down arrow keys to highlight Audio and pressing enter. When enabled, there will be an 'X' in the box next to Audio.

When Audio is enabled, the Pro30 will beep twice to indicate stability when Auto Stable is enabled. The instrument will also beep when a key is pressed. When Audio is disabled, the Pro30 will not beep.

## CONTRAST

---

To adjust the display Contrast, use the up or down arrow keys to highlight Contrast, then press enter. Next, use the up or down arrow keys to adjust the contrast. The up arrow key will darken the contrast and the down arrow key will lighten the contrast. After adjusting the contrast, press enter to save and exit the Contrast adjustment option.

### EMERGENCY CONTRAST ADJUSTMENT

---

If necessary, there is an alternate method of adjusting the contrast. To adjust the contrast, press and hold the menu key, then press the up arrow key to darken the contrast or press the down arrow key to lighten the contrast.

## CONDUCTIVITY UNITS (COND. UNITS)

---

Highlight Cond. Units (Conductivity Units) and press enter to open a submenu that allows you to select the conductivity units to be displayed on the Run screen. Highlight a unit and press enter to enable or disable it. An enabled conductivity unit will have an 'X' in the box next to it. Highlight the ESC-Exit box along the bottom of the display and press enter to save any changes and to close the conductivity units submenu.

There are seven options for displaying conductivity. Only four units can be enabled at the same time:

- COND-mS/cm displays conductivity in milliSiemens per centimeter.
- COND-uS/cm displays conductivity in microSiemens per centimeter.
- SPC-mS/cm displays Specific Conductance in milliSiemens per centimeter. Specific Conductance is temperature compensated conductivity.
- SPC-uS/cm displays Specific Conductance in microSiemens per centimeter. Specific Conductance is temperature compensated conductivity.
- Sal ppt displays salinity in parts per thousand. The salinity reading is calculated from the instrument's conductivity and temperature values using algorithms found in *Standard Methods for the Examination of Water and Wastewater*.
- TDS g/L displays Total Dissolved Solids in grams per liter. TDS is calculated from conductivity and temperature using a user-selectable TDS constant.
- TDS mg/L displays Total Dissolved Solids in milligrams per liter. TDS is calculated from conductivity and temperature using a user-selectable TDS constant.

Note: 1 milliSiemen = 1,000 microSiemens.

---

## SPECIFIC CONDUCTANCE

---

The conductivity of a sample is highly dependent on temperature, varying as much as 3% for each change of one degree Celsius (temperature coefficient = 3%/°C). In addition, the temperature coefficient itself varies with the nature of the ionic species present in the sample. Therefore, it is useful to compensate for this temperature dependence in order to quickly compare conductivity readings taken at different temperatures.

The Pro30 can display non-temperature compensated conductivity as well as temperature compensated Specific Conductance. If Specific Conductance is selected, the Pro30 uses the temperature and conductivity values associated with

each measurement to calculate a specific conductance value compensated to a user selected reference temperature, see below. Additionally, the user can select the temperature coefficient from 0% to 4%.

Using the Pro30's default reference temperature and temperature coefficient (25 °C and 1.91%), the calculation is carried out as follows:

$$\text{Specific Conductance (25°C)} = \frac{\text{Conductivity of sample}}{1 + 0.0191 * (T - 25)}$$

T = Temperature of the sample in °C

## CONDUCTIVITY AUTO STABLE (COND. AUTO STABLE)

---

Auto Stable utilizes preset values to indicate when a reading is stable. The preset values are adjustable in the System Setup menu. The user can input a % change in readings (0.0 to 1.9) over 'x' amount of time in seconds (3-19).

Highlight Cond. Auto Stable, then press enter to open the submenu.

Use the up or down arrow keys to highlight the % change or seconds (secs) input field, then press enter to make the highlighted field adjustable. Use the up or down arrow keys to adjust the selected value, then press enter to confirm changes. Once you have confirmed any changes, highlight the ESC-Exit box along the bottom of the display and press enter to close the Auto Stable submenu.

To disable Auto Stable, set the % Change input to 0.0.

When Auto Stable is enabled, an  $\text{AS}$  symbol will display next to the reading on the Run screen and blink during stabilization. When the dissolved oxygen and/or conductivity reading stabilizes based on the Auto Stable settings, the  $\text{AS}$  symbol will display steadily and the instrument will beep twice if Audio is turned on.

---

## TEMPERATURE UNITS

---

Highlight Temperature Units and press enter to open a submenu that allows you to change the temperature units displayed on the Run screen. Highlight the desired unit (Celsius or Fahrenheit) and press enter to enable. The enabled temperature unit will have an 'X' in the box next to it. Only one unit may be enabled at a time. Highlight the ESC-Exit box and press enter to save any changes and to close the Temperature Units submenu.

## **SPECIFIC CONDUCTANCE REFERENCE TEMPERATURE (SPC REF. TEMP.)**

---

SPC Ref. Temp. (Specific Conductance Reference Temperature) is the reference temperature used to calculate Specific Conductance. The reference temperature range is 15 and 25 °C. The default value is 25 °C.

To change the reference temperature, highlight SPC Ref. Temp. and press enter to open the submenu. With the reference temperature highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. Press enter to save the new reference temperature. Next, highlight the ESC-Exit box and press enter to close the submenu.

## **SPECIFIC CONDUCTANCE TEMPERATURE COEFFICIENT (SPC %/°C)**

---

SPC %/°C (Specific Conductance Temperature Coefficient) is the temperature coefficient used to calculate Specific Conductance. The coefficient range is 0.00 to 4.00. The default value is 1.91% which is based on KCl standards.

To change the temperature coefficient, highlight SPC %/°C and press enter to open the submenu. With the temperature coefficient highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. Press enter to save the new coefficient. Next, highlight the ESC-Exit box and press enter to close the submenu.

## **TDS CONSTANT**

---

TDS Constant is a multiplier used to calculate an estimated TDS (Total Dissolved Solids) value from conductivity. The multiplier is used to convert Specific Conductance in mS/cm to TDS in g/L. The Pro30's default value is 0.65. This multiplier is highly dependent on the nature of the ionic species present in the water sample. To be assured of moderate accuracy for the conversion, you must determine a multiplier for the water at your sampling site. Use the following procedure to determine the multiplier for a specific sample:

1. Determine the specific conductance of a water sample from the site;
2. Filter a sample of water from the site;
3. Completely evaporate the water from a carefully measured volume of the filtered sample to yield a dry solid;
4. Accurately weigh the remaining solid;
5. Divide the weight of the solid (in grams) by the volume of water used (in liters) to yield the TDS value in g/L for this site;
6. Divide the TDS value in g/L by the specific conductance of the water in mS/cm to yield the conversion multiplier. Be certain to use the correct units.

If the nature of the ionic species at the site changes between sampling studies, the TDS values will be in error. TDS cannot be calculated accurately from specific conductance unless the make-up of the chemical species in the water remains constant.

To change the TDS Constant in the Pro30, highlight TDS Constant and press enter to open the submenu. With the TDS Constant highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. The input range is 0.30 to 1.00. Press enter to save the new TDS Constant. Next, highlight the ESC-Exit box and press enter to close the submenu.

## **LANGUAGE**

---

Highlight Language and press enter to open a submenu that allows you to change the language. Highlight the desired language (English, Spanish, German, or French) and press enter to enable. The enabled language will have an 'X' in the box next to it. Highlight ESC-Exit box and press enter to save any changes and to close the Language submenu.

The text in the boxes along the bottom of the Run screen will always be displayed in English regardless of the language enabled in the System Setup menu.

## **AUTO SHUTOFF**

---

Auto Shutoff allows you to set the instrument to turn off automatically after a period of time. Use the up or down arrow keys to highlight Auto Shutoff, then press enter to open the submenu. Press enter while the minute field is highlighted to make it adjustable. Next, use the up or down arrow keys to adjust the shut off time from 0 to 60 minutes. Press enter to save the new shutoff time. Next, highlight the ESC-Exit box and press enter to close the submenu.

To disable Auto Shutoff, set the Time in Minutes to 0 (zero).

## **CELL CONSTANT**

---

The Cell Constant displays the cell constant of the conductivity cell. The cell constant is calculated and updated each time a conductivity calibration is performed. The cell constant range is 4.0 to 6.0. Resetting the System Menu resets the cell constant to 5.0.

## RESETTING THE SYSTEM SETUP MENU TO FACTORY DEFAULT

To reset the Pro30 settings to factory default, press the down arrow key while in the System Setup menu until the Reset -  box is highlighted, then press enter. The instrument will ask you to confirm the reset. Highlight Yes and press enter to continue with the reset or highlight No and press enter to cancel the reset. A Factory Reset will not affect data saved in the instrument's memory.

The following will be set in the Pro30 after performing a reset:

<i>Parameter</i>	<i>Reset Defaults</i>
Audio	On
Contrast	Set to mid range
Conductivity Units	cond uS/cm, spc mS/cm, spc uS/cm and sal ppt
Conductivity Auto Stable	Off (0.0 % Change and 10 seconds)
SPC Reference Temperature	25°C
SPC Temperature Coefficient	1.91%/°C
TDS Constant	0.65
Temperature Units	°C
Language	English
Auto Shutoff	30 minutes
Conductivity Cell Constant	Cell constant reset to 5.0*

\*It is recommended to perform a Conductivity calibration after performing a reset.

## CALIBRATION

### TEMPERATURE

All Pro30 cables have built-in temperature sensors. Temperature calibration is not required nor is it available.

### CONDUCTIVITY CALIBRATION

Ensure the conductivity sensor is clean and dry before performing a conductivity, specific conductance or salinity calibration.



*It is not necessary to calibrate conductivity, specific conductance and salinity. Calibrating one of these parameters will simultaneously calibrate the others. YSI recommends calibrating specific conductance for greatest ease.*

### CALIBRATING SPECIFIC (SP.) CONDUCTANCE OR CONDUCTIVITY

Note: When calibrating Specific Conductance, the Pro30 uses the factory default values for the Specific Conductance Reference Temperature and the Specific Conductance Temperature Coefficient regardless of what is configured in the System Setup Menu. The default value for the Reference Temperature is 25°C and the default value for the Temperature Coefficient is 1.91%/°C. It is important to note that the Temperature Coefficient of a calibration solution is dependent on the contents of the solution. Therefore, YSI recommends using a traceable calibration solution made of KCl (potassium chloride) when calibrating Specific Conductance since these solutions typically have a Temperature Coefficient of 1.91%/°C. Additionally, be sure to enter the value of the solution as it is listed for 25°C when calibrating Specific Conductance.

1. Fill a clean container (i.e. plastic cup or glass beaker) with fresh, traceable conductivity calibration solution and place the sensor into the solution. The solution must cover the holes of the conductivity sensor that are closest to the cable (figure 7). Ensure the entire conductivity sensor is submerged in the solution or the instrument will read approximately half the expected value. Gently move the probe up and down to remove any air bubbles from the conductivity sensor.

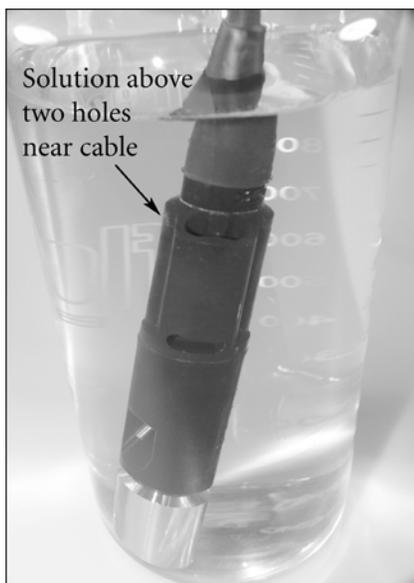


Figure 7, solution above two holes near cable.

2. Turn the instrument on and allow the conductivity and temperature readings to stabilize. Press and hold the Cal key for 3 seconds. Highlight Conductivity and press enter. Next, highlight the desired calibration method, Sp. Conductance or Conductivity, and press enter.
3. Highlight the units you wish to calibrate, either uS/cm or mS/cm, and press enter. 1 mS = 1,000 uS. Next, use the up or down arrow key to adjust the value on the display to match the value of the conductivity calibration solution. If calibrating conductivity, it is necessary to look up the value of the solution at the current temperature and enter that value into the Pro30. Most conductivity solutions are labeled with a value at 25°C. If calibrating specific conductance, enter the value listed for 25°C. Depressing either the up or down arrow key for 5 seconds will move the changing digit one place to the left. The Pro30 will remember the entered calibration value and display it the next time a conductivity calibration is performed.
4. Press enter to complete the calibration. Or, press Cal to cancel the calibration and return to the Run screen.
5. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
6. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting guide for possible solutions.

---

## CALIBRATING IN SALINITY

---

1. Fill a clean container (i.e. plastic cup or glass beaker) with fresh, traceable salinity calibration solution and place the sensor into the solution. The solution must cover the holes of the conductivity sensor that are closest to the cable (figure 7). Ensure the entire conductivity sensor is submerged in the solution or the instrument will read approximately half the expected value. Gently move the probe up and down to remove any air bubbles from the conductivity sensor.
2. Turn the instrument on and allow the conductivity and temperature readings to stabilize. Press and hold the Cal key for 3 seconds. Highlight Conductivity and press enter. Next, highlight Salinity and press enter.
3. Use the up or down arrow key to adjust the value on the display to match the value of the salinity solution. Depressing either the up or down arrow key for 5 seconds will move the changing digit one place to the left. The Pro30 will remember the entered calibration value and display it the next time a salinity calibration is performed.
4. Press enter to complete the calibration. Or, press Cal to cancel the calibration and return to the Run screen.
5. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
6. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting guide for possible solutions.

---

## TAKING MEASUREMENTS

---

Before taking measurements, be sure the instrument has been calibrated to ensure the most accurate readings. Place the probe in the sample to be measured and give the probe a quick shake to release any air bubbles. Be sure the conductivity sensor is completely submerged in the sample. The two holes near the cable should be covered by the sample for accurate conductivity readings (figure 7). Allow the temperature readings to stabilize.

---

## SAVING AND VIEWING DATA

---

The Pro30 can store 50 data sets in non-volatile memory for later viewing. A data set includes the values currently on the display, i.e. temperature, dissolved oxygen and two conductivity parameters. Each data point is referenced with a data set number, 01 through 50.

## SAVING DATA



The Pro30 can not communicate to a PC via a Pro Series communications saddle. Connecting the Pro30 to a communication saddle may cause erratic instrument behavior.

From the Run screen, use the up or down arrow keys to highlight the Save box and press enter to save the current readings. The instrument will indicate the data set is saved and display the saved data set's number (figure 8).

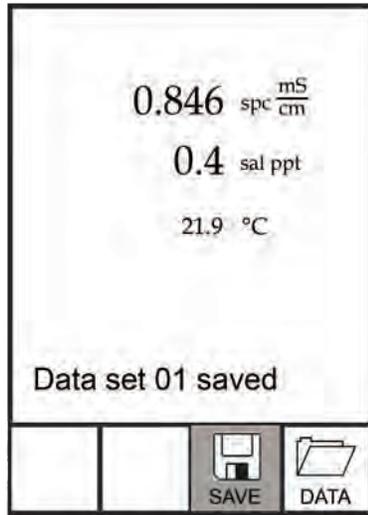


Figure 8, data set saved.

The instrument will display 'Memory Full' if all 50 data sets have been saved and you attempt to save another data set.

## VIEWING AND ERASING SAVED DATA - DATA MODE

Data mode allows you to view and erase saved data. From the Run screen, use the up or down arrow keys to highlight Data and press enter to access Data mode. Note that the function boxes at the bottom of the display are different in Data mode (figure 9).

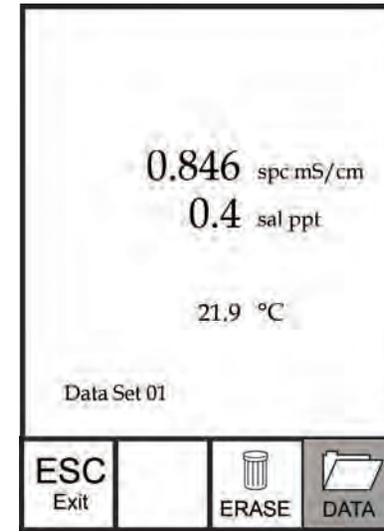


Figure 9, Data mode.

## VIEWING DATA

Once in Data mode, use the up and down arrow keys to view saved data sets in sequential order or press enter to access the bottom functions. After accessing the bottom functions, highlight the Data box and press enter to regain access to viewing data. The data set displayed is indicated by the data set number, 01 through 50.

## ERASING DATA

While viewing saved data, press the enter key to access the function boxes at the bottom of the display. Next, use the up or down arrow keys to highlight Erase, then press enter. The instrument will give you the option to erase one data set or all data sets (figure 10).



Figure 10, Erase data mode.

Use the up or down arrow key to select Erase Data Set, Erase All Sets or the ESC-Exit function box, then press enter to confirm.

Select ESC-Exit and press enter to exit Erase mode without erasing any data.

Select Erase Data Set and press enter to erase the data set that was displayed before entering Erase mode. For example, if data set 12 was displayed before entering erase mode, and Erase Data Set is selected, Data Set 12 will be erased from memory and the data sets AFTER that number will move up to keep them sequential. For example, if there are 15 records and number 12 is erased then 13 becomes 12, 14 becomes 13, and 15 becomes 14. The instrument will return to Data mode after erasing one data set.

Select Erase All Data Sets and press enter to clear the Pro30 memory and return to Data mode.

---

#### EXITING DATA MODE

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While in Data mode, press enter to access the bottom functions. Next, highlight the ESC-Exit box and press enter to return to the Run screen.

## CARE, MAINTENANCE AND STORAGE

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This section describes the proper procedures for care, maintenance and storage of the instrument. The goal is to maximize their lifetime and minimize downtime associated with improper instrument usage.

### GENERAL MAINTENANCE

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#### GENERAL MAINTENANCE - GASKET

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The instrument utilizes a gasket as a seal to prevent water from entering the battery compartment. Following the recommended procedures will help keep the instrument functioning properly.

If the gasket and sealing surfaces are not maintained properly, it is possible that water can enter the battery compartment. If water enters this area, it can severely damage the battery terminals causing loss of battery power and corrosion to the battery terminals. Therefore, when the battery compartment lid is removed, the gasket that provides the seal should be carefully inspected for contamination (i.e. debris, grit, etc.) and cleaned with water and mild detergent if necessary.

### SENSOR MAINTENANCE

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#### SENSOR MAINTENANCE - TEMPERATURE

---

You must keep the temperature sensor free of build up. Other than that, no additional maintenance is required. A toothbrush can be used to scrub the temperature sensor if needed.

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#### SENSOR MAINTENANCE - CONDUCTIVITY

---

The openings that allow sample access to the conductivity electrodes should be cleaned regularly. The small cleaning brush included in the Maintenance Kit is intended for this purpose. Dip the brush in clean water and insert it into each hole 10 to 12 times. In the event that deposits have formed on the electrodes, it may be necessary to use a mild detergent (laboratory grade soap or bathroom foaming tile cleaner) with the brush. Rinse thoroughly with clean water, then check the response and accuracy of the conductivity cell with a calibration solution.

## SENSOR STORAGE

### SHORT AND LONG TERM STORAGE

For both short and long term storage, the conductivity sensor should be stored clean and dry.

Remove the batteries from the instrument when storing it for long periods of time (>30 days).

Long Term Storage Temperature: -5 to 70°C (23 to 158°F)

## TROUBLESHOOTING

<i>Symptom</i>	<i>Possible Solution</i>
Instrument will not turn on, a battery symbol appears, or “Critical Shutdown” displays on the screen.	<ol style="list-style-type: none"> <li>1. Low battery voltage, replace batteries.</li> <li>2. Batteries installed incorrectly, check battery polarity.</li> <li>3. Return system for service.</li> </ol>
Temperature values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Sample temperature is less than -5° C or more than +55°C. Increase or decrease the sample temperature to bring within the allowable range.</li> <li>2. Contact YSI Tech Support.</li> </ol>
Instrument will not calibrate the Conductivity sensor; instrument displays “Calibration Over”, “Calibration Under”, or “Unstable Reading” during calibration.	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the calibration solution is above the two holes near the cable, see figure 8.</li> <li>3. Verify the calibration solution is not expired or contaminated. Try a new bottle of solution.</li> <li>4. Ensure you are entering in the correct value for the solution according to the measurement units. 1 mS = 1,000 uS.</li> <li>5. Allow sufficient stabilization time for conductivity and temperature AND wait at least 3 seconds before confirming a calibration.</li> <li>6. Contact YSI Tech Support.</li> </ol>

<i>Symptom</i>	<i>Possible Solution</i>
Conductivity readings are inaccurate.	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the sample is above the two holes near the cable, see figure 8.</li> <li>3. Verify calibration.</li> <li>4. Verify temperature readings are accurate.</li> <li>5. Verify the correct units are setup in the System Setup menu, i.e. uS vs mS and Conductivity vs. Specific Conductance.</li> <li>6. Contact YSI Tech Support.</li> </ol>
Conductivity values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the sample is above the two holes near the cable, see figure 8</li> <li>3. Verify calibration.</li> <li>4. Verify temperature readings are accurate.</li> <li>5. Sample conductivity is outside the measurement range of the instrument, i.e. 0-200 mS.</li> <li>6. Contact YSI Tech Support.</li> </ol>

## SPECIFICATIONS

These specifications represent typical performance and are subject to change without notice. For the latest product specification information, please visit YSI's website at [www.ysi.com](http://www.ysi.com) or contact YSI Tech Support.

<i>Parameter</i>	<i>Range</i>	<i>Resolution</i>	<i>Accuracy</i>
Temperature	-5 to 55°C	0.1°C	± 0.2°C
Conductivity	0-500 uS/cm 0-5 mS/cm 0-50 mS/cm 0-200 mS/cm (auto ranging)	0.0001 to 0.1 mS/cm; 0.1 to 0 uS/cm (range dependent)	Instrument only: ± 0.5% of the reading or 1 uS/cm, whichever is greater. Instrument with 1 or 4 meter cables: ± 1.0% of the reading or 1 uS/cm, whichever is greater. Instrument with 10, 20, or 30 meter cables: ± 2.0% of the reading or 1 uS/cm, whichever is greater.
Salinity	0 to 70 ppt	0.1 ppt	± 1.0% of the reading or ± 0.1 ppt, whichever is greater.
Total Dissolved Solids (TDS)	0 to 100 g/L. TDS Constant range: 0.3 to 1.00 (0.65 default)	0.0001 to 0.1 g/L (range dependent)	Dependent on accuracy of temperature, conductivity and TDS Constant.

## ACCESSORIES / PART NUMBERS

<i>Part Number</i>	<i>Description</i>
6050030	Pro30 Instrument
60530-1, -4, -10, -20, or -30	1, 4, 10, 20, 30-meter cable assembly*
603077	Flow cell
603056	Flow cell mounting spike
603075	Carrying case, soft-sided
603074	Carrying case, hard-sided
603069	Belt clip
063517	Ultra clamp for instrument
063507	Tripod for instrument
603062	Cable management kit, included with all cables longer than 1 meter.
605978	Cable weight, 4.9 oz, stackable
603070	Shoulder strap
060907	Conductivity Calibration Solution, 1,000 µS/cm. 1 box of 8 pints.
060911	Conductivity Calibration Solution, 10,000 µS/cm. 1 box of 8 pints.
060660	Conductivity Calibration Solution, 50,000 µS/cm. 1 box of 8 pints.
065274	Conductivity Calibration Solution, 100,000 µS/cm. 1 box of 8 pints.

\*All cables include a temperature and conductivity sensor.

# DECLARATION OF CONFORMITY

The undersigned hereby declares on behalf of the named manufacturer under our sole responsibility that the listed product conforms to the requirements for the listed European Council Directive(s) and carries the CE mark accordingly.

<i>Manufacturer:</i>	YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA
<i>Product Name:</i>	Pro30 Water Quality Instrument
<i>Model Numbers</i>	
<i>Instrument/Accessory:</i>	Pro30 (6050030)
<i>Probe/Cable Assemblies:</i>	60530-1, -4, -10, -20, and -30
<i>Conforms to the following:</i>	
<i>Directives:</i>	IEC 61326-1:2005 RoHS 2002/95/EC WEEE 2002/96/EC IP-67 Protection per ANSI/IEC 60529-2004
<i>Harmonized Standards:</i>	<ul style="list-style-type: none"> <li>EN61326-1:2006 (IEC 61326-1:2005) Basic Immunity</li> </ul>
<i>Supplementary Information:</i>	All performance met the operation criteria as follows: 1. ESD, IEC 61000-4-2:2001, Performance Criterion B 2. Radiated Immunity, IEC 61000-4-3, Performance Criterion A 3. Electrical Fast Transient (EFT), IEC 61000-4-4:2004, +Corr. 1:2006 + Corr. 2:2007, Performance Criterion B 4. Radio Frequency, Continuous Conducted Immunity, IEC61000-4-6, Performance Criterion A 5. Radiated Emissions, EN 61326-1:2006 (IEC61326-1:2005) Class B
<i>Authorized EU Representative</i>	YSI Hydrodata Ltd Unit 2 Focal Point, Lacerta Court, Works Road Letchworth, Hertfordshire, SG6 1FJ UK



Signed: Lisa M. Abel  
Title: Director of Quality

Date: 27 June 2011

# RECYCLING

YSI is committed to reducing the environmental footprint in the course of doing business. Even though materials reduction is the ultimate goal, we know there must be a concerted effort to responsibly deal with materials after they've served a long, productive life-cycle. YSI's recycling program ensures that old equipment is processed in an environmentally friendly way, reducing the amount of materials going to landfills.

- Printed Circuit Boards are sent to facilities that process and reclaim as much material for recycling as possible.
- Plastics enter a material recycling process and are not incinerated or sent to landfills.
- Batteries are removed and sent to battery recyclers for dedicated metals.

When the time comes for you to recycle, follow the easy steps outlined at [www.yisi.com](http://www.yisi.com).

## BATTERY DISPOSAL

The Pro30 is powered by alkaline batteries which the user must remove and dispose of when the batteries no longer power the instrument. Disposal requirements vary by country and region, and users are expected to understand and follow the battery disposal requirements for their specific locale.

# CONTACT INFORMATION

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## ORDERING AND TECHNICAL SUPPORT

---

Telephone: 800 897 4151 (USA)  
+1 937 767 7241 (Globally)  
Monday through Friday, 8:00 AM to 5:00 ET

Fax: +1 937 767 9353 (orders)  
+1 937 767 1058 (technical support)

Email: environmental@ysi.com  
Mail: YSI Incorporated  
1725 Brannum Lane  
Yellow Springs, OH 45387 USA

Internet: www.ysi.com

When placing an order please have the following available:

- 1.) YSI account number (if available)
- 2.) Name and phone number
- 3.) Purchase Order or Credit Card number
- 4.) Model Number or brief description
- 5.) Billing and shipping addresses
- 6.) Quantity

## SERVICE INFORMATION

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YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit [www.ysi.com](http://www.ysi.com) and click 'Support' or contact YSI Technical Support directly at 800-897-4151 (+1 937-767-7241).

When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for a YSI Service Center to accept the instrument for service. The form may be downloaded from [www.ysi.com](http://www.ysi.com) by clicking on the 'Support'.

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Rev A  
Drawing # A606082  
July 2011

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# Operations Manual EcoSense® EC300A

Portable  
Conductivity, Salinity  
and Temperature  
Instrument



- English
- Français
- Español
- Português



## **WARRANTY**

The EcoSense® EC300A Instrument is warranted for one year from date of purchase by the end user against defects in materials and workmanship. EC300A probes and cables are warranted for one year from date of purchase by the end user against defects in material and workmanship. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

### **Limitation of Warranty**

This Warranty does not apply to any YSI product damage or failure caused by: (i) failure to install, operate or use the product in accordance with YSI's written instructions; (ii) abuse or misuse of the product; (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure; (iv) any improper repairs to the product; (v) use by you of defective or improper components or parts in servicing or repairing the product; or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI's LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

## **CONTACT INFORMATION**

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1725 Brannum Lane  
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Tel: 800-897-4151; 937-767-7241  
Fax: 937-767-1058  
Email: [environmental@ysi.com](mailto:environmental@ysi.com)  
Website: [www.ysi.com](http://www.ysi.com)

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## GENERAL INTRODUCTION

The EC300A is a precision tool that measures conductivity, salinity and temperature. A built-in microprocessor calculates and compensates for all parameters related to conductivity and temperature determinations.

This instrument is waterproof (IP67) when the connector cap is installed. The mechanical touch keys are highly reliable with tactile and audio feedback. This instrument uses one 9V battery. Re-calibration is not required when power is restored.

The front of the instrument has a large LCD that displays temperature and either temperature compensated or non-temperature compensated conductivity, salinity or TDS simultaneously along with user prompts and mode indicators. The unit prompts users through calibration and measurement procedures.

The model EC300A is available with a single four-electrode cell. Other features include automatic conductivity ranging, automatic temperature compensation, long battery life, and 50/60 Hz AC noise rejection. This meter is universal and user-friendly for field, industrial and laboratory applications.

## INITIAL INSPECTION

Carefully unpack the unit and accessories, and inspect for shipping damages. Compare received parts with materials listed on the packing list. Notify YSI immediately of any damage or missing parts. Save all packing materials until satisfactory operation is confirmed.

## THE INSTRUMENT

Though the instrument is housed in a water-proof IP67 case, DO NOT use it underwater. The connector is not waterproof unless the cap is installed. In case of submersion without the cap connected, follow these steps immediately:

1. Dry the connector if necessary, and replace the conductivity probe. Rinse unit carefully with distilled water. After rinsing and drying, inspect and clean connectors to remove all contaminants that may affect probe connections.
2. Wait for the unit and probe to dry completely before resuming operation.
3. If the unit does not function correctly after steps 1 and 2, call YSI for possible repair or replacement (see Warranty).

## BATTERY INSTALLATION

An initial display of "BAT" on the LCD indicates approximately one hour of battery life for unit operation within specifications. Replace battery when "BAT" appears on the LCD. (See Figure 1.)

To replace battery, remove the two battery cover screws, battery cover and o-ring. Replace the 9V battery. Replace battery cover and o-ring (align the o-ring properly to insure a good seal) and fasten the two battery cover screws for the splash-resistant feature.

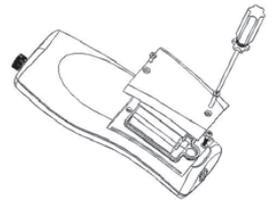


Figure 1.  
Battery Installation

## **Battery Disposal**

This instrument is powered by a 9 volt battery, which the user must remove and dispose of when the batteries no longer power the instrument. Disposal requirements vary by country and region, and users are expected to understand and follow the battery disposal requirements for their specific locale.

## KEY FUNCTIONS OF THE MODEL EC300A

1. **Power**: Turns the unit ON or OFF. Calibration values are not erased when the unit is turned off. When the unit is not in use, turn it off to save battery life. The instrument has a 30 minute auto shut off feature when not in use. For long-term storage, remove the battery.
2. **MODE**: Selects display mode. In Normal operation, press MODE to switch the display between uncompensated conductivity, temperature compensated conductivity, salinity, total dissolved solids (TDS), Recall and Delete. In calibration mode, this key exits the current calibration and displays the next calibration parameter.
3. **CAL**: In normal operation, changes the mode from Normal to Calibration.
4. **Enter**: In Calibration Set-up, press this key to save the current parameter to memory.
5. **Δ and ∇ Keys**: Increases or decreases the display value as desired.

## THE LCD DISPLAY

1. **CONDUCTIVITY**: Displays when measuring conductivity.
2. **BAT**: Low battery indicator.
3. **CELL**: Indicates conductivity cell constant value.
4. Main display for compensated and uncompensated conductivity, salinity and TDS values.
5. **TDS**: Displays when measuring total dissolved solids.

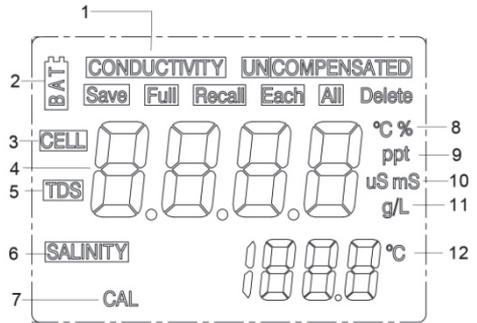


Figure 1. LCD Display

6. **SALINITY**: Displays when measuring salinity.
7. **CAL**: Calibration mode indicator.
8. **°C**: Flashes during temperature compensated conductivity measurement. During calibration, indicates temperature reference unit.
- %**: Displays during calibration; indicates temperature coefficient unit.
9. **ppt**: Parts per thousand; indicates salinity measurement.
10. **uS, mS**: micro Siemens, milli Siemens; Indicates conductivity measurement.
11. **g/L**: grams/Liter; indicates TDS measurement.
12. **°C**: Temperature display.

## MEASUREMENT MODES

1. **Temperature** - Current solution temperature continually displays.
2. **Temperature Compensated Conductivity** - Measurement of conductivity, compensated to 25°C or another specified value between 15 and 25°C. Expressed as uS/cm or mS/cm with a flashing "°C".
3. **Uncompensated Conductivity** - Direct measurement of conductivity, not compensated to a specific temperature. Expressed as uS/cm or mS/cm.
4. **Salinity** - Measurement of salinity; expressed in parts per thousand (ppt).

5. **TDS** - Measurement of total dissolved solids (TDS); expressed in grams per liter (g/L). Carefully observe the units displayed at the far side of the LCD to determine the desired mode.

## **CALIBRATION**

Calibration setup contains five sections: TDS, Cell, Temperature Coefficient, Temperature reference, and Conductivity Calibration. To access these sections:

1. Connect the conductivity probe and cable assembly to the unit and turn the unit on. The screen will display **CELL** and the cell constant of the conductivity probe.
2. Allow temperature readings to stabilize, then press **CAL** to enter the calibration mode; **CAL** appears on the LCD. Press **MODE** to sequentially display the following sections:

**Note:** Press Enter (  ) to accept any values changes in each section and automatically advance to the next section. If there are no changes, the unit accepts the current value and proceeds to the next section.

### **TDS**

TDS is determined by multiplying conductivity (mS) by a TDS factor. The default factor value is 0.65. To change the TDS factor, use the **Δ** and **∇** keys to adjust the value between 0.30 and 1.00. Press Enter (  ) to save the new value, or press **MODE** to cancel the change and display the **CELL** screen.

### **CELL**

The second screen will display **CELL** and the current cell value. The default cell value is 5.00 and is displayed in the lower right of the screen. The unit allows a variance of  $\pm 0.50$  before displaying an error message. The cell value cannot be adjusted at this screen; calibrating conductivity is the only way to adjust the cell constant. Press Enter (  ) to reset the cell constant to 5.00 and display the **Temperature Coefficient** screen.

**Note:** Be certain to press Enter (  ) to reset the cell constant to 5.00. If **MODE** is pressed, the unit retains the previous cell constant and calibrates from a value that is already offset.

### **Temperature Coefficient**

The unit uses the temperature coefficient to calculate temperature compensated conductivity. The default value is 1.91%. To change the temperature coefficient, use the **Δ** and **∇** keys to adjust the value between 0 and 4.00%. Press Enter (  ) to save the new value, or press **MODE** to cancel the change and display the **Temperature Reference** screen.

### **Temperature Reference**

The unit uses the temperature reference value to calculate temperature compensated conductivity. The default value is 25°C. To change the temperature coefficient, use the **Δ** and **∇** keys to adjust the value between 15 and 25°C. Press Enter (  ) to save the new value, or press **MODE** to cancel the change and display the **Conductivity Calibration** screen.

### **Conductivity Calibration**

1. Immerse the probe in a standard of known conductivity, preferably a standard in the middle range of the solutions to be measured. Completely submerge the probe without touching the sides of the calibration container. Shake the probe lightly to remove any air bubbles trapped in the conductivity cell.

2. Allow temperature to stabilize. The message 'rAng' (range) may display briefly to indicate unit auto-ranging; this is normal. After temperature stabilization, use the  $\Delta$  and  $\nabla$  keys to adjust the conductivity value to that of the conductivity standard at 25°C. Press Enter ( $\rightarrow$ ) to calibrate. The unit beeps twice to indicate a successful calibration, then automatically switches to normal operation mode.

## CONDUCTIVITY MEASUREMENTS

1. Turn the unit on. Place the probe in the solution to be measured. Completely submerge the probe. Shake the probe lightly to remove any trapped air bubbles in the conductivity cell.
2. Press **MODE** to enter the desired measurement mode. The message 'rAng' (range) may appear briefly on the display indicate auto-ranging; this is normal. Allow temperature to stabilize before taking measurements.

## SAVING, VIEWING AND DELETING DATA

The EC300A can save 50 data records. When in measurement mode, press  $\rightarrow$  to save a record. The instrument will confirm the saved data by displaying SAVE and the record number for one second. "Full" is displayed when trying to save data and the memory is full.

To view saved data, press mode until RECALL is displayed and then press  $\rightarrow$ . Use the Up or Down arrow keys to review different saved records. Press Mode to escape back to measurement mode.

To delete data records, press Mode while in measurement mode until DELETE is displayed. Press  $\rightarrow$ . "All" will be displayed and blinking. Press the Up or Down arrow key to switch between delete 'All' or 'Each' options. Select either 'All' or 'Each' by pressing  $\rightarrow$  while that option is displayed.

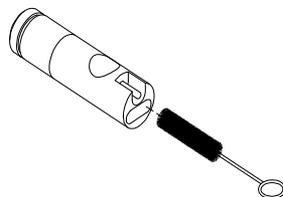
If 'All' is selected, all records will be deleted from memory and 'None' will be displayed. Press Mode twice to return to the measurement mode.

If 'Each' is selected, the Up and Down arrow keys will allow you to scroll through the saved data records. Press  $\rightarrow$  to delete the selected record. All records after the deleted record will shift up to keep the records in sequential order. For example, if record 3 is deleted, record 4 will become record 3 and record 5 will become record 4. Press Mode twice to return to the measurement mode.

## PROBE MAINTENANCE

The most important requirement for accurate and reproducible conductivity measurements is a clean cell. A dirty cell changes the conductivity of a solution through contamination. Clean the cell thoroughly before storing it. To clean the conductivity cell:

1. Dip the cell in cleaning solution and agitate for two to three minutes. Any foaming acid tile cleaner, such as Dow Chemical Bathroom Cleaner, should clean adequately. For a stronger cleaner, use a solution of 1:1 isopropyl alcohol and 1 N HCl. Remove the cell from the cleaning solution.
2. Use the nylon brush (supplied) to dislodge any contaminants from inside the electrode chamber.



Repeat steps one and two until the cell is completely clean. Rinse the cell thoroughly in deionized, or clean tap water.

## TROUBLESHOOTING

MAIN DISPLAY		PROBLEM	POSSIBLE SOLUTION
OvEr		<ul style="list-style-type: none"> <li>• Conductivity is &gt;200.0 mS</li> <li>• Salinity is &gt; 70.00 ppt</li> </ul>	<ul style="list-style-type: none"> <li>• Completely submerge the probe.</li> <li>• Allow sufficient time for the electrode and Temp probe stabilization.</li> </ul>
OvEr/Undr during calibration		<ul style="list-style-type: none"> <li>• Cell Constant Calibration is out of range</li> </ul>	<ul style="list-style-type: none"> <li>• Recalibrate with correct value for the conductivity standard.</li> <li>• Replace conductivity standard.</li> <li>• Clean cell.</li> <li>• Return for service.</li> </ul>
MAIN DISPLAY	SECONDARY DISPLAY		
OvEr/Undr	OvEr <hr/> Undr	Temperature >90.0 °C <hr/> Temperature < -10.0 °C	<ul style="list-style-type: none"> <li>• Decrease/Increase the sample temperature.</li> <li>• Return for service.</li> </ul>

## SPECIFICATIONS

Display	Range	Accuracy	Resolution
Conductivity, Auto-ranging	0.0 to 499.9 uS/cm 500 to 4999 uS/cm 5.00 to 49.99 mS/cm 50.0 to 200.0 mS/cm	±1% of reading plus 2 uS/cm ±1% of reading plus 5 uS/cm ±1% of reading plus 0.05 uS/cm ±2.5% of reading plus 0.5 mS/cm	0.01 uS/cm 1 mS/cm 0.01 mS/cm 0.1 mS/cm
Salinity	0.0 to 70.0 ppt	0.2% Full Scale	0.1 ppt
Temperature °C	-10.0 to 90 °C	±0.2 °C or ±0.4% Full Scale, whichever is greater	0.1 °C

Reference Temperature	15.0 to 25.0 °C
Temperature Coefficient	0.0% to 4.0%
Cell Constant	5.00 ± 0.50
TDS Constant Range	0.30 to 1.00
Power/Battery life	One 9V battery Approximately 500 hour
Calibration Back-up	Yes
Audio Feedback	Yes, on all touch keys
Instrument Case	Waterproof when connector cap installed, IP 67
Operating Temp. Range	0 to 50 °C
Operating Relative Humidity Range	up to 95%
Temperature Probe	Thermistor, 10kΩ / 25 °C
Dimensions (L x W x H)	18.7 cm x 7.6 cm x 3.8 cm (7.37 in x 3 in x 1.5 in)
Weight (batteries included)	270 grams (.6 lb)

## RECOMMENDED SPARE PARTS LIST

PART #	DESCRIPTION
300-1	1-meter probe and cable assembly
300-4	4-meter probe and cable assembly.
300-10	10-meter probe and cable assembly.
606043	Carrying case, hard sided.
485	Carrying case, soft sided.

Item #606042REF

Revision A, July 2012

For the latest version of this manual, visit [www.ysi.com](http://www.ysi.com)

## GARANTIE

L'appareil EcoSense® EC300A est garanti pour une période d'un an, à compter de la date d'achat par l'utilisateur final, contre tout défaut matériel et de fabrication. Les sondes et les câbles de l' EC300A sont garantis pour une période d'un an, à compter de la date d'achat par l'utilisateur final, contre tout défaut matériel et de fabrication. Pendant la période de garantie, YSI s'engage à réparer ou à remplacer, gratuitement et à sa discrétion, tout produit qu'YSI peut établir comme étant couvert par la garantie.

Pour faire valoir cette garantie, écrivez ou appelez votre représentant YSI ou contactez le Service clientèle d'YSI à Yellow Springs, Ohio, États-Unis. Envoyez le produit et son justificatif d'achat en port payé au Centre de service homologué sélectionné par YSI. La réparation ou le remplacement seront effectués et le produit vous sera retourné en port payé. Les produits réparés ou remplacés sont garantis jusqu'à expiration de la période de garantie originale ou pour au moins 90 jours, à compter de la date de réparation ou de remplacement.

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Cette garantie ne s'applique pas aux produits YSI endommagés ou présentant des dysfonctionnements pour les raisons suivantes : (i) installation, exploitation ou utilisation du produit d'une façon non conforme aux instructions écrites d'YSI ; (ii) abus ou mésusage du produit ; (iii) manquement à l'entretien du produit conformément aux instructions écrites d'YSI ou aux procédures industrielles normales ; (iv) réparation non conforme du produit ; (v) utilisation par vous de pièces ou de composants défectueux ou non conformes lors de l'entretien ou de la réparation du produit, ou ; (vi) modification du produit d'une façon non expressément autorisée par YSI.

CETTE GARANTIE REMPLACE TOUTES LES AUTRES GARANTIES, EXPRESSES OU INDUITES, Y COMPRIS LES GARANTIES DE COMMERCIALITÉ OU D'ADAPTATION À UN USAGE PARTICULIER. LA RESPONSABILITÉ D'YSI SELON LES TERMES DE CETTE GARANTIE SE LIMITE À LA RÉPARATION OU AU REMPLACEMENT DU PRODUIT, CONSTITUANT VOTRE SEUL ET UNIQUE RECOURS POUR TOUT PRODUIT DÉFECTUEUX COUVERT PAR CETTE GARANTIE. YSI NE POURRA EN AUCUN CAS ÊTRE TENU RESPONSABLE DE DOMMAGES SPÉCIAUX, INDIRECTS, ACCIDENTELS OU CONSÉCUTIFS RÉSULTANT DE L'UTILISATION DE TOUT PRODUIT DÉFECTUEUX COUVERT PAR CETTE GARANTIE.

## COMMENT NOUS CONTACTER

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## **INTRODUCTION GÉNÉRALE**

Le modèle EC300A est un outil de précision qui mesure la conductivité, la salinité et la température. Un microprocesseur incorporé calcule et compense tous les paramètres relatifs à la détermination de la conductivité et de la température.

Cet appareil est étanche (IP67) uniquement lorsque le capuchon recouvre le connecteur. Les touches mécaniques sont très fiables et produisent une réaction tactile et sonore. Cet appareil utilise une pile de 9 V. Aucun réétalonnage n'est nécessaire lorsque l'alimentation électrique est rétablie.

L'avant de l'instrument est muni d'un grand écran à cristaux liquides affichant la conductivité, compensée ou non par la température, la salinité ou le TSD (total des solides dissous) et, simultanément, des invites utilisateur et des indicateurs de mode. L'appareil émet des invites destinées à l'utilisateur lors des procédures d'étalonnage et de mesure.

Le modèle EC300A est disponible avec une cellule unique à quatre électrodes. Parmi les autres fonctionnalités, on notera le calcul automatique de la fourchette de conductivité, la compensation automatique de la température, la longue durée de vie des piles et une élimination du bruit de 50/60 Hz c. a. Cet appareil de mesure est convivial et particulièrement souple dans les applications sur le terrain, industrielles et en laboratoire.

## **INSPECTION INITIALE**

Déballer soigneusement l'appareil et les accessoires et vérifiez qu'ils n'ont pas été endommagés lors de l'expédition. Comparez les pièces reçues aux matériaux répertoriés dans le bordereau d'emballage. Notifiez immédiatement YSI s'il s'avère que des pièces sont endommagées ou manquantes. Mettez de côté les matériaux d'emballage jusqu'à ce que le fonctionnement correct de l'appareil soit confirmé.

## **L'APPAREIL**

L'appareil est en effet protégé par un boîtier étanche IP67, mais NE doit PAS être utilisé sous l'eau. Le connecteur n'est pas étanche, sauf si le capuchon le recouvre. En cas d'immersion sans capuchon, suivre immédiatement les étapes suivantes:

1. Séchez le connecteur, le cas échéant, et remplacez la sonde de conductivité. Rincez soigneusement l'appareil avec de l'eau distillée. Après le rinçage et le séchage, inspectez et nettoyez les connecteurs en vue d'éliminer tout contaminant pouvant affecter les connexions de la sonde.
2. Attendez que l'appareil et la sonde soient parfaitement secs avant de reprendre les opérations.
3. Si l'appareil ne fonctionne pas correctement après les étapes 1 et 2, appelez YSI en vue d'une réparation ou d'un remplacement éventuels (voir la Garantie).

## INSTALLATION DE LA PILE

Lorsque l'écran à cristaux liquides affiche pour la première fois « BAT », il reste environ une heure de fonctionnement sur pile selon les spécifications. Remplacez la pile lorsque l'indication « BAT » s'affiche sur l'écran. (Voir Figure 1.)

Pour remplacer la pile, enlevez les deux vis du compartiment ainsi que le couvercle et le joint torique. Remplacez la pile de 9 V. Remplacez le couvercle et le joint torique (alignez le joint correctement afin d'assurer une bonne étanchéité) et revissez les deux vis du compartiment pour conserver une bonne résistance aux éclaboussures.

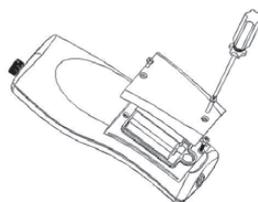


Figure 1.  
Installation de la pile

## Mise Au Rebut De La Piles

L'appareil est alimenté par de pile (9V) que l'utilisateur doit retirer et jeter lorsque la pile n'alimente plus l'appareil. Les exigences concernant la mise au rebut sont différentes en fonction du pays et de la région, et il est attendu de l'utilisateur qu'il comprenne et suive les règlements spécifiques à sa juridiction concernant la mise au rebut des piles.

## FONCTIONNALITÉS CLÉS DU MODÈLE EC300A

1.  Met l'appareil hors ou sous tension. Les valeurs d'étalonnage ne sont pas effacées lorsque l'appareil est mis hors tension. Lorsque l'appareil n'est pas utilisé, mettez-le hors tension pour économiser la pile. L'appareil s'éteint automatiquement s'il n'est pas utilisé après 30 minutes. Enlevez la pile pour un entreposage prolongé.
2. **MODE** : Permet de sélectionner le mode d'affichage. En service normal, appuyez sur MODE pour faire défiler l'affichage : conductivité non compensée, conductivité compensée par la température, salinité, TDS (total des solides dissous), Supprimer, Rappeler. En mode d'étalonnage, cette touche permet de quitter l'étalonnage actuel et d'afficher le paramètre d'étalonnage suivant.
3. **CAL** : En fonctionnement normal, passe du mode Normal au mode Étalonnage (Calibration).
4.  (Entrée) : Lors de la configuration de l'étalonnage, appuyez sur cette touche pour enregistrer le paramètre actuel en mémoire.
5. **Touches Δ et ∇** : Augmentent ou diminuent la valeur affichée, comme voulu.

## ÉCRAN À CRISTAUX LIQUIDES

1. **CONDUCTIVITY**: S'affiche lors de la mesure de la conductivité.
2. **BAT**: Indicateur de pile déchargée.
3. **CELL**: Indique la valeur constante de la cellule de conductivité.
4. Affichage principal des valeurs de conductivité compensée ou non, de salinité et de TSD.
5. **TDS**: S'affiche lors de la mesure du total des solides dissous (TSD).
6. **SALINITY** : S'affiche lors de la mesure de la salinité.

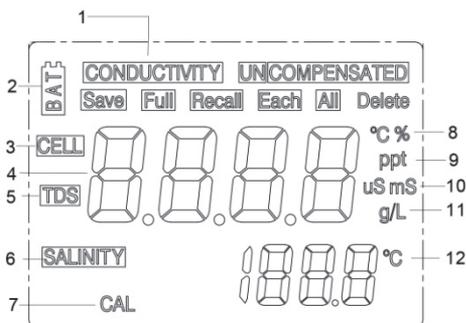


Figure 2. Écran à cristaux liquides

7. **CAL**: Indicateur de mode d'étalonnage (Calibration).
8. **°C** : Clignote lors de la mesure de la conductivité compensée par la température. Lors de l'étalonnage, indique l'unité de référence de la température.  
**%** : S'affiche lors de l'étalonnage ; indique l'unité du coefficient de la température.
9. **ppt** : Parties par millier ; indique la mesure de salinité.
10. **uS, mS** : microsiemens, millisiemens ; indique la mesure de conductivité.
11. **g/L** : Grammes/litre ; indique la mesure du TDS.
12. **°C** : Affichage de la température.

## MODES DE MESURE

1. **Température** – La température actuelle de la solution s'affiche constamment.
2. **Conductivité compensée par la température** – Mesure de conductivité, compensée de 25 °C ou d'une autre valeur spécifiée entre 15 et 25 °C. Exprimée en uS/cm ou mS/cm avec un « °C » clignotant.
3. **Conductivité non compensée** – Mesure directe de la conductivité, non compensée par une température spécifique. Exprimée en uS/cm ou mS/cm.
4. **Salinité** – Mesure de la salinité ; exprimée en parties par millier (ppt).
5. **TSD** – Mesure du total des solides dissous (TSD) ; exprimée en grammes par litre (g/L).

Observez soigneusement les unités affichées à l'extrémité de l'écran à cristaux liquides pour déterminer le mode voulu.

## ÉTALONNAGE

La configuration de l'étalonnage comporte cinq sections : étalonnage du TSD, de la cellule, du coefficient de la température, de la référence de la température et de la conductivité. Pour accéder à ces sections :

1. Connectez la sonde de conductivité et l'assemblage du câble à l'appareil et mettez l'appareil sous tension. L'écran affiche **CELL** et la constante de la cellule de la sonde de conductivité.
2. Laissez la lecture de température se stabiliser, puis appuyez sur **CAL** pour entrer en mode d'étalonnage ; la mention **CAL** s'affiche sur l'écran à cristaux liquides. Appuyez sur **MODE** pour afficher successivement les sections suivantes :

**Remarque** : appuyez sur Entrée (  ) pour accepter la modification des valeurs dans chacune des sections et avancer automatiquement à la section suivante. Si aucune modification n'est apportée, l'appareil accepte la valeur actuelle et passe à la section suivante.

### **TSD**

Le TSD est déterminé en multipliant la valeur de conductivité (mS) par un facteur de TSD. La valeur du facteur par défaut est de 0,65. Pour modifier la valeur du facteur de TSD, utilisez les touches  $\Delta$  et  $\nabla$  pour définir la valeur entre 0,30 et 1,00. Appuyez sur Entrée (  ) pour enregistrer la nouvelle valeur ou sur **MODE** pour annuler la modification et afficher l'écran **CELL**.

### **CELL**

Le deuxième écran affiche **CELL** et la valeur actuelle de la cellule. La valeur par défaut de la cellule, affichée dans la partie inférieure droite de l'écran, est de 5,00. L'appareil accepte une variation de  $\pm 0,50$  avant d'afficher un message d'erreur. La valeur de la cellule ne peut pas être réglée dans cet écran ; le seul moyen de régler la constante de la cellule est d'effectuer

un étalonnage de la conductivité. Appuyez sur Entrée (  ) pour rétablir la constante de la cellule à la valeur 5,00 et afficher l'écran **Coefficient de la température**.

**Remarque :** Veillez à bien appuyer sur la touche Entrée (  ) pour rétablir la constante de la cellule sur la valeur par défaut 5,00. Si vous appuyez sur la touche **MODE**, l'appareil retient la constante précédente de la cellule et effectue l'étalonnage d'après une valeur qui est déjà erronée.

## Coefficient de la température

L'appareil utilise le coefficient de la température pour calculer la conductivité compensée par la température. La valeur par défaut est de 1,91 %. Pour modifier la valeur du coefficient de la température, utilisez les touches **Δ** et **∇** pour définir la valeur entre 0 et 4,00 %. Appuyez sur Entrée (  ) pour enregistrer la nouvelle valeur ou sur **MODE** pour annuler la modification et afficher l'écran **Référence de la température**.

## Référence de la température

L'appareil utilise la valeur de référence de la température pour calculer la conductivité compensée par la température. La valeur par défaut est de 25 °C. Pour modifier la valeur de la référence de la température, utilisez les touches **Δ** et **∇** pour définir la valeur entre 15 et 25 °C. Appuyez sur Entrée (  ) pour enregistrer la nouvelle valeur ou sur **MODE** pour annuler la modification et afficher l'écran **Étalonnage de la conductivité**.

## Étalonnage de la conductivité

1. Plongez la sonde dans un standard de conductivité connue, se trouvant de préférence au milieu de la fourchette des solutions devant être mesurées. Submergez complètement la sonde sans toucher les parois du conteneur d'étalonnage. Agitez doucement la sonde pour enlever les bulles d'air piégées dans la cellule de conductivité.
2. Laissez la température se stabiliser. Le message « rAng » (fourchette) peut s'afficher brièvement pour indiquer que l'appareil calcule automatiquement la fourchette. Cela est normal. Une fois que la température est stabilisée, utilisez les touches **Δ** et **∇** pour régler la valeur de conductivité sur celle du standard de conductivité à 25 °C. Appuyez sur Entrée (  ) pour étalonner. L'appareil émet deux signaux sonores pour indiquer que l'étalonnage est réussi, puis passe automatiquement en mode de fonctionnement normal.

## MESURE DE LA CONDUCTIVITÉ

1. Mettez l'appareil sous tension. Placez la sonde dans la solution devant être mesurée. Submergez complètement la sonde. Agitez doucement la sonde pour enlever les bulles d'air piégées dans la cellule de conductivité.
2. Appuyez sur **MODE** pour entrer dans le mode de mesure voulu. Le message « rAng » (fourchette) peut s'afficher brièvement pour indiquer que l'appareil calcule automatiquement la fourchette. Cela est normal. Laissez la température se stabiliser avant d'effectuer des mesures.

## ENREGISTREMENT, AFFICHAGE ET SUPPRESSION DES DONNÉES

Le EC300A enregistre jusqu'à 50 jeux de données. En mode de mesure, appuyez sur  pour enregistrer un jeu. L'appareil confirmera l'enregistrement des données en affichant pendant une seconde **SAVE** (Enregistrer) et le numéro du jeu. Si la mémoire est pleine, l'appareil affiche « Full » (Pleine) lorsque vous essayez d'enregistrer des données.

Pour afficher des données enregistrées, appuyez sur Mode jusqu'à ce que **RECALL** (Rappeler) s'affiche, puis appuyez sur . Parcourez alors les jeux enregistrés à l'aide des touches de déplacement vers le haut ou vers le bas. Appuyez sur Mode pour revenir au mode de mesure.

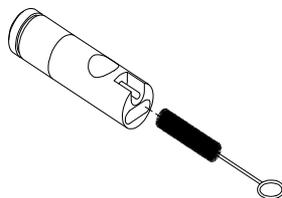
Pour supprimer des jeux de données, appuyez sur Mode en mode de mesure jusqu'à ce que DELETE (Supprimer) s'affiche. Appuyez sur  $\leftarrow$ . « All » (Tous) s'affiche et clignote. À l'aide des touches de déplacement vers le haut ou vers le bas, passez de All (Tous) à Each (Chaque). Appuyez sur  $\leftarrow$  pour valider l'option affichée (All ou Each).

Si vous choisissez All, tous les jeux seront supprimés de la mémoire et None (Aucun) sera affiché. Appuyez deux fois sur Mode pour revenir au mode de mesure. Si vous sélectionnez Each, faites défiler les jeux de données enregistrés avec les touches de déplacement vers le haut ou vers le bas. Appuyez sur  $\leftarrow$  pour supprimer le jeu sélectionné. La suppression d'un jeu modifie le classement des jeux suivants, de manière à garder les jeux en suite ordonnée. Par exemple, si le jeu 3 est supprimé, le jeu 4 deviendra le jeu 3, le jeu 5 deviendra le no 4, etc. Appuyez sur Mode pour revenir au mode de mesure.

## **ENTRETIEN DE LA SONDE**

La condition la plus importante pour obtenir des mesures de conductivité précises et reproductibles est d'utiliser une cellule propre. Une cellule sale altère la conductivité d'une solution par contamination. Nettoyez soigneusement la cellule avant de l'entreposer. Pour nettoyer la cellule de conductivité :

1. Plongez la cellule dans une solution de nettoyage et agitez pendant deux à trois minutes. Tout acide de nettoyage moussant pour céramique, tel que le produit de nettoyage pour salle de bain de Dow Chemical, doit convenir à cette opération. Pour obtenir un produit de nettoyage plus puissant, utilisez une solution d'alcool isopropylique et de chlorure d'hydrogène 1N à mélange égal. Retirez la cellule de la solution de nettoyage.



2. Utilisez la brosse en nylon (fournie) pour déloger tout contaminant se trouvant à l'intérieur de la chambre de l'électrode.
3. Répétez les étapes un et deux jusqu'à ce que la cellule soit complètement nettoyée. Rincez soigneusement la cellule avec de l'eau désionisée ou de l'eau courante propre.

## DÉPANNAGE

AFFICHAGE PRINCIPAL		PROBLÈME	SOLUTION POSSIBLE
OvEr		<ul style="list-style-type: none"> <li>• Conductivité &gt; 200,0 mS</li> <li>• Salinité &gt; 70,00 ppt (<math>\times 10^{-3}</math>)</li> </ul>	<ul style="list-style-type: none"> <li>• Submergez complètement la sonde.</li> <li>• Attendez suffisamment longtemps pour que l'électrode et la sonde Temp se stabilisent.</li> </ul>
OvEr/Undr lors de l'étalonnage		<ul style="list-style-type: none"> <li>• L'étalonnage de la constante de la cellule est hors limites</li> </ul>	<ul style="list-style-type: none"> <li>• Effectuez un nouvel étalonnage avec une valeur de standard de conductivité correcte.</li> <li>• Remplacez le standard de conductivité.</li> <li>• Nettoyez la cellule.</li> <li>• Retournez l'appareil au centre de service.</li> </ul>
AFFICHAGE PRINCIPAL	AFFICHAGE SECONDAIRE		
OvEr/Undr	OvEr <hr/> Undr	Température > 90,0 °C <hr/> Température < -10,0 °C	<ul style="list-style-type: none"> <li>• Augmentez/Diminuez la température de l'échantillon.</li> <li>• Retournez l'appareil au centre de service.</li> </ul>

## SPÉCIFICATIONS

Affichage	Fourchette	Précision	Résolution
Calcul automatique de la fourchette de conductivité	0,0 à 499,9 uS/cm	±1 % de la lecture plus 2 uS/cm	0,01 uS/cm
	500 à 4 999 uS/cm	±1 % de la lecture plus 5 uS/cm	1 mS/cm
	5,00 à 49,99 mS/cm	±1 % de la lecture plus 0,05 uS/cm	0,01 mS/cm
	50,0 à 200,0 mS/cm	±2,5% de la lecture plus 0,5 mS/cm	0,1 mS/cm
Salinité	0,0 à 70,0 x 10 <sup>-3</sup>	0,2 % pleine échelle	0,1 x 10 <sup>-3</sup>
Température °C	-10,0 à 90 °C	Le plus grand de ±0,2 °C ou ±0,4 % pleine échelle	0,1 °C

Référence de la température	15,0 à 25,0 °C
Coefficient de la température	0,0 % à 4,0 %
Constante de la cellule	5,00 ± 0,50
Fourchette de constantes du TSD	0,30 à 1,00
Alimentation/Autonomie des piles	Une pile de 9V/ 500 heures environ
Sauvegarde de l'étalonnage	Oui
Touches sonores	Oui, toutes les touches tactiles
Boîtier de l'appareil	Etanche, norme IP67
Fourchette de températures de fonctionnement	0 à 50 °C
Limite d'humidité relative lors du fonctionnement	Jusqu'à 95 %
Sonde de température	Thermistor, 10 kΩ / 25 °C
Dimensions (L x l x P)	18.7 cm x 7.6 cm x 3.8 cm
Poids (avec pile)	270 g

## LISTE DES PIÈCES DÉTACHÉES RECOMMANDÉES

N° RÉF.	DESCRIPTION
300-1	Assemblage, câble de 1 mètres et sonde.
300-4	Assemblage, câble de 4 mètres et sonde.
300-10	Assemblage, câble de 10 mètres et sonde.
606043	Sacoche de transport, flancs durs.
485	Sacoche de transport, flancs souples.

Article n° #606042REF

Révision A • Juillet 2012

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## GARANTÍA

El medidor EcoSense® EC300A tiene un año de garantía contra defectos de materiales y fabricación, contado a partir de la fecha de compra por el usuario final. Las sondas y cables del medidor EC300A tienen un año de garantía contra defectos de materiales y fabricación, contado a partir de la fecha de compra por el usuario final. Durante el período de garantía, YSI reparará o reemplazará, según su criterio, sin coste alguno, cualquier producto que YSI determine que está cubierto por esta garantía.

Para hacer valer esta garantía, escriba o llame al representante local de YSI, o comuníquese con el Servicio de atención al cliente de YSI en Yellow Springs, Ohio, EE.UU. Envíe el producto y la factura de compra, con el flete prepagado, al centro de servicio técnico autorizado seleccionado por YSI. Se realizará la reparación necesaria o el reemplazo del producto y este será enviado de vuelta, con el flete prepagado. Los productos reparados o reemplazados se garantizan durante el resto del período de la garantía original, o al menos durante 90 días contados a partir de la fecha de reparación o reemplazo.

### **Limitación de la garantía**

Esta garantía no tendrá validez en caso de daños o fallos en el producto de YSI debido a lo siguiente: (i) la instalación, funcionamiento o utilización del producto de manera contraria a las instrucciones escritas suministradas por YSI; (ii) abuso o uso inadecuado del producto; (iii) falta de mantenimiento del producto de acuerdo con las instrucciones escritas suministradas por YSI o con los procedimientos estándar de la industria; (iv) cualquier reparación indebida realizada en el producto; (v) utilización por parte del usuario de componentes o repuestos defectuosos o inadecuados para el mantenimiento o reparación del producto; o (vi) cualquier modificación del producto no autorizada de manera expresa por YSI.

ESTA GARANTÍA SE OTORGA EN LUGAR DE CUALQUIER OTRA GARANTÍA, EXPLÍCITA O IMPLÍCITA, LO QUE INCLUYE TODA GARANTÍA DE COMERCIALIZACIÓN O IDONEIDAD PARA UN PROPÓSITO ESPECÍFICO. DE CONFORMIDAD CON ESTA GARANTÍA, LA RESPONSABILIDAD DE YSI SE LIMITA A LA REPARACIÓN O REEMPLAZO DEL PRODUCTO, LO CUAL SERÁ LA SOLUCIÓN ÚNICA Y EXCLUSIVA QUE TENDRÁ EL COMPRADOR POR CUALQUIER PRODUCTO DEFECTUOSO CUBIERTO POR ESTA GARANTÍA. EN NINGÚN CASO YSI SERÁ RESPONSABLE POR NINGÚN DAÑO CUANTIFICABLE, INDIRECTO, INCIDENTAL O CONSIGUIENTE QUE RESULTARA DE ALGÚN PRODUCTO DEFECTUOSO CUBIERTO POR ESTA GARANTÍA.

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## **INTRODUCCIÓN GENERAL**

El EC300A es una herramienta de precisión que mide la conductividad, la salinidad y la temperatura. Tiene un microprocesador integrado que calcula y realiza la compensación de todos los parámetros relacionados con las determinaciones de la conductividad y de la temperatura.

Cuando está instalado la tapa conectora, el instrumento es a prueba de agua (IP67). Las teclas de contacto mecánico son muy confiables y al pulsarlas proporcionan una respuesta táctil y audible. Este instrumento utiliza una pila de 9 voltios. No requiere nueva calibración cuando se restablece la corriente.

La parte delantera del medidor tiene una pantalla grande de cristal líquido que muestra simultáneamente la temperatura y la conductividad, salinidad o TDS con compensación de temperatura o sin compensación de temperatura, junto con las indicaciones para el usuario y los indicadores del modo de funcionamiento. La unidad orienta a los usuarios durante los procedimientos de calibración y medición.

El modelo EC300A está disponible con una sola celda de cuatro electrodos. Entre otras características se incluyen la calibración automática de la conductividad, compensación automática de la temperatura, larga duración de la pila y rechazo de ruido de CA de 50/60 Hz. Este medidor es universal y fácil de usar en aplicaciones in situ, industriales y de laboratorio.

## **INSPECCIÓN INICIAL**

Saque la unidad de su embalaje con cuidado y verifique que no haya sufrido daños durante el envío. Compare las piezas recibidas con los materiales enumerados en la lista de embalaje. Avise inmediatamente a YSI en caso de que haya piezas faltantes o dañadas. Guarde todos los materiales de embalaje hasta que confirme que la unidad funciona satisfactoriamente.

## **EL INSTRUMENTO**

Aunque el instrumento se encuentra en un estuche IP67 a prueba de agua, NO lo utilice bajo agua. El conector no es a prueba de agua a menos que la tapa esté instalado. En caso de sumergirlo sin la tapa conectado, siga estos pasos inmediatamente.

1. Seque el conector, si es necesario, y cambie la sonda de medida de la conductividad. Enjuague la unidad cuidadosamente con agua destilada. Después del enjuague y secado, revise y limpie los conectores para eliminar cualquier contaminante que pueda afectar las conexiones de la sonda.
2. Espere hasta que la unidad y la sonda se sequen por completo antes de reanudar el funcionamiento.
3. Si la unidad no funciona correctamente después de realizar los pasos 1 y 2, comuníquese con YSI para su posible reparación o reemplazo (consulte la garantía).

## INSTALACIÓN DE LA PILA

En la pantalla de cristal líquido aparecerá el mensaje de "BAT" (pila) para indicar que queda aproximadamente una hora de carga de la pila para el funcionamiento de la unidad según las especificaciones. Cambie la pila cuando aparezca el mensaje "BAT" (pila) en la pantalla de cristal líquido. (Vea la figura 1).

Para cambiar la pila, saque los dos tornillos de la tapa, luego retire la tapa y el aro tórico. Coloque una nueva pila de 9 voltios. Vuelva a colocar la tapa y el aro tórico (alineee este aro correctamente para garantizar un buen sellado) y ajuste los dos tornillos de la tapa para que funcione la protección contra salpicaduras.

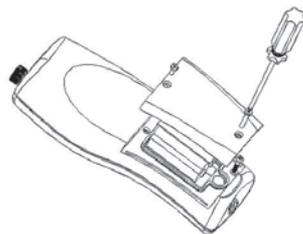


Figura 1.  
Instalación de la pila

## Eliminación de las pilas

Este instrumento funciona con pila (9V) que el usuario debe extraer y desechar cuando ya no funcionan. Los requisitos de desecho varían según el país y la región, y se espera que los usuarios entiendan y sigan los requisitos de desecho de pilas para su área específica.

## FUNCIONES PRINCIPALES DEL MODELO EC300A

1. **⏻** : para encender y apagar la unidad. Los valores de calibración no se suprimen cuando se apaga la unidad. Cuando la unidad no esté en uso, apáguela para ahorrar carga de la pila. El instrumento tiene una función de apagado automático a los 30 minutos cuando no está en uso. Para el almacenamiento a largo plazo debe quitar la pila.
2. **MODE**: Selecciona el modo de la pantalla. Durante el funcionamiento normal, pulse MODE (modo) para cambiar la pantalla entre conductividad sin compensación, conductividad compensada por temperatura, salinidad, cantidad de sólidos disueltos (TDS, por sus siglas en inglés), borrar y recordar. En el modo de calibración, esta tecla sale de la calibración actual y muestra el siguiente parámetro de calibración.
3. **CAL**: durante el funcionamiento normal, cambia el modo de Normal a Calibration (Calibración).
4. **↵** (Enter) : en el ajuste de la calibración, pulse esta tecla para guardar el parámetro actual en la memoria.
5. **Δ** y **∇** teclas: para aumentar o disminuir el valor que aparece en la pantalla según lo desee.

## PANTALLA DE CRISTAL LÍQUIDO

1. **CONDUCTIVITY**: aparece al medir la conductividad.
2. **BAT**: indicador de pila agotada.
3. **CELL**: indica el valor constante de la celda de conductividad.
4. Pantalla principal para los valores de conductividad, salinidad y TDS compensados y no compensados.

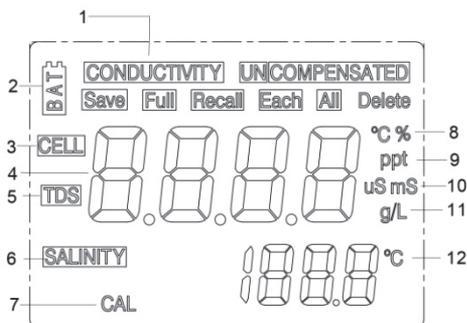


Figura 2. Pantalla de cristal líquido

5. **TDS:** aparece al medir la cantidad de sólidos disueltos.
6. **SALINITY:** aparece al medir la salinidad.
7. **CAL:** indicador del modo de calibración.
8. **°C:** parpadea durante la medición de la conductividad con compensación de temperatura. Durante la calibración, indica la unidad de referencia de la temperatura.  
**%:** aparece durante la calibración; indica la unidad del coeficiente de temperatura.
9. **ppt:** partes por millar; indica la medición de la salinidad.
10. **uS, mS:** micro Siemens, milli Siemens; indica la medición de la conductividad.
11. **g/L:** gramos/Litro; indica la medición de TDS.
12. **°C:** indicador de la temperatura.

## MODOS DE MEDICIÓN

1. **Temperatura:** la pantalla muestra constantemente la temperatura actual de la solución.
2. **Conductividad con compensación de temperatura:** medición de la conductividad, compensada a 25°C o a otro valor especificado entre 15 y 25°C. Expresada en uS/cm o en mS/cm con un "°C" parpadeante.
3. **Conductividad no compensada:** medición directa de la conductividad, sin compensar a una temperatura específica. Expresada en uS/cm o en mS/cm.
4. **Salinidad:** medición de la salinidad; expresada en partes por millar (ppt).
5. **TDS:** medición de la cantidad de sólidos disueltos (TDS); expresada en gramos por litro (g/L)

Observe detenidamente las unidades mostradas en el extremo de la pantalla de cristal líquido para determinar el modo deseado.

## CALIBRACIÓN

La configuración de calibración contiene cinco secciones: TDS, celda, coeficiente de temperatura, referencia de la temperatura y calibración de la conductividad. Para acceder a estas secciones:

1. Conecte el conjunto de sonda de medida de la conductividad y cables a la unidad y enciéndala. La pantalla mostrará **CELL** y la constante de la celda de la sonda de medida de la conductividad.
2. Deje que las lecturas de temperatura se estabilicen, luego pulse **CAL** para entrar en el modo de calibración; **CAL** aparece en la pantalla de cristal líquido. Pulse **MODE** para ver las siguientes secciones de manera secuencial:

**Nota:** Pulse Enter  para aceptar cualquier cambio de valor en cada sección y pasar automáticamente a la próxima sección. Si no hay cambios, la unidad acepta el valor actual y procede a la próxima sección.

### TDS

La TDS se determina al multiplicar la conductividad (mS) por un factor de TDS. El valor predeterminado del factor es 0,65. Para cambiar el factor de TDS, use las teclas **Δ** y **∇** para ajustar el valor entre 0,30 y 1,00. Pulse Enter  para guardar el nuevo valor o pulse **MODE** para cancelar el cambio y mostrar la pantalla **CELL**.

### CELDA

La segunda pantalla mostrará **CELL** y el valor actual de la celda. El valor predeterminado de la celda es 5,00 y se muestra en la parte inferior derecha de la pantalla. La unidad permite una variación de  $\pm 0,50$  antes de mostrar un mensaje de error. El valor de la celda no se

puede ajustar en esta pantalla; la única manera de ajustar la constante de la celda es calibrando la conductividad. Pulse Enter  para reajustar la constante de la celda a 5,00 y para que aparezca la pantalla **Coefficiente de temperatura**.

**Nota:** Asegúrese de pulsar Enter  para restablecer la constante de la celda en 5,00. Si se pulsa **MODE**, la unidad conserva la constante anterior de la celda y calibra a partir de un valor que ya está desfasado.

## Coefficiente de temperatura

La unidad utiliza el coeficiente de temperatura para calcular la conductividad con compensación de temperatura. El valor predeterminado es de 1,91%. Para cambiar el coeficiente de temperatura, use las teclas **Δ** y **∇** para ajustar el valor entre 0 y 4,00%. Pulse Enter  para guardar el nuevo valor o pulse **MODE** para cancelar el cambio y mostrar la pantalla **Referencia de la temperatura**.

## Referencia de la temperatura

La unidad utiliza el valor de referencia de la temperatura para calcular la conductividad con compensación de temperatura. El valor predeterminado es 25°C. Para cambiar la referencia de la temperatura, utilice las teclas **Δ** y **∇** para ajustar el valor entre 15 y 25°C. Pulse Enter  para guardar el nuevo valor o pulse **MODE** para cancelar el cambio y mostrar la pantalla **Calibración de la conductividad**.

## Calibración de la conductividad

1. Sumerja la sonda en una solución estándar de conductividad conocida, preferentemente una en la escala intermedia de las soluciones que se van a medir. Sumerja por completo la sonda sin tocar los lados del recipiente de calibración. Sacuda ligeramente la sonda para eliminar cualquier burbuja de aire que esté atrapada en la celda de conductividad.
2. Deje que la temperatura se estabilice. Es posible que aparezca brevemente el mensaje 'rAng' (escala) para indicar la calibración automática de la unidad; esto es normal. Después de la estabilización de la temperatura, utilice las teclas **Δ** y **∇** para ajustar el valor de conductividad a aquél del estándar de conductividad a 25°C. Pulse Enter  para calibrar. La unidad emite dos tonos para indicar una calibración exitosa, luego cambia automáticamente al modo de operación normal.

## MEDICIONES DE CONDUCTIVIDAD

1. Encienda la unidad. Coloque la sonda en la solución que se va a medir. Sumerja por completo la sonda. Sacuda ligeramente la sonda para eliminar cualquier burbuja de aire que esté atrapada en la celda de conductividad.
2. Pulse **MODE** para entrar en el modo de medición deseado. Es posible que aparezca brevemente en la pantalla el mensaje 'rAng' (escala) para indicar la calibración automática de la unidad; esto es normal. Deje que la temperatura se estabilice antes de tomar las mediciones.

## CÓMO GUARDAR, VER Y BORRAR DATOS

El EC300A puede almacenar 50 registros de datos. Cuando esté en modo de medición, pulse  para guardar un registro. El instrumento confirmará los datos guardados indicando SAVE (guardar) y el número de registro durante un segundo. Se muestra "Full" (llena) cuando se intenta guardar datos y la memoria está llena.

Para ver los datos guardados, pulse "mode" (modo) hasta que se muestre RECALL (recordar) y luego pulse  Use las teclas de flecha hacia arriba y abajo para revisar diferentes registros guardados. Pulse "Mode" (modo) para volver al modo de medición.

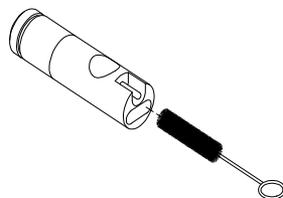
Para borrar los registros de datos, pulse "Mode" (modo) mientras está en el modo de medición hasta que se muestre DELETE (borrar). Pulse  $\leftarrow$ . Se mostrará "All" (todo) parpadeando. Pulse la flecha hacia arriba y hacia abajo para cambiar entre las opciones para borrar "All" (todo) o "Each" (cada uno). Seleccione "All" o "Each" presionando  $\leftarrow$  mientras se muestra esa opción.

Si está seleccionado "All" (todo), se borrarán todos los registros de la memoria y se mostrará "None" (ninguno). Pulse "Mode" (modo) dos veces para volver al modo de medición. Si se selecciona "Each" (cada uno), las flechas hacia arriba y hacia abajo le permitirán desplazarse a través de los registros de datos guardados. Pulse  $\leftarrow$  para borrar el registro seleccionado. Todos los registros después del registro borrado pasarán hacia arriba para mantener los registros en orden secuencial. Por ejemplo, si se borra el registro 3, el registro 4 se volverá el 3 y el 5 se volverá el registro 4. Pulse "Mode" (modo) dos veces para volver al modo de medición.

## **MANTENIMIENTO DE LA SONDA**

El requisito más importante para realizar mediciones de conductividad precisas y reproducibles es que la celda esté limpia. Una celda sucia cambia la conductividad de una solución a través de la contaminación. Limpie a fondo la celda antes de almacenarla. Para limpiar la celda de conductividad:

1. Sumerja la celda en una solución de limpieza y agite durante dos a tres minutos. Cualquier limpiador de azulejos ácido y espumoso, como Dow Chemical Bathroom Cleaner, sirve para limpiar la celda adecuadamente. Para obtener un limpiador más fuerte, utilice una solución 1:1 de alcohol isopropílico y 1 N HCl. Saque la celda de la solución de limpieza.
2. Utilice el cepillo de nylon (incluido) para eliminar cualquier contaminante del interior de la cámara de electrodos.
3. Repita los pasos uno y dos hasta que la celda esté completamente limpia. Enjuague a fondo la celda con agua desmineralizada o limpia de grifo.



## LOCALIZACIÓN DE FALLOS

PANTALLA PRINCIPAL		PROBLEMA	POSIBLE SOLUCIÓN
OvEr		<ul style="list-style-type: none"> <li>• La conductividad es &gt;200,0 mS</li> <li>• La salinidad es &gt; 70,00 ppt</li> </ul>	<ul style="list-style-type: none"> <li>• Sumerja por completo la sonda.</li> <li>• Deje transcurrir suficiente tiempo para que se estabilicen el electrodo y la sonda Temp.</li> </ul>
OvEr/Undr durante la calibración		La calibración de la constante de la celda está fuera de la escala	<ul style="list-style-type: none"> <li>• Recalibrar con el valor correcto para el estándar de conductividad.</li> <li>• Cambie el estándar de conductividad.</li> <li>• Limpie la celda.</li> <li>• Envíelo al servicio técnico.</li> </ul>
PANTALLA PRINCIPAL	PANTALLA SECUNDARIA		
OvEr/Undr	<u>OvEr</u> <u>Undr</u>	<p>La temperatura es &gt;90,0 °C</p> <hr/> <p>o la temperatura es &lt; -10,0 °C</p>	<ul style="list-style-type: none"> <li>• Disminuya/aumente la temperatura de la muestra.</li> <li>• Envíelo al servicio técnico.</li> </ul>

## ESPECIFICACIONES

Pantalla	Escala	Precisión	Resolución
Conductividad, calibración automática	0,0 a 499,9 uS/cm 500 a 4999 uS/cm 5,00 a 49,99 mS/cm 50,0 a 200,0 mS/cm	±1% de lectura más 2 uS/cm ±1% de lectura más 5 uS/cm ±1% de lectura más 0,05 uS/cm ±2,5% de lectura más 0,5 mS/cm	0,01 uS/cm 1 mS/cm 0,01 mS/cm 0,1 mS/cm
Salinidad	0,0 a 70,0 ppt	0,2% de la escala completa	0,1 ppt
Temperatura en °C	-10,0 a 90 °C	±0,2 °C o ±0,4% de la escala completa, lo que sea mayor	0,1° C

Temperatura de referencia	15,0 a 25,0 °C
Coefficiente de temperatura	0,0% a 4,0%
Constante de la celda	5,00 ± 0,50
Escala de la constante TDS	0,30 a 1,00
Energía/Duración de la pila	Una pila de 9 voltios/Aproximadamente 500 horas
Respaldo de la calibración	Sí
Respuesta audible	Sí, en todas las teclas
El Instrumento	IP 67, A prueba de agua con la tapa conector
Escala de temperatura de funcionamiento	0 a 50° C
Escala de humedad relativa de funcionamiento	Hasta 95%
Sonda de medida de la temperatura	Termistor, 10kΩ / 25° C
Dimensiones (lar x anc x prof)	18.7 cm x 7.6 cm x 3.8 cm (7,3 pulg. X 3 pulg. X 1,5 pulg.)
Peso (con la pila)	270 gramos

## LISTA DE PIEZAS DE RECAMBIO RECOMENDADAS

PIEZA N°	DESCRIPCIÓN
300-1	Conjunto de sonda de 1 metros y cables.
300-4	Conjunto de sonda de 4 metros y cables.
300-10	Conjunto de sonda de 10 metros y cables.
606043	Estuche portátil, de lados rígidos.
485	Estuche portátil, no rígido.

Artículo #606042REF

Revisión A • Julio de 2012

Para la versión más reciente de este manual, visite [www.ysi.com](http://www.ysi.com)

## **GARANTIA**

O instrumento EC300A da YSI tem uma garantia durante um período de um ano válido a partir da data de compra pelo utilizador final contra defeitos de material e mão-de-obra. As sondas e cabos do instrumento EC300A da YSI têm uma garantia durante um período de um ano a partir da data de compra pelo utilizador final contra defeitos de material e mão-de-obra. Durante o período da garantia, a YSI reparará ou substituirá, sob sua discricção, gratuitamente, quaisquer produtos que determine como estando abrangidos pelos termos desta garantia.

Para exercer os termos desta garantia, escreva ou contacte o representante local da YSI ou o Serviço de Apoio ao Cliente da YSI. Envie o produto e a prova de compra, com transporte pré-pago, para o Centro de Assistência Autorizado seleccionado pela YSI. A reparação ou substituição será efectuada e o produto devolvido, sendo o transporte pré-pago. Os produtos reparados ou substituídos têm uma garantia que cobre o período restante do período original da garantia ou de pelo menos 90 dias a partir da data da reparação ou substituição.

### **Limitação da garantia**

Esta Garantia não se aplica a quaisquer danos ou falhas/avarias dos produtos da YSI provocados por: (i) falha em instalar, operar ou utilizar o produto de acordo com as instruções escritas da YSI; (ii) abuso ou uso indevido do produto; (iii) falha em manter o produto de acordo com as instruções escritas da YSI ou procedimento padrão da indústria; (iv) quaisquer reparações indevidas ao produto; (v) uso por parte do utilizador de quaisquer componentes ou peças defeituosas ou indevidos nas tarefas de assistência ou reparação do produto; ou (vi) modificação do produto de qualquer maneira não expressamente autorizada pela YSI.

ESTA GARANTIA VEM SUBSTITUIR TODAS AS DEMAIS GARANTIAS, EXPRESSAS OU IMPLÍCITAS, INCLUINDO QUAISQUER GARANTIAS DE COMERCIALIZAÇÃO OU ADEQUAÇÃO/APTIDÃO PARA UM DETERMINADO OBJECTIVO. A RESPONSABILIDADE DA YSI AO ABRIGO DOS TERMOS DESTA GARANTIA ENCONTRA-SE LIMITADA À REPARAÇÃO OU SUBSTITUIÇÃO DO PRODUTO, E ESTA SERÁ A SUA ÚNICA E EXCLUSIVA SOLUÇÃO PARA QUAISQUER PRODUTOS DEFEITUOSOS ABRANGIDOS PELOS TERMOS DESTA GARANTIA. EM CASO ALGUM, SERÁ A YSI RESPONSÁVEL POR QUAISQUER DANOS ESPECIAIS, INDIRECTOS, ACIDENTAIS OU CONSEQUENTES RESULTANTES DA COBERTURA DE QUAISQUER PRODUTOS DEFEITUOSOS ABRANGIDOS POR ESTA GARANTIA.

## **INFORMAÇÃO DE CONTACTO**

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## INTRODUÇÃO GERAL

O modelo EC300A é um instrumento preciso que mede condutividade, salinidade e temperatura. Um microprocessador integrado calcula e compensa todos os parâmetros relacionados com as determinações da condutividade e temperatura.

A unidade tem um invólucro IP67 resistente a salpicos. As teclas mecânicas de pressão são altamente fiáveis com um feedback táctil e áudio. Este instrumento usa uma pilha de 9V. Não é necessário efectuar uma nova calibração ao restaurar a energia.

O instrumento tem um ecrã LCD de grande dimensão na parte frontal que apresenta a temperatura e a condutividade compensada pela temperatura ou a condutividade não compensada pela temperatura, a salinidade ou TDS simultaneamente a par dos comandos do utilizador e indicadores do modo. A unidade apresenta os comandos para o utilizador através dos procedimentos de calibração e medição.

O modelo EC300A encontra-se disponível com uma célula única com quatro eléctrodos. Outras funções disponíveis incluem a amplitude automática da condutividade, compensação automática da temperatura, longa duração da pilha e rejeição do ruído de 50/60 Hz CA. Este contador é universal e fácil de utilizar, para aplicações no terreno, industriais e laboratoriais.

## INSPECÇÃO INICIAL

Desempacote cuidadosamente a unidade e os acessórios e inspecione-os com vista a detectar danos de envio. Compare as peças recebidas com os materiais listados na lista de empacotamento. Notifique a YSI imediatamente de quaisquer danos ou peças em falta. Guarde todos os materiais da embalagem até a operação satisfatória ser confirmada.

## O INSTRUMENTO

NÃO use o instrumento debaixo de água embora ele se encontre alojado num invólucro IP67 impermeável. O conector não é impermeável excepto quando a tampa esteja instalado. Se o instrumento for submerso sem a tampa ou cabo ligado, cumpra os seguintes passos prontamente:

1. Seque o conector se necessário, e substitua a sonda da condutividade. Enxágue a unidade cuidadosamente com água destilada. Após o enxaguamento e secagem, inspecione e limpe os conectores para remover todas as substâncias contaminantes que possam afectar as ligações da sonda.
2. Aguarde até a unidade e sonda estarem completamente secas antes de retomar a operação.
3. Contacte a YSI para fins de possível reparação ou substituição (consultar a Garantia) se a unidade não funcionar correctamente após os passos 1 e 2.

## INSTALAÇÃO DA PILHA

A indicação inicial "BAT" quando apresentada no ecrã LCD indica cerca de uma hora de duração da pilha para funcionamento da unidade de acordo com as especificações. Substitua a pilha quando a indicação "BAT" surgir no ecrã LCD. (ver Figura 1.)

Para substituir a pilha, remova os dois parafusos da tampa do compartimento da pilha e tampa do compartimento e anel O. Substitua a pilha de 9V. Instale a tampa do compartimento da pilha e o anel O (alinhe o anel O devidamente para assegurar uma boa vedação) e aperte os dois parafusos da tampa do compartimento da pilha para assegurar a função de resistência a salpicos.

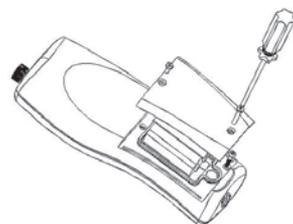


Figura 1.  
Instalação da pilha

## Descarte da pilha

Este instrumento é alimentado por uma pilha de 9V, que o utilizador deve remover e eliminar quando já não tiver carga para alimentar o instrumento. Os requisitos de descarte variam por país e região e espera-se que os utilizadores compreendam e cumpram os requisitos de descarte das pilhas para o seu local específico.

## FUNÇÕES CHAVE DO MODELO EC300A

- : Liga (ON) ou desliga (OFF) a unidade. Os valores de calibração não são eliminados quando a unidade é desligada. Quando a unidade não está em uso, desligue-a para poupar a pilha. O instrumento tem uma função de desactivação automática de 30 minutos quando não está em uso. Remova a pilha no caso de armazenamento prolongado.
- MODE (Modo):** Selecciona o modo de visualização. No funcionamento Normal, prima MODE para comutar a visualização entre Uncompensated Conductivity (Conductividade não compensada), Temperature Compensated Conductivity (Conductividade compensada pela temperatura), Salinity (Salinidade), Total Dissolved Solids (Total de sólidos dissolvidos) (TDS), Delete (Eliminar) e Recall (Consultar). No modo Calibration (Calibração), esta tecla sai da actual calibração e apresenta o próximo parâmetro de calibração.
- CAL (Calibração):** No funcionamento normal, passa do modo Normal para o modo Calibration.
-  (Enter): Na Configuração da calibração, prima esta tecla para guardar o actual parâmetro na memória do instrumento.
- Teclas  $\Delta$  e  $\nabla$**  Aumenta ou diminui o valor do ecrã conforme desejado.

## O ECRÃ LCD

### 1. CONDUCTIVITY (Conductividade):

Apresentado ao medir a condutividade.

### 2. BAT (Pilha):

Indicador de carga fraca da pilha.

### 3. CELL (Célula):

Indica o valor constante da célula da condutividade.

### 4. Ecrã principal para os valores de condutividade compensada e não compensada, salinidade e TDS.

### 5. TDS:

Apresentado ao medir o total de sólidos dissolvidos.

### 6. SALINITY (Salinidade):

Apresentado ao medir a salinidade.

### 7. CAL (Calibração):

Indicador do modo Calibration (Calibração).

### 8. °C:

Pisca no ecrã durante a medição da condutividade compensada pela temperatura. Durante a calibração, indica a unidade de referência da temperatura.

**%:** Durante a calibração, indica a unidade do coeficiente da temperatura.

### 9. ppt:

Peças por mil; indica a medição da salinidade.

### 10. uS, mS:

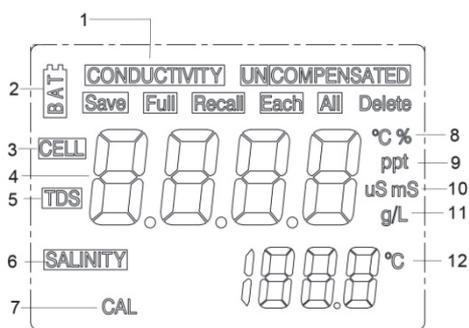
micro Siemens, mili Siemens; Indica a medição da condutividade.

### 11. g/L:

gramas/Litro; indica a medição dos TDS.

### 12. °C:

Ecrã da temperatura.



## MODOS DE MEDIÇÃO

1. **Temperature (Temperatura)** - A temperatura da solução actual é apresentada continuamente.
2. **Temperature Compensated Conductivity (Condutividade compensada pela temperatura)** - Medição da condutividade, compensada para 25°C ou outro valor especificado entre 15 e 25°C. Expressa como  $\mu\text{S}/\text{cm}$  ou  $\text{mS}/\text{cm}$  com a indicação "°C" a piscar.
3. **Uncompensated Conductivity (Condutividade não compensada)** - Medição directa da condutividade, não compensada para uma temperatura específica. Expressa em  $\mu\text{S}/\text{cm}$  ou  $\text{mS}/\text{cm}$ .
4. **Salinity (Salinidade)** - Medição da salinidade; expressa em partes por mil (ppt).
5. **TDS (Total de sólidos dissolvidos)** - Medição do total de sólidos dissolvidos (TDS); expressa como gramas por litro (g/L). Respeite cuidadosamente as unidades apresentadas no lado oposto do ecrã LCD para determinar o modo desejado.

## CALIBRAÇÃO

A configuração da calibração contém cinco secções: TDS (Total de sólidos dissolvidos), Cell (Célula), Temperature Coefficient (Coeficiente da temperatura), Temperature reference (Referência da temperatura) e Conductivity Calibration (Calibração da condutividade). Para aceder a estas secções:

1. Ligue o conjunto da sonda da condutividade e do cabo à unidade e ligue esta última. A indicação **CELL (Célula)** surgirá no ecrã e a constante da célula da sonda da condutividade.
2. Permita a estabilização das leituras da temperatura, e prima depois **CAL (Calibração)** para aceder ao modo Calibration (Calibração); a indicação **CAL** surge no ecrã LCD. Prima **MODE (Modo)** para apresentar sequencialmente as seguintes secções:

**Nota:** Prima Enter (↵) para aceitar quaisquer alterações dos valores em cada secção e avançar automaticamente para a próxima secção. Se não houverem quaisquer alterações, a unidade aceita o actual valor e avança para a secção seguinte.

### **TDS (Total de sólidos dissolvidos)**

O valor TDS é determinado multiplicando a condutividade (mS) por um factor TDS. O valor predefinido do factor é de 0,65. Para alterar o factor TDS, use as teclas  $\Delta$  e  $\nabla$  para ajustar o valor entre 0,30 e 1,00. Prima Enter (↵) para guardar o novo valor, ou prima **MODE (Modo)** para cancelar a alteração e apresentar o ecrã **CELL (Célula)**.

### **CÉLULA**

O segundo ecrã apresentará a indicação **CELL (Célula)** e o actual valor da célula. O valor predefinido da célula é de 5,00 e é apresentado no canto inferior do ecrã. A unidade permite uma variação de  $\pm 0,50$  antes de apresentar uma mensagem de erro. Não é possível ajustar o valor da célula neste ecrã; a calibração da condutividade é a única maneira de ajustar a constante da célula. Prima Enter (↵) para reconfigurar a constante da célula para 5,00 e apresentar o ecrã **Temperature Coefficient (Coeficiente da temperatura)**.

**Nota:** Prima Enter (↵) para reconfigurar a constante da célula para 5,00. Se premir **MODE (Modo)**, a unidade mantém a constante da célula anterior e efectua a calibração a partir de um valor já desviado.

## **Coeficiente da temperatura**

A unidade usa o coeficiente da temperatura para calcular a condutividade compensada pela temperatura. O valor predefinido é de 1,91%. Para alterar o coeficiente da temperatura, use as teclas  $\Delta$  e  $\nabla$  para ajustar o valor entre 0 e 4,00%. Prima Enter ( $\rightarrow$ ) para guardar o novo valor, ou prima **MODE (Modo)** para cancelar a alteração e apresentar o ecrã **Temperature Reference (Referência da temperatura)**.

## **Referência da temperatura**

A unidade usa o valor de referência da temperatura para calcular a condutividade compensada pela temperatura. O valor predefinido é de 25°C. Para alterar o coeficiente da temperatura, use as teclas  $\Delta$  e  $\nabla$  para ajustar o valor entre 15 e 25°C. Prima Enter ( $\rightarrow$ ) para guardar o novo valor, ou prima **MODE (Modo)** para cancelar a alteração e apresentar o ecrã **Conductivity Calibration (Calibração da condutividade)**.

## **Calibração da condutividade**

1. Submirja a sonda num padrão com condutividade conhecida, de preferência num padrão na amplitude média das soluções a medir. Submirja completamente a sonda sem tocar nos lados do recipiente da calibração. Abane a sonda ligeiramente para remover quaisquer bolhas de ar presas na célula de condutividade.
2. Permita que a temperatura estabilize. A mensagem 'rAng' (amplitude) pode ser brevemente apresentada para indicar a amplitude automática da unidade; isto é normal. Após a estabilização da temperatura, use as teclas  $\Delta$  e  $\nabla$  para ajustar o valor da condutividade ao do padrão da condutividade a 25°C. Prima Enter ( $\rightarrow$ ) para calibrar. A unidade emite dois breves sinais sonoros para indicar uma calibração bem sucedida, e muda automaticamente para o modo de funcionamento normal.

## **MEDIÇÕES DA CONDUTIVIDADE**

1. Ligue a unidade. Coloque a sonda na solução a medir. Mergulhe completamente a sonda. Abane a sonda ligeiramente para remover quaisquer bolhas de ar presas na célula de condutividade.
2. Prima **MODE (Modo)** para aceder ao modo de medição desejado. A mensagem 'rAng' (amplitude) pode ser brevemente apresentada no ecrã para indicar a amplitude automática da unidade; isto é normal. Permita a estabilização da temperatura antes de efectuar as medições.

## **GUARDAR, VISUALIZAR E ELIMINAR DADOS**

O instrumento EC300A consegue guardar até 50 registos de dados. Prima  $\rightarrow$  para guardar um registo estando no modo de medição. O instrumento confirmará os dados guardados apresentando a indicação SAVE (Guardar) e o número do registo durante um segundo. A indicação "Full" (Completa) é apresentada no ecrã quando tentar guardar dados e a memória do instrumento estiver cheia.

Prima Mode (Modo) até a indicação RECALL (Consultar) ser apresentada no ecrã e prima então  $\rightarrow$  para consultar os dados guardados. Use as teclas com setas para Cima ou para Baixo para consultar os diferentes registos guardados. Prima Mode para regressar ao modo de medição.

Prima Mode enquanto no modo de medição até a indicação DELETE (Eliminar) surgir no ecrã para apagar registos de dados. Prima  $\rightarrow$ . A indicação "All" (Tudo) será apresentada no ecrã a piscar. Prima as teclas com setas para Cima ou Baixo para alternar entre as opções 'All' ou 'Each' (Cada). Seleccione a opção 'All' ou 'Each' premindo  $\rightarrow$  enquanto essa opção é apresentada.

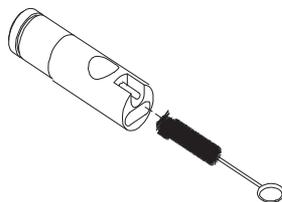
Se seleccionar a opção 'All', todos os registos serão eliminados da memória e a opção 'None' (Nenhum) será apresentada. Prima Mode duas vezes para regressar ao modo de medição.

Se seleccionar a opção 'Each', as teclas com setas para Cima e Baixo permitem-lhe consultar em deslocamento os registos de dados guardados. Prima  $\leftarrow$  para apagar o registo seleccionado. Todos os registos após o registo eliminado irão avançar uma posição nos registos na ordem sequencial. Por exemplo, se apagar o registo 3, o registo 4 torna-se então o registo 3 e o registo 5 torna-se o registo 4. Prima Mode duas vezes para regressar ao modo de medição.

## MANUTENÇÃO DA Sonda

Uma célula limpa é o requisito mais importante para a execução de medições da condutividade precisas e reproduzíveis. Uma célula suja altera a condutividade de uma solução através da contaminação. Limpe a célula cuidadosamente antes de a armazenar. Para limpar a célula de condutividade:

1. Mergulhe a célula numa solução de limpeza e agite durante 2 a 3 minutos. Qualquer agente de limpeza de tijoleira ácida com espuma, como o detergente de casa-de-banho químico Dow, deve limpar adequadamente. No caso de um agente de limpeza, use uma solução de 1:1 de álcool isopropilo e 1 N HCl. Remova a célula da solução de limpeza.
2. Use a escova de nylon (fornecida) para deslocar quaisquer substâncias contaminantes a partir do interior da câmara do eléctrodo.
3. Repita os passos 1 e 2 até a célula estar completamente limpa. Enxagúe a célula cuidadosamente em água desionizada ou da torneira limpa.



## RESOLUÇÃO DE PROBLEMAS

ECRÃ PRINCIPAL		PROBLEMA	SOLUÇÃO POSSÍVEL
OvEr (Sobre)		<ul style="list-style-type: none"> <li>• Condutividade é &gt;200,0 mS</li> <li>• Salinidade é &gt; 70,00 ppt</li> </ul>	<ul style="list-style-type: none"> <li>• Mergulhe completamente a sonda.</li> <li>• Aguarde tempo suficiente até o eléctrodo e sonda Temp estabilizarem.</li> <li>• Recalibre com o valor correcto do padrão da condutividade.</li> <li>• Substitua o padrão de condutividade.</li> <li>• Limpe a célula.</li> <li>• Regressar ao serviço.</li> </ul>
OvEr/Undr (Sobre/Sub) durante a calibração		A Calibração da constante da célula está fora da amplitude	
ECRÃ PRINCIPAL	ECRÃ SECUNDÁRIO		
OvEr/Undr (Sobre/Sub)	OvEr (Sobre)	Temperatura >90,0 °C	<ul style="list-style-type: none"> <li>• Diminua/Aumente a temperatura da amostra.</li> <li>• Regressar ao serviço.</li> </ul>
	Undr (Sub)	Temperatura < -10,0 °C	

## ESPECIFICAÇÕES

Ecrã	Amplitude	Precisão	Resolução
Conductividade, Auto-amplitude	0,0 a 499,9 uS/cm 500 a 4999 uS/cm 5,00 a 49,99 mS/cm 50,0 a 200,0 mS/cm	±1% da leitura mais 2 uS/cm ±1% da leitura mais 5 uS/cm ±1% da leitura mais 0,05 uS/cm ±2,5% da leitura mais 0,5 mS/cm	0,01 uS/cm 1 mS/cm 0,01 mS/cm 0,1 mS/cm
Salinidade	0,0 a 70,0 ppt	0,2% Escala completa	0,1 ppt
Temperatura °C	-10,0 a 90 °C	±0,2 °C ou ±0,4% Escala completa, o que for superior.	0,1 °C

Referência da temperatura	15,0 a 25,0 °C
Coefficiente da temperatura	0,0% a 4,0%
Constante da célula	5,00 ± 0,50
Amplitude da constante do TDS	0,30 a 1,00
Energia/Duração da pilha	Uma pilha de 9V, cerca de 500 horas
Cópia da calibração	Sim
Feedback áudio	Sim, em todas as teclas de pressão
Invólucro do instrumento	Impermeável, IP 67
Amplitude da temp. operacional	0 a 50 °C
Amplitude da humidade relativa operacional	Até 95%
Sonda da temperatura	Termistor, 10kΩ / 25 °C
Dimensões (C x L x P)	18,7 cm x 7,6 cm x 3,8 cm (7,37 x 3 x 1,5 pol)
Peso (com pilha)	270 gramas (0,6 lb)

## LISTA DE PEÇAS SOBRESSALENTES RECOMENDADAS

PEÇA N.º	DESCRIÇÃO
300-1	Conjunto de sonda com cabo de 1 m.
300-4	Conjunto de sonda com cabo de 4 m
300-10	Conjunto de sonda com cabo de 10 m
606043	Carregar de lados duro caso
485	Carregar de lados macio caso

Artigo N° 606042REF  
 Revisão A; Julho 2012  
 Para a versão mais recente do manual, visita [www.ysi.com](http://www.ysi.com)







Item #606042REF

Revision A; July 2012

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YSI

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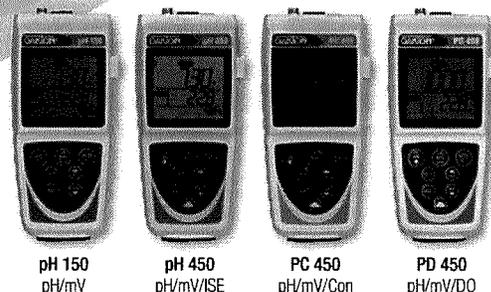
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Email: [environmental@ysi.com](mailto:environmental@ysi.com); Website: [www.yisi.com](http://www.yisi.com)

# 150 & 450 Series Waterproof Handheld Meters pH/mV Operation Instructions

**OAKTON®**

## Models:



## pH Buffer Options

- Select the desired pH Buffer Calibration Group:
  - USA** (1.68, 4.01, 7.00, 10.01, 12.45) or
  - NIST** (1.68, 4.01, 6.86, 9.18, 12.45) or
  - DIN** (1.09, 3.06, 4.65, 6.79, 9.23, 12.75) or
  - MAN** (manual adjustment of any custom pH values that are  $\geq 1$  pH unit apart, 450 series only)
- Select number of calibration points
- Select Calibration Due Reminder**
  - Set number of days from 0-60 for desired parameter
- View Calibration Data**
  - Press **ENTER** to view each point that is calibrated.
- View Electrode Data**
  - Press **ENTER** to view mV **Offset** and **Slope %** of the measured reading.

## System Settings

- Data Logging:**
  - MANUAL** upon key press only
  - TIMED** interval. Choose (SEC / MIN / HOUR) interval.
- Automatic shut off after 10 minutes. Choose **ON** or **OFF**.
- Clock Settings:**
  - Date: Choose **USA** (MM/DD/YYYY) or **Euro** (DD/MM/YYYY).
  - Time: Choose (24HR or 12HR). If 12HR, choose **AM** or **PM**.
- Set Printer Type:**
  - CSV** (Comma Separated Values) – best format for computer
  - Printer** (Text) – best format for printer.
  - Choose **Manual (MAN)** upon key press or **TIMED** interval.
  - If timed, choose (SEC / MIN / HOUR).

## Reset

- NO**. Exits from reset menu options without action.
- FACTORY RESET**. Returns all settings except date/time and ATC calibration to factory default values after **ENTER** is pressed then restarts meter.
- DATA RESET**. Erases data stored in memory while retaining other settings after **ENTER** is pressed.
- CALIBRATION RESET**. Erases non-ATC calibration data while retaining other settings after **ENTER** is pressed.

## pH Calibration

For best results, periodic calibration with known, accurate standards is recommended. Calibrate with standards that bracket your intended measuring range while including a neutral standard (pH 7.00 or 6.86). For example, if you expect to measure samples from pH 6.2 to pH 9.5, calibration with 4.01, 7.00, and 10.01 standards will work well. Provide stirring for best results. After calibration with two or more points, the active slope segment of the measurement will be visible on the bottom display during measurement. 100 % slope will be shown if only one calibration point is performed and " - - " if no calibration is performed. The meter will automatically return to measurement mode upon successful completion of the number of specified calibration points. To specify a different number of pH calibration points, see **pH Buffer Options**.

## Using Automatic Buffer Recognition

- While in pH measurement mode, dip the pH and ATC sensor(s) into your first standard, then press **CAL**. The primary display will search for the nearest standard value, while the secondary display will show the un-adjusted value.
- When the "READY" indicator appears, press **ENTER** to accept. The primary reading will flash "DONE".
- Rinse your electrode(s) then dip into the next pH standard. The primary display will search for the nearest standard value that has not yet been calibrated, while the secondary display will show the unadjusted value. When the "READY" indicator appears, press **ENTER** to accept.
- To calibrate another pH standard repeat Step 3 or press **MEAS** to return to pH measurement mode.

## Using Manual Recognition / Custom Buffers (450 Series Only)

- While in pH measurement mode, dip the pH and ATC sensor(s) into your first standard then press **CAL**.
- When the **READY** indicator appears, use up/down arrows to adjust the primary reading to match the standard value at the measured temperature, then press **ENTER**.
- Rinse your electrode(s) then repeat Step 2 with a standard that is  $\geq 1$  pH unit from the previous standard value.
- To calibrate another pH standard, repeat Step 3 or press **MEAS** to return to pH measurement mode.

## mV Offset Adjustment

- While in mV measurement mode, dip the ORP and ATC sensors into a solution with a known mV value (i.e. Zobel, Light's, quinhydrone, or iodide/triiodide) and stir.
- When the "READY" indicator appears, use up/down arrows to adjust the primary reading to match the mV value at the measured temperature, then press **ENTER**. The meter allows an adjustable maximum value of  $\pm 200$  mV from the factory default mV value. When an offset has been stored successfully, R.mV replaces mV.

## Temperature Calibration/Manual ATC

- Press **CAL** from any measurement, then press **MODE**.
- Skip to step 3 for manual ATC, otherwise, dip the temperature sensor into a solution with a known accurate temperature. The upper display shows the active temperature while the lower display shows the factory default temperature without adjustment.
- Use up/down arrows to adjust the upper display. Press **ENTER** to accept the calibration temperature. The maximum adjustable value is  $\pm 10$  °C (or  $\pm 18$  °F) from factory default.

## Error Messages

- ERR** "ERR" will appear when an error condition exists or the incorrect key is pressed. Common examples include:
- Pressing **ENTER** during calibration before the "READY" indicator appears. Wait for the "READY" indicator before pressing **ENTER**.
  - UR** (Under Range) • **OR** (Over Range)

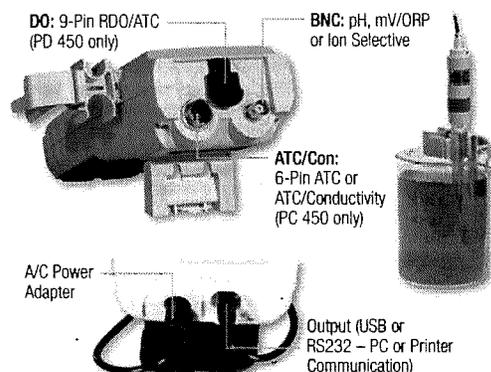
## Intended Use, Maintenance & Precautions

- These handheld meters use sensors to detect various parameters for water-based measurements. For routine maintenance disconnect the power cord or battery, then dust or wipe the display using a damp cloth. If necessary, warm water or a mild water based detergent can be used. Immediately remove any spilled substance from contact with the meter using the proper cleaning procedure for the type of spill.
- Do not use this equipment in potentially explosive atmospheres.
  - Refer to the electrode instructions for use, storage and cleaning.
  - Ensure that no liquid enters the instrument.
  - Do not use any aggressive cleaning chemicals (solvents or similar agents).
  - There are no user serviceable parts inside. Attempts to service internal parts may void the warranty.
  - Not intended for medical applications or patient use.
  - WARNING:** No modification of this equipment is allowed.

Instrument Operating Conditions	
Operating Ambient Temp.	5 to 45 °C
Operating Relative Humidity	5 to 85 %, non-condensing
Storage Temp.	-20 to +60 °C
Storage Relative Humidity	5 to 85 %, non-condensing
Pollution	Degree 2
Overvoltage	Category II
Weight	500 g
Size (L x W x H)	21.15 x 9.87 x 5.85 cm
Regulatory & Safety	CE, TUV 3-1, FCC Class A
Power Rating	DC Input: 9 VDC 1 A
Battery Requirement	2 x AA (LR6) 1.5 V batteries (replace batteries when battery sign blinks)
Vibration	Shipping/handling per ISTA #1A
Shock	Drop test in packaging per ISTA #1A
Enclosure (Designed To Meet)	IP67 (using rubber covers)
Universal Power Adapter Operating Conditions	
Operating Ambient Temp.	0 to 50 °C
Operating Relative Humidity	0 to 90 %, non-condensing
Storage Temp.	-20 to +75 °C
Storage Relative Humidity	0 to 90 %, non-condensing
Pollution	Degree 2
Overvoltage	Category II
Power Rating	I/P: 100 - 240 V, 50/60 Hz, 0.3A O/P: 9 VDC 1 A

## Getting Started/Connections

After installing (2) AA batteries and/or connecting the optional 110/220 VAC power supply, connect the desired sensors to the corresponding ports.



12 mm and 16 mm probes can utilize the **Grip-Clip™** to attach one or more sensors to a beaker and to the instrument as needed. The stand can be extended as shown above or used for wall-mounting.

## Keypad Functions

	Press <b>once</b> to power <b>ON</b> in the mode that was previously used. Press <b>again</b> to turn backlight on for one minute or off (450 series only). Hold for 3 seconds to power <b>OFF</b> .
	Toggle between measurement and calibration modes. In <b>SETUP</b> mode, <b>BACK</b> serves to return to the previous menu option or setting.
	Confirm calibration values in <b>CAL</b> mode. Confirm selections in <b>SETUP</b> mode. Freeze or release the measured reading.
	Customize instrument settings and preferences. (See also <b>Setup Programs</b> )
	Toggle between available measurement types.
	Save measurement into memory. Increase value or scroll up in <b>SETUP</b> or manual calibration.
	Recall saved values from memory. Decrease value or scroll down in <b>SETUP</b> or manual calibration.
	Send output data to printer or computer. (450 series only).

## Setup Programs

To access the settings below, press **SETUP**. Up/down arrows will display the available options. Press **ENTER** to accept the desired setting, or **BACK** to return to the previous option and/or exit.

## Configuration Options

- Ready indicator **ON / OFF** / or Automatic **HOLD** when stable
- Choose °Celsius or °Fahrenheit

## Oakton Instruments

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Y S I Environmental

## YSI 556 Multiparameter System

*Versatile, multiparameter handheld instrument*

Rugged and reliable, the YSI 556 MPS (Multiprobe System) combines the versatility of an easy-to-use, easy-to-read handheld unit with all the functionality of a multiparameter system.



*The 556 has multiple language capabilities and graphing!*

- Simultaneously measures dissolved oxygen, pH, conductivity, temperature, and ORP
- Field-replaceable electrodes
- Compatible with EcoWatch<sup>®</sup> for Windows<sup>®</sup> data analysis software
- Stores over 49,000 data sets, time and date stamped, interval or manual logging
- Three-year warranty on the instrument; one-year on the probes
- GLP assisting, records calibration data in memory
- Available with 4, 10, and 20-m cable lengths
- IP-67, impact-resistant, waterproof case
- Easy-to-use, screw-on cap DO membranes
- RS-232 interface for PC connection

### **Options to Fit Your Applications!**

- **Battery Options** – The unit is powered by alkaline batteries or an optional rechargeable battery pack with quick-charge feature.
- **Optional Barometer** – Internal barometer can be user-calibrated and displayed along with other data, used in dissolved oxygen calibrations, and logged to memory for tracking changes in barometric pressure. (Choose 556-02)
- **Optional Flow Cell** - The 5083 flow cell can be used for ground water applications or anytime water is pumped for sampling.
- **Carrying Case** – The instrument comes standard with YSI 5061, a soft-sided carrying case with enough space for the 556, a 20-meter cable, and calibrating supplies. An optional 5080 hard-sided carrying case is also available.
- **Confidence Solution<sup>®</sup>** - Quality assurance ensured. Quickly check conductivity, pH, and ORP readings with one solution.

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*A rugged, cost-effective  
multiparameter handheld  
system designed for the field!*



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## 5563 MPS Sensor Specifications

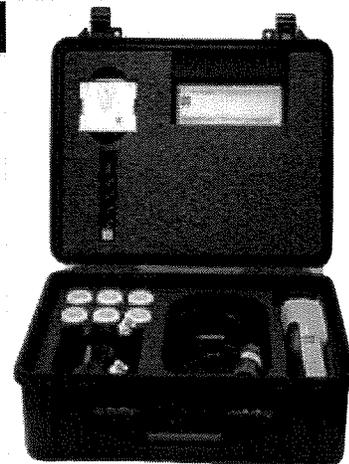
Dissolved Oxygen (% saturation)	Sensor Type Range Accuracy Resolution	Steady state polarographic 0 to 500% air saturation 0 to 200% air saturation, $\pm 2\%$ of the reading or $\pm 2\%$ air saturation, whichever is greater; 200 to 500% air saturation, $\pm 6\%$ of the reading 0.1% air saturation
Dissolved Oxygen (mg/L)	Sensor Type Range Accuracy Resolution	Steady state polarographic 0 to 50 mg/L 0 to 20 mg/L, $\pm 2\%$ of the reading or $\pm 0.2$ mg/L, whichever is greater; 20 to 50 mg/L, $\pm 6\%$ of the reading 0.01 mg/L
Temperature	Sensor Type Range Accuracy Resolution	YSI Temperature Precision <sup>™</sup> thermistor -5 to 45°C $\pm 0.15^\circ\text{C}$ 0.1°C
Conductivity	Sensor Type Range Accuracy Resolution	4-electrode cell with autoranging 0 to 200 mS/cm $\pm 0.5\%$ of reading or $\pm 0.001$ mS/cm; whichever is greater (4-meter cable) $\pm 1.0\%$ of reading or $\pm 0.001$ mS/cm; whichever is greater (20-meter cable) 0.001 mS/cm to 0.1 mS/cm (range-dependent)
Salinity	Sensor Type Range Accuracy Resolution	Calculated from conductivity and temperature 0 to 70 ppt $\pm 1.0\%$ of reading or $\pm 0.1$ ppt, whichever is greater 0.01 ppt
pH (optional)	Sensor Type Range Accuracy Resolution	Glass combination electrode 0 to 14 units $\pm 0.2$ units 0.01 units
ORP (optional)	Sensor Type Range Accuracy Resolution	Platinum button -999 to +999 mV $\pm 20$ mV 0.1 mV
Total Dissolved Solids (TDS)	Sensor Type Range Resolution	Calculated from conductivity (variable constant, default 0.65) 0 to 100 g/L 4 digits
Barometer (optional)	Range Accuracy Resolution	500 to 800 mm Hg $\pm 3$ mm Hg within $\pm 10^\circ\text{C}$ temperature range from calibration point 0.1 mm Hg

## YSI 556 Instrument Specifications

Size	11.9 cm width x 22.9 cm length (4.7 in. x 9 in.)
Weight with batteries	2.1 lbs. (916 grams)
Power	4 alkaline C-cells; optional rechargeable pack
Cables	4-, 10-, and 20-m (13.1, 32.8, 65.6 ft.) lengths
Warranty	3-year instrument; 1-year probes and cables
Communication Port	RS-232 Serial
Data Logger	49,000 data sets, date and time stamp, manual or logging, with user-selectable intervals

## 556 Ordering Information (Order all items separately)

556-01	Instrument (with 5061 large, soft-sided carrying case)
556-02	Instrument with barometer option (with 5061 carrying case)
5563-4	4-m cable and DO/temp/conductivity
5563-10	10-m cable and DO/temp/conductivity
5563-20	20-m cable and DO/temp/conductivity
5564	pH Probe for any 5563 cable
5565	pH/ORP Probe for any 5563 cable
6118	Rechargeable battery pack kit (includes battery, adapter, charger)
614	Ultra clamp, C-clamp mount
616	Charger, cigarette lighter
4654	Tripod (small tripod for instrument)
5060	Small carrying case, soft-sided (fits instrument and 4-m cable)
5065	Form-fitted carrier with shoulder strap
5080	Small carrying case, hard-sided (fits instrument, 4-m cable, flow cell, batteries, membrane kit, calibration bottles)
5083	Flow cell
5085	Hands-free harness
5580	Confidence Solution <sup>™</sup> (insure probe accuracy with a simple field-check for conductivity, pH, and ORP)



The 5080 carrying case with 556, 5563-4 cable, and 5083 flow cell.



*YSI incorporated*



**YSI Model 63**  
**Handheld pH,**  
**Conductivity,**  
**Salinity and**  
**Temperature**  
**System**  
**Operations**  
**Manual**



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# 1. Introduction

---

The YSI Model 63 Handheld pH, Conductivity, Salinity and Temperature System is a rugged, micro-processor based, digital meter with an attached pH, conductivity and temperature probe. The pH sensor can be easily replaced in the field.

The Model 63 has a non-detachable cable available in lengths of 10, 25, 50 or 100 feet (3, 7.6, 15.2 or 30.5 meters). The probe body has been manufactured with stainless steel to add rugged durability and sinking weight.

The YSI Model 63 has the following features:

- Capability to measure at depths of up to 100 feet (30.5 meters)
- Microprocessor control
- Field replaceable low maintenance pH sensor
- Push-button calibration
- Simultaneous display of pH, conductivity or salinity and temperature
- Automatic temperature compensation for conductivity readings
- Autoranging
- Data storage for 50 sets of readings with on screen recall
- Waterproof case (IP65)

The Model 63's micro-processor allows the system to be easily calibrated with the press of a few keys. Additionally, the micro-processor performs a self-diagnostic routine each time the instrument is turned on. The self-diagnostic routine provides useful information about the function of the instrument and probe.

A transport chamber, built into the instrument case, provides a convenient place to store the probe when transporting the system. The Model 63 case is waterproof (rated to IP65) allowing operation in the rain without damage to the instrument.

The Model 63 is powered by six AA-size alkaline batteries. A new set of alkaline batteries will provide approximately 100 hours of continuous operation. When batteries need to be replaced, the LCD will display a **“LO BAT”** message.

The YSI Model 63 is designed for use in environmental, aquaculture, and industrial applications where accurate pH, conductivity, salinity and temperature measurements are desired.

## 2. Preparing the Meter

---

### 2.1 Unpacking

When you unpack your new YSI Model 63 Handheld pH, conductivity, salinity and Temperature System for the first time, check the packing list to make sure you have received everything you should have. If there is anything missing or damaged, call the dealer from whom you purchased the system. If you do not know which of our authorized dealers sold the system to you, call YSI Customer Service at 800-765-4974 or 937-767-7241, and we'll be happy to help you.

### 2.2 Warranty Card

Please complete the Warranty Card and return it to YSI. This will record your purchase of this instrument in our computer system. Once your purchase is recorded, you will receive prompt, efficient service in the event any part of your YSI Model 63 should ever need repair.

### 2.3 Batteries

There are a few things you must do to prepare your YSI Model 63 for use. First, locate the six AA-size alkaline batteries and the battery cover kit which were included. Then locate the markings inside each of the two battery-chamber sleeves that illustrate the correct way to install the batteries. Install the batteries as shown.

**NOTE:** It is very important that the batteries be installed ONLY as illustrated. The instrument will not function and may be damaged if the batteries are installed incorrectly.

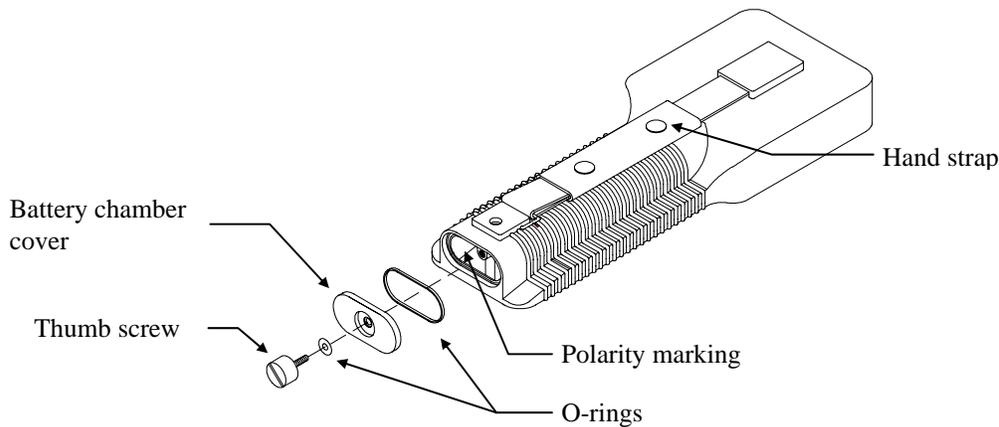


Figure 1

Attach the battery chamber cover to bottom of the instrument using the thumb screw as shown in Figure 1. Make sure that the o-rings are in place. The battery-chamber cover is marked with the words "OPEN" and "CLOSE."

Turn the instrument on by pressing and releasing the **ON/OFF** key on the front of the instrument. The liquid crystal display (LCD) should come on. Allow a few seconds for the instrument to complete its diagnostic routine. If the instrument does not operate, consult the chapter entitled *Troubleshooting*.

You may also want to take the instrument into a dark location and, with the instrument ON, hold down the **LIGHT** key. The instrument back-light should illuminate the LCD so that the display can be easily read.

## 2.4 Transport Chamber

The Model 63 has a convenient transport chamber built into the instrument's side. This chamber provides a storage area and protection for the probe while transporting the system in the field. Insert the round sponge (provided with the Model 63) into the bottom of the chamber. Put 6-8 drops of tap water into the sponge. The wet sponge creates a humid environment for the pH sensor to prevent it from drying out during transport in the field (up to one week). The transport chamber is NOT intended for long term storage of the pH sensor. See 6.3 *pH Sensor Storage*.

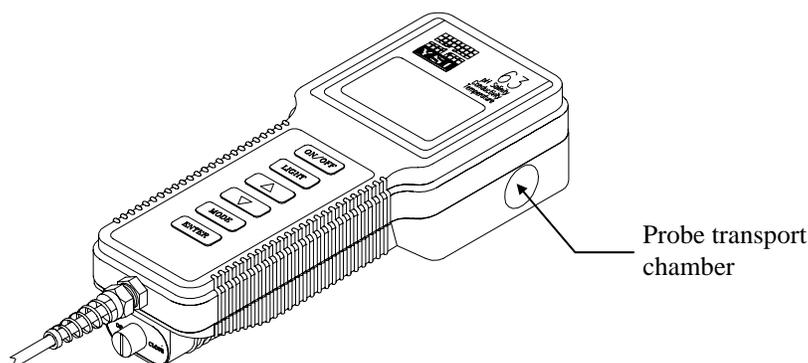


Figure 2

## 2.5 Hand Strap

The hand strap (see Figure 1 on previous page) is designed to allow comfortable operation of the Model 63 with minimum effort. If the hand strap is adjusted correctly, it is unlikely that the instrument will be easily dropped or bumped from your hand.

To adjust the hand strap on the back of the meter, unsnap the vinyl cover and pull the two Velcro strips apart. Place your hand between the meter and the strap and adjust the strap length so that your hand is snugly held in place. Press the two Velcro strips back together and snap the vinyl cover back into place.

## 2.6 The Meter Case

The meter case is sealed at the factory and is not intended to be opened, except by authorized service technicians. **Do not attempt to separate the two halves of the meter case as this may damage the instrument, break the water-resistant seal and may void the manufacturer's warranty.**

## 2.7 Calibration Vessels

To do a calibration you will need a plastic 100 mL graduated cylinder. A plastic container is provide with the Model 63. The graduated cylinder provides a convenient place to calibrate the pH sensor minimizing the amount of solution needed. The plastic container can be used as a conductivity calibration vessel or filled with distilled water and used as a rinse vessel while in the field. See section 4.2 *pH Calibration* and section 4.3 *Conductivity Calibration* for details.

## 3. Preparing the Probe

---

The YSI Model 63 is shipped without the pH sensor installed. The pH sensor must be installed before using the system (see section 3.1 *Installing the pH Sensor*, below). The sensor is shipped with a protective bottle filled with a mixture of pH 4 buffer and KCl solution. Do not remove the bottle until you are ready to use the instrument. Save the bottle for long term storage of the probe.

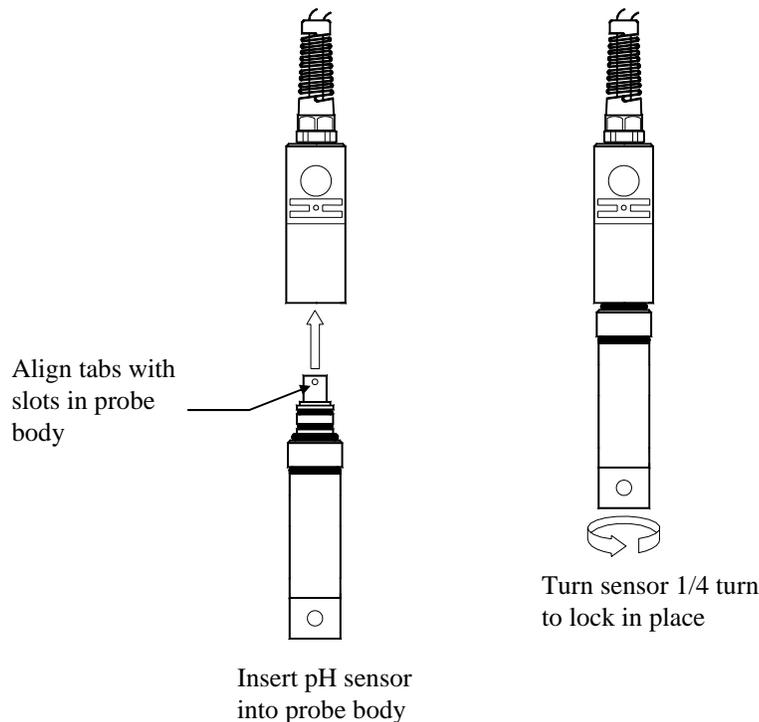
### 3.1 *Installing the pH Sensor*

A pH sensor is included with the Model 63. Install the pH sensor as follows:

1. Remove the sensor from its protective packing.
2. Insert the pH sensor into the probe body (be sure to align the tabs on the sensor with the slots in probe body) and twist 1/4 turn to lock in place. See *Figure 3*.

NOTE: Once installed, leave the pH sensor attached to the probe until replacement is needed.

3. Carefully remove the protective bottle (containing pH 4 buffer/KCl solution) from the sensor. Save the bottle and solution for long term (more than 1 week) storage of the sensor. Seal the storage bottle with the cap provided.
4. Rinse the sensor tip with distilled or deionized water.
5. **Calibrate the system before use.** See section 4.2 *pH Calibration*.



*Figure 3*

## 4. Operation

---

The following diagram is an overview of the operation of the Model 63. See the following sections for details of operation.

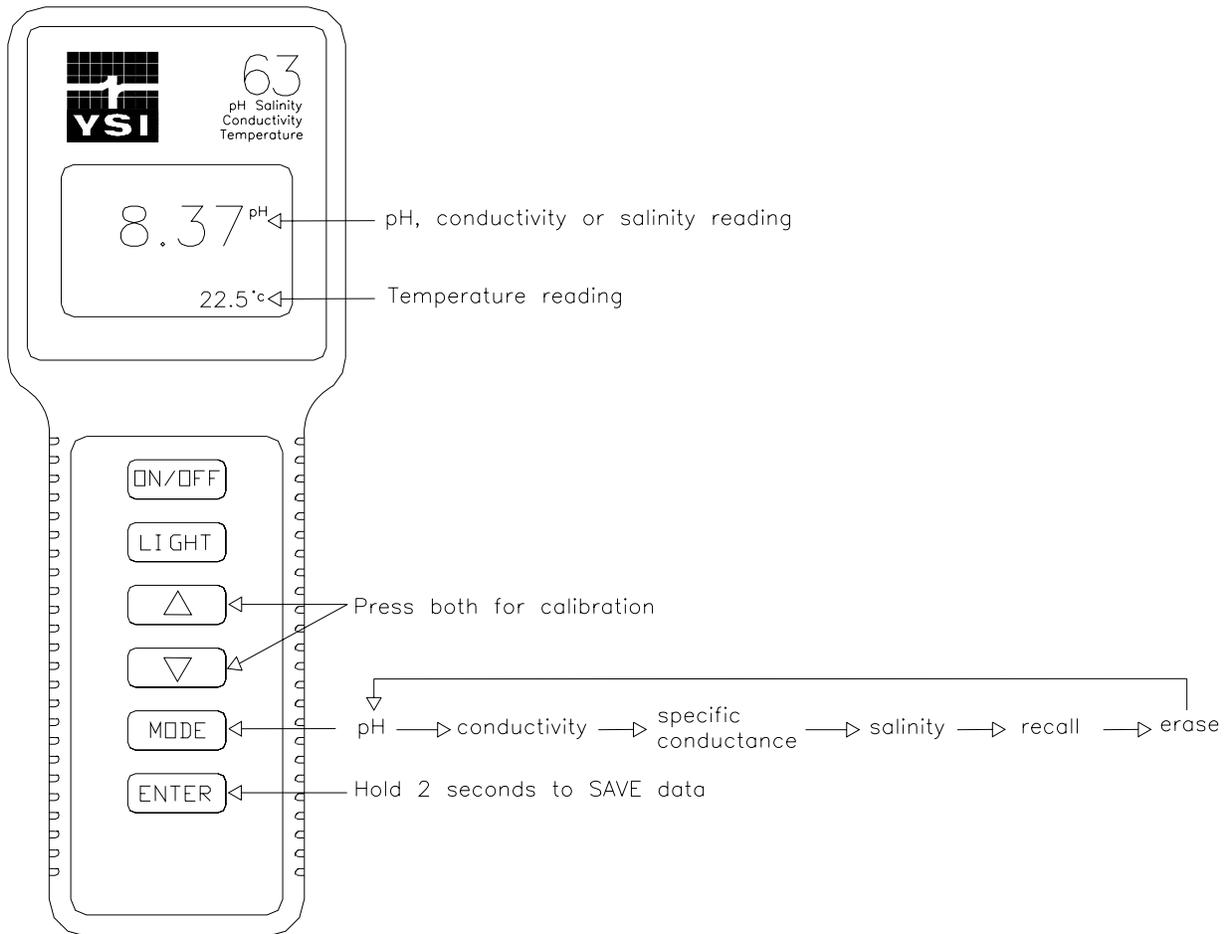


Figure 4

## 4.1 Turning The Instrument On

With the batteries installed correctly, press the **ON/OFF** key. The instrument will activate all segments of the display for a few seconds, which will be followed by a self test procedure which will last for several more seconds. The Model 63 will briefly display the cell constant of the conductivity probe when the self test is complete. During this power on self test sequence, the instrument's microprocessor is verifying that the system is working properly. If the instrument were to detect a problem, a **continuous** error message would be displayed. See the chapter entitled *Troubleshooting* for a list of error messages.



## 4.2 pH Calibration

The YSI Model 63 **MUST** be calibrated before making pH measurements. Calibration may be performed at 1, 2 or 3-points (at pH 7, 4 and 10, or at pH 6.86, 4.01 and 9.18). Perform a 1-point calibration (at pH 7 or at pH 6.86) **ONLY** if a previous 2 or 3-point calibration has been performed recently. In most cases, a 2-point pH calibration will be sufficient for accurate pH measurements, but if the general range of pH in the sample is not known, a 3-point calibration may be necessary. 3-point calibration assures accurate pH readings regardless of the pH value of the sample. See 9.1 *pH* for more details.

**WARNING:** Calibration reagents may be hazardous to your health. Refer to *Appendix B - Health and Safety* for more information.

**Before calibrating the YSI Model 63, complete the procedures discussed in the *Preparing the Meter* and *Preparing the Probe* chapters of this manual.**

The user can choose from two sets of pH buffer values for 3-point calibration. The first set consists of the standard YSI pH buffer values of pH 7 (YSI 3822), pH 4 (YSI 3821) and pH 10 (YSI 3823). The second set available is the NIST pH 6.86, 4.01 and 9.18. **Note that the first calibration point must be either pH 7 or pH 6.86.** Calibration is performed as follows:

1. Turn the instrument on by pressing the **ON/OFF** key. Press the **MODE** key until pH is displayed.
2. Rinse the probe with deionized or distilled water, then carefully dry the probe (or rinse it with some of the pH buffer solution to be used for calibration).
3. Place 30 to 35 mL of the pH buffer you have chosen to calibrate the system with (pH 7 or 6.86) in the 100 mL graduated cylinder. The graduated cylinder minimizes the amount of solution needed. Immerse the probe making sure that both the pH and temperature sensors are covered by the solution (see *Figure 5* on the following page).

**For best results:**

- Calibrate as close as possible to the sample temperature.
- After storage in pH 4 buffer/KCl solution, place the pH sensor in pH 7 (6.86) buffer and allow to acclimate before calibrating (5 to 10 minutes).
- Always give the pH and temperature sensors enough time to equilibrate with the temperature of the buffer.

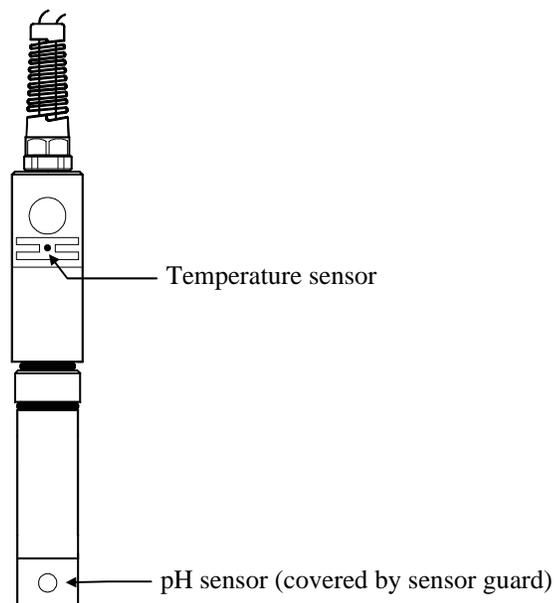
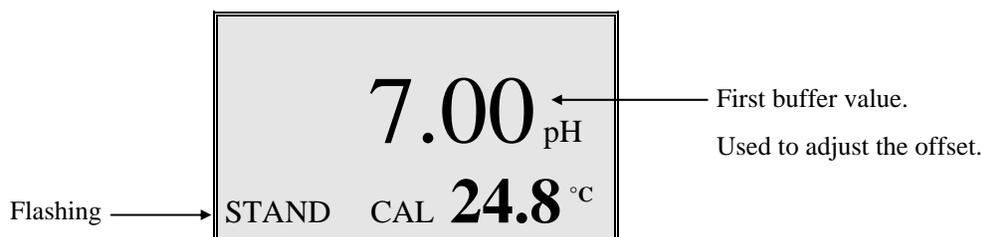


Figure 5

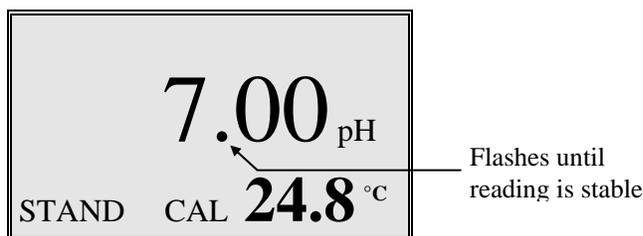
4. To enter the calibration menu, use two fingers to press and release both the **UP ARROW** and **DOWN ARROW** keys at the same time. The Model 63 display will show **CAL** at the bottom, **STAND** will be flashing and the pH reading will show **7.00** (the buffer to be used to adjust the offset).



NOTE: If you will be calibrating with pH buffers of 6.86, 4.01 and 9.18 (instead of 7, 4 and 10), press both the **UP ARROW** and **DOWN ARROW** keys again. The display will change to **6.86**.

NOTE: The Model 63 automatically accounts for the fact that the true pH of the buffers changes with temperature, therefore, the pH values displayed during calibration will vary with temperature. For example, pH 7 buffer at 20°C (rather than 25°C) has an actual pH of 7.02 and this number (rather than 7.00) will appear on the display when the probe is placed in the solution. See *Appendix C - pH Buffer Values*.

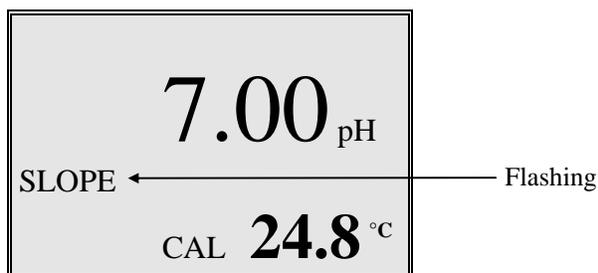
5. Press the **ENTER** key. The Model 63 display will show **CAL** at the bottom, **STAND** will stop flashing and the pH calibration value is shown with the middle decimal point flashing.



6. When the reading is stable (does not change by 0.01 pH in 10 seconds), the decimal point will stop flashing. Press and hold the **ENTER** key to save the calibration point. The Model 63 will flash **SAVE** on the display along with **OFS** to indicate that the offset value has been saved.



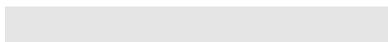
7. **SLOPE** will now appear on the display and be flashing. This indicates that the slope is ready to be set using a second pH buffer. The system is now calibrated at a single point. If you are only performing a single point calibration, press the **MODE** key to return to normal operation.

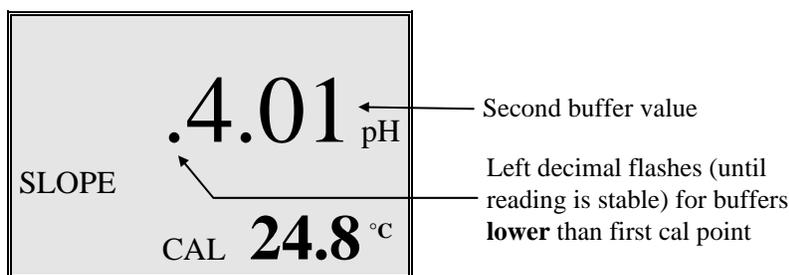


8. Rinse the probe with deionized or distilled water, then carefully dry the probe.

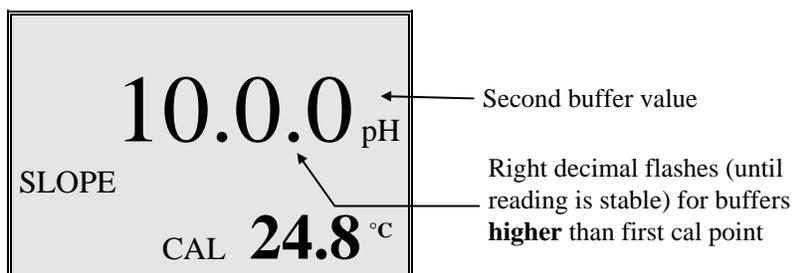
**STOP HERE IF PERFORMING A 1-POINT CALIBRATION.**

9. If you are performing a 2-point or 3-point calibration, fill a clean container with the second value pH buffer (pH 4 or 10, or pH 4.01 or 9.18) and immerse the probe into the solution. Make sure that the temperature sensor is immersed.
10. Press the **ENTER** key. The Model 63 should now show **CAL** at the bottom, **SLOPE** will stop flashing and the pH calibration value (automatically sensed by the instrument) is shown with one of the decimal points flashing.





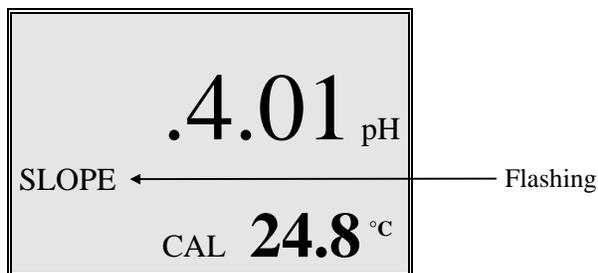
If the second pH buffer is less than the first buffer (which was used to adjust the offset; pH 7 or pH 6.86), the left decimal point will flash as shown above. If the second pH buffer is greater than the first, the right decimal point will flash as shown below.



11. When the reading is stable (does not change by 0.01 pH in 10 seconds), the decimal point will stop flashing. Press and hold the **ENTER** key to save the first SLOPE. The Model 63 will flash **SAVE** on the display along with **SLP** to indicate that the first slope value has been saved.



12. **SLOPE** will start flashing again indicating that the slope is ready to be set using a third pH buffer.



13. The system is now calibrated at two points. If you are only performing a two point calibration, press the **MODE** key to return to normal operation.

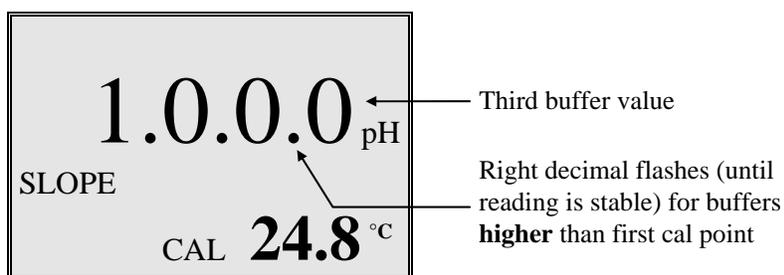
14. Rinse the probe with deionized or distilled water, then carefully dry the probe.

**STOP HERE IF PERFORMING A 2-POINT CALIBRATION.**

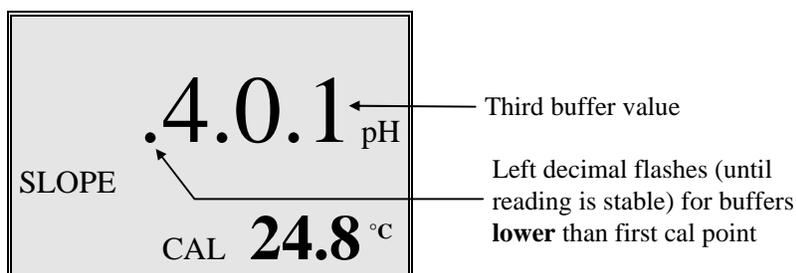
15. If you are performing a 3-point calibration, fill a clean container with the third value pH buffer (pH 4 or 10, or pH 4.01 or 9.18) and immerse the probe into the solution. Make sure that the temperature sensor is immersed.

NOTE: The third buffer must not be the same as the second buffer. For example; if the second buffer was less than pH 7, the third buffer must be greater than pH 7.

16. Press the **ENTER** key. The Model 63 display will now show **CAL** at the bottom, **SLOPE** will stop flashing and the pH calibration value (automatically sensed by the instrument) is shown with one of the decimal points flashing. If the third pH buffer is less than the first buffer (which was used to adjust the offset; usually pH 7), the left decimal point will flash. If the third pH buffer is greater than the first, the right decimal point will flash.



or



17. When the reading is stable (does not change by 0.01 pH in 10 seconds), the decimal point will stop flashing. Press and hold the **ENTER** key to save the second SLOPE. The Model 63 will flash **SAVE** on the display along with **SLP** to indicate that the second slope value has been saved.



The system is now calibrated at three points and will return to normal operation.

18. Rinse the probe with deionized or distilled water.

### 4.3 Conductivity Calibration

**IMPORTANT:** System calibration is rarely required because of the factory calibration of the YSI Model 63. However, from time to time it is wise to check the system calibration and make adjustments when necessary.

**Prior to calibration of the YSI Model 63, it is important to remember the following:**

1. Always use clean, properly stored, NIST traceable calibration solutions (see *12 Accessories and Replacement Parts*). When filling a calibration container prior to performing the calibration procedures, make certain that the level of calibrant buffers is high enough in the container to cover the entire probe. Gently agitate the probe to remove any bubbles in the conductivity cell.
2. Rinse the probe with distilled water (and wipe dry) between changes of calibration solutions.
3. During calibration, allow the probe time to stabilize with regard to temperature (approximately 60 seconds) before proceeding with the calibration process. The readings after calibration are only as good as the calibration itself.
4. Perform conductivity calibration at a temperature as close to 25°C as possible. This will minimize any temperature compensation error.

**Follow these steps to perform an accurate calibration of the YSI Model 63:**

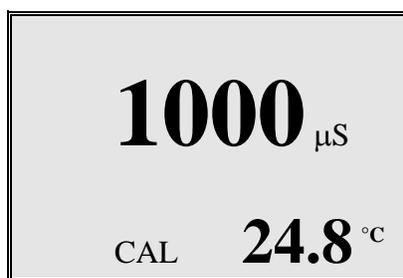
1. Turn the instrument on and allow it to complete its self test procedure.
2. Select a calibration solution which is most similar to the sample you will be measuring.
  - For sea water choose a 50 mS/cm conductivity standard (YSI Catalog# 3169)
  - For fresh water choose a 1 mS/cm conductivity standard (YSI Catalog# 3167)
  - For brackish water choose a 10 mS/cm conductivity standard (YSI Catalog # 3168)
3. Place at least 7 inches of solution in the plastic container or a clean glass beaker.

**NOTE: Do NOT use the 100 mL graduated cylinder.** The diameter of the cylinder is too small for accurate conductivity measurements.

4. Use the **MODE** key to advance the instrument to display conductivity.
5. Insert the probe into the solution deep enough to completely cover the probe. Both conductivity ports must be submerged (see *Figure 6* on the following page).
6. Allow at least 60 seconds for the temperature reading to become stable.
7. Move the probe vigorously from side to side to dislodge any air bubbles from the electrodes.
8. Press and release the **UP ARROW** and **DOWN ARROW** keys at the same time.

The **CAL** symbol will appear at the bottom left of the display to indicate that the instrument is now in Calibration mode.



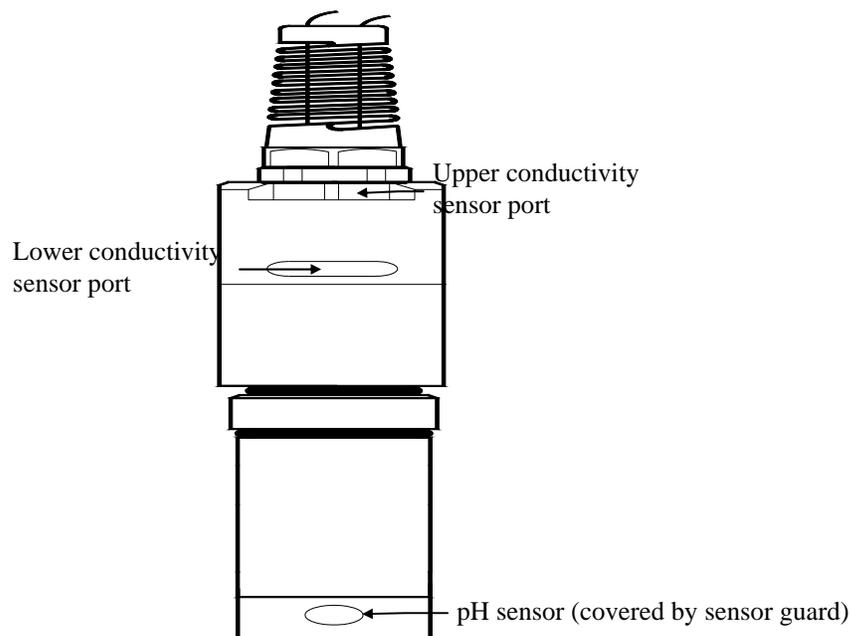


9. Use the **UP ARROW** or **DOWN ARROW** key to adjust the reading on the display until it matches the value of the calibration solution you are using.
10. Once the display reads the exact value of the calibration solution being used (the instrument will make the appropriate compensation for temperature variation from 25°C), press the **ENTER** key. The word “**SAVE**” will flash across the display for a second indicating that the calibration has been accepted.

The YSI Model 63 is designed to retain its last conductivity calibration permanently. Therefore, there is no need to calibrate the instrument after battery changes or power down.

#### **4.4 Making Measurements**

After the system has been set-up and pH has been calibrated as described in *4.2 pH Calibration*, it is ready to make measurements. Simply insert the probe into the sample, shake gently to remove any trapped air bubbles and wait for the readings to stabilize (approximately 60 seconds). The first pH reading after storage in buffers may take longer to stabilize (5 to 10 minutes), therefore, the probe should be stored in the transport chamber when making field measurements. It is important that the probe be inserted into the sample far enough so that the pH, temperature and conductivity sensors are covered by the liquid (see *Figure 6*).

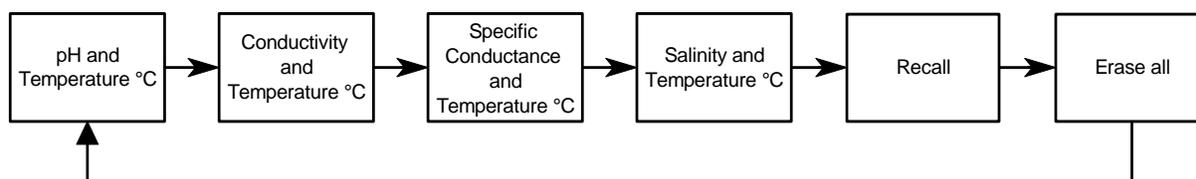


*Figure 6*

The Model 63 has six modes:

- **pH** -- Displays pH and temperature (°C).
- **Conductivity** -- A measurement of the conductive material in the liquid sample without regard to temperature. Also displays temperature (°C).
- **Specific Conductance** -- Also known as temperature compensated conductivity which automatically adjusts the reading to a calculated value which would have been read if the sample had been at 25° C (or some other reference temperature which you choose). See section 5 *Advanced Conductivity Setup*. Also displays temperature (°C).
- **Salinity**-- A calculation done by the instrument electronics, based upon the conductivity and temperature readings. Also displays temperature (°C).
- **Recall** -- Allows previously stored data to be displayed.
- **Erase all** -- Allows ALL previously stored data to be deleted.

To change between the Model 63 modes, simply press and release the **MODE** key. The Model 63 will cycle through the modes as follows:

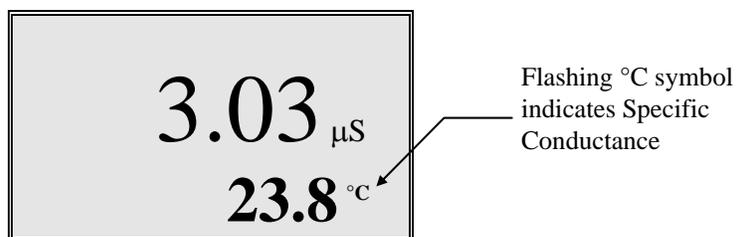


NOTE: When the Model 63 is turned off, it will “remember” which mode you used last and will return to that mode the next time it is turned on. If turned off while in recall or erase mode, it will default to pH mode when turned on.

To determine the current mode of the Model 63, carefully observe the small legends at the far right side of the LCD. If the instrument is reading **pH**, the large numbers on the display will be followed by **pH** as shown below.



If the instrument is reading **Conductivity**, (not temperature compensated) the large numbers on the display will be followed by either a **µS** or an **mS**. Additionally, the small portion of the display will show the ° C **NOT** flashing.



If the instrument is reading **Specific Conductance**, the large numbers on the display will be followed by either a **μS** or an **mS**. Additionally, the small portion of the display will show the **°C** flashing on and off.

If the instrument is reading **Salinity**, the large numbers on the display will be followed by a **ppt**.



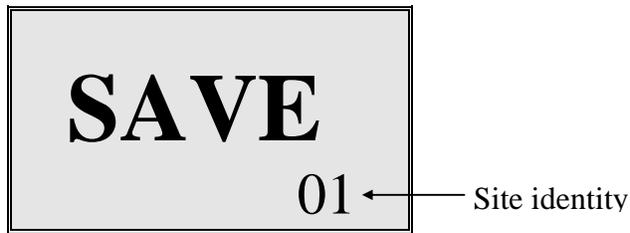
#### **4.5 Autoranging & Range Searching**

The YSI Model 63 is an autoranging instrument. This means that regardless of the conductivity or salinity of the solution (within the specifications of the instrument) all you need to do to get the most accurate reading is to put the probe in the sample. This feature makes the Model 63 as simple as possible to operate.

When you first place the Model 63 probe into a sample or calibration solution, and again when you first remove the probe the instrument will go into a range search mode that may take as long as 5 seconds. During some range searches the instrument display will flash **rANG** to indicate its movement from one range to another. The length of the range search depends on the number of ranges which must be searched in order to find the correct range for the sample. During the range search, the instrument will appear to freeze on a given reading for a few seconds then, once the range is located, will pinpoint the exact reading on the display. The display may also switch to **00.0** for a second or two during a range search before it selects the proper range.

#### **4.6 Saving Data**

The Model 63 is equipped with non-volatile memory that is capable of storing up to 50 different sets of readings. Non-volatile means that you do not need to worry that your data will be lost due to a power failure or interruption, such as when the batteries are removed. Each set consists of pH, conductivity, specific conductance, salinity and temperature. The Model 63 will also assign a site identity number to each set of readings to allow easy review of the data. This feature is useful in situations where transcribing data is difficult or not available.



While pH, conductivity, specific conductance or salinity is displayed on the screen, press the **ENTER** key and hold it for approximately 2 seconds. The meter will flash **SAVE** on the display along with the current site identity (1 through 50) being used.

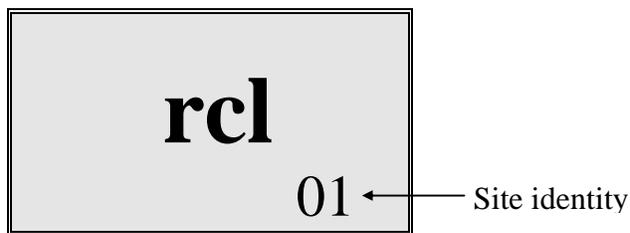
When all 50 sites are full, the display will flash **FULL** on the screen. This message will remain on the screen (even after power down) until a key is pushed.



Once you have acknowledged the memory is full, any subsequent saved data will begin overwriting existing data starting with site #1. No additional warning will be displayed.

#### **4.7 Recalling Stored Data**

1. To put the Model 63 into the **RECALL** mode, press the **MODE** key until “**rcl**” is displayed on the screen along with the site ID number in the lower right corner.



2. Press the **ENTER** key to review the last set of data that was saved. The Model 63 will display the pH and temperature. Another press of the **ENTER** key will display the conductivity and the temperature.
3. Depress the **ENTER** key again and again to review the specific conductance and salinity readings. All readings are displayed with the temperature.
4. Press the **UP ARROW** key to move up through the saved sets of data.
5. Press the **DOWN ARROW** key to move down through the saved sets of data.
6. When the correct Site ID# is displayed, press the **ENTER** key to display the data.

7. When you have finished recalling data, press **MODE** two times to return to normal operation.

**NOTE:** The Model 63 will recall data as a list. When the **UP ARROW** is pressed the Model 63 will display the Site ID# for the previously recorded data. For example: If you are reviewing Site ID# 5 and the **UP ARROW** is pressed, the Model 63 will display Site ID#4. If you are reviewing Site ID# 5 and Site ID# 5 was the last set of data stored, the **DOWN ARROW** key will display Site ID# 1.

Here is an example of the Model 63 memory.

Site ID #1  
Site ID #2  
Site ID #3 ←If the **UP ARROW** key was pressed the Model 63 would display Site ID #2  
Site ID #4  
Site ID #5

#### **4.8 Erasing Stored Data**

1. To erase the data that is stored in the Model 63's memory, press the **MODE** key until the Model 63 displays **ErAS** on the screen.
2. Press and hold the **DOWN ARROW** and **ENTER** keys simultaneously for approximately 5 seconds.



**ErAS**

3. Successful erasure is indicated by the Model 63 displaying **DONE** on the display for 1 to 2 seconds.



**dOnE**

The instrument will automatically change to pH mode after completion and the next saved data will be stored in site ID# 1.

**IMPORTANT:** Data in all 50 site ID's will be erased completely and will be lost forever. Do not use the erase function until all recorded data has been transcribed to an archive outside the Model 63.

## **4.9 Display Backlight**

At times it may be necessary to take measurements with the Model 63 in dark or poorly lit areas. To help in this situation, the Model 63 comes equipped with a backlight which will illuminate the display so that it can be easily read. To activate the backlight, press and hold the **LIGHT** key. The display will remain lit as long as the key is pressed. When you release it, the light goes out to preserve battery life.

## 5. Advanced Conductivity Setup

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The default settings of the YSI Model 63 are appropriate for the vast majority of measurement applications. However, some measurement applications require very specific measurement criteria. For that reason, we have made the YSI Model 63 flexible to accommodate these “advanced users.”

If, for example, you are using the YSI Model 63 for a process control application which requires that the conductivity readings be compensated to 20°C instead of 25°C -- this is the chapter to read. Or, if your application for the YSI Model 63 involves the measurement of a very specific saline solution, the default temperature coefficient may need to be changed to get the very best measurement of that specific salt.

**IMPORTANT:** There is never a need to enter Advanced Setup Mode unless your special measurement application calls for a change in reference temperature and or temperature coefficient. Therefore, unless you are certain that your application requires a change to one or both of these criteria, do not modify the default reference temperature (25°C) or the default temperature coefficient (1.91%).

**NOTE:** Changing the reference temperature or temperature coefficient does not affect salinity readings which are always referenced to seawater at 15°C. See 9.3 *Salinity* for details.

### 5.1 Changing The Temperature Coefficient

Follow these steps to modify the temperature coefficient of the Model 63.

1. Turn the instrument on and wait for it to complete its self test procedure.
2. Use the **MODE** key to advance the instrument to display conductivity.
3. Press and release both the **DOWN ARROW** and the **MODE** keys at the same time.

The **CAL** symbol will appear at the bottom left of the display. The large portion of the display will show **1.91 %** (or a value set previously using Advanced Setup).

4. Use the **UP ARROW** or **DOWN ARROW** key to change the value to the desired new temperature coefficient.
5. Press the **ENTER** key. The word “**SAVE**” will flash across the display for a second to indicate that your change has been accepted.
6. Press the **MODE** key to return to normal operation; the **CAL** symbol will disappear from the display.

## 5.2 Changing The Reference Temperature

Follow these steps to modify the reference temperature of the Model 63.

1. Turn the instrument on and wait for it to complete its self test procedure.
2. Use the **MODE** key to advance the instrument to display conductivity.
3. Press and release both the **DOWN ARROW** and the **MODE** keys at the same time.

The **CAL** symbol will appear at the bottom left of the display. The large portion of the display will show **1.91 %** (or a value set previously using Advanced Setup).

4. Press and release the **MODE** key; the large portion of the display will show **25.0C** (or a value set previously using Advanced Setup).
5. Use the **UP ARROW** or **DOWN ARROW** key to change the value to the desired new reference temperature (any value between 15°C and 25°C is acceptable).
6. Press the **ENTER** key. The word “**SAVE**” will flash across the display for a second to indicate that your change has been accepted.
7. The instrument will automatically return to normal operation mode.

## 5.3 Changing Conductivity From Autoranging To Manual Ranging

If your application is easier to perform using a manual range which you select, the YSI Model 63 allows you to turn off the default autoranging feature. While you are making conductivity or temperature compensated conductivity measurements, simply press and release the **UP ARROW** key. Each additional press of the **UP ARROW** key will cycle the Model 63 to a different manual range until you return again to autoranging. Five pushes of the **UP ARROW** key will cycle the Model 63 through the four manual ranges and return the instrument to autoranging.

**NOTE:** You may see an error message in some manual ranges if the manual range selected is not adequate for the sample you are measuring. If this happens, simply press and release the **UP ARROW** key again until a range is selected which is suitable for your sample. If you get lost and don't know if you're in a manual range or autoranging, simply turn the instrument off and back on. Also note that the conductivity units will flash while you are in manual range. The instrument will always default to autoranging when first turned on.

The four conductivity ranges of the YSI Model 63 are:

<b>Range 1</b>	<b>Range 2</b>	<b>Range 3</b>	<b>Range 4</b>
0 to 499.9 $\mu$ S/cm	0 to 4999 $\mu$ S/cm	0 to 49.99 mS/cm	0 to 200.0 mS/cm

## 6. Maintenance

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### 6.1 *pH Sensor Precautions*

1. When making measurements or performing the calibration procedure, make certain that the level of sample or pH buffer is high enough to cover both the pH and temperature sensors.
2. Rinse the probe with deionized water between changes of calibration buffer solutions.
3. During pH calibration, allow the sensors time to stabilize with regard to temperature (approximately 60 seconds) before proceeding with the calibration protocol. The pH readings after calibration are only as good as the calibration itself.
4. Clean and store the probe according to the instructions found below.

### 6.2 *pH Sensor Cleaning*

Cleaning is required whenever deposits or contaminants appear on the glass pH sensor. Unscrew and remove the small guard that protects the pH sensor. Use tap water and a clean cloth or lens cleaning tissue to remove all foreign material from the glass sensor.

If good pH response is not restored by the above procedure, perform the following additional procedure:

1. Soak the probe for 10 to 15 minutes in clean water containing a few drops of commercial dishwashing liquid.
2. GENTLY clean the glass bulb by rubbing with a cotton swab soaked in the cleaning solution.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, and then rerinse with clean water.

If good pH response is still not restored by the above procedure, perform the following additional procedure:

1. Soak the pH sensor for 5 minutes in one molar (1 M) hydrochloric acid (HCl).
2. GENTLY clean the glass bulb by rubbing with a cotton swab soaked in the acid.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, and then rerinse with clean water.
4. Reinstall the small guard that protects the pH sensor.

If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

1. Soak the probe for approximately 1 hour in a 1 to 1 dilution of commercially-available chlorine bleach.
2. Rinse the probe with clean water and then soak for 1 hour in clean water to remove residual bleach from the junction.

### **6.3 pH Sensor Storage**

For short term storage between measurements in the field (up to one week), place the probe in the transport chamber in the side of the instrument case. Make sure that the sponge inside the chamber is wet (tap water).

For long term storage (over one week), place the probe in the storage bottle (provided) containing a mixture of 50% pH 4 buffer and 50% 1.5M KCl. This will assure the fastest possible pH response. If this mixture is not available, storage in tap water is the next best choice. **Do NOT store the probe dry or in distilled or deionized water.**

**NOTE:** After storage in the pH 4/KCl solution described above, place the probe in the transport chamber in the side of the instrument case or soak the probe in pH 7 buffer for 5 to 10 minutes allowing it to acclimate before calibrating.

If the probe has been inadvertently left in air and the reference electrode junction has dried out, good function can usually be restored by soaking the probe in the pH 4/KCl solution described above.

### **6.4 Conductivity Sensor Cleaning**

The single most important requirement for accurate and reproducible results in conductivity measurement is a clean cell. A dirty cell will change the conductivity of a solution by contaminating it.

**NOTE:** Always rinse the conductivity cell with clean water after each use.

To clean the conductivity cell:

1. Dip the cell in cleaning solution and agitate for two to three minutes. Any one of the foaming acid tile cleaners, such as Dow Chemical Bathroom Cleaner, will clean the cell adequately. When a stronger cleaning preparation is required, use a solution of 1:1 isopropyl alcohol and one molar (1 M) hydrochloric acid (HCl). Remove the cell from the cleaning solution.
2. Use the nylon brush (supplied) to dislodge any contaminants from inside the electrode chamber.
3. Repeat steps one and two until the cell is completely clean. Rinse the cell thoroughly in deionized, or clean tap water.

## 7. Discussion of Measurement Errors

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### 7.1 pH Errors

There are two basic types of pH errors. The first type are errors related to limitations of instrument design and tolerances of components. The second type are errors due to basic sensor accuracy tolerances, mainly background signal, linearity, and variations in temperature coefficient. It is unlikely that the actual error in any measurement will be the maximum possible error.

#### Errors

- Component and circuitry error:  $\pm 0.03$  pH
- pH error caused by sensor accuracy and temperature compensation:
  - $\pm 0.1$  pH for measurements at  $10^{\circ}\text{C}$  from calibration temperature
  - $\pm 0.2$  pH for measurements at  $20^{\circ}\text{C}$  from calibration temperature

### 7.2 Conductivity Errors

System accuracy for conductivity measurements is equal to the sum of the errors contributed by the environment and the various components of the measurement setup. These include:

- Instrument accuracy
- Cell-constant error
- Solution temperature offset
- Cell contamination (including air bubbles)
- Electrical noise
- Galvanic effects

Only the first three are of major concern for typical measurements, although the user should also be careful to see that cells are clean and maintained in good condition at all times.

**Instrument Accuracy** =  $\pm .5\%$  maximum

The accuracy specified for the range being used is the worst case instrument error.

**Cell-Constant Error** =  $\pm .5\%$  maximum

Although YSI cells are warranted to be accurate to within one percent, you should still determine the exact cell constant of your particular cell. Contamination or physical damage to the cell can alter the cell constant. Performing a calibration will eliminate any error which might arise because of cell constant change.

YSI cells are calibrated to within one percent of the stated cell constant at a single point. We consider these products to be usefully linear over most instrument ranges. The cell constant can be calibrated to  $\pm 0.35\%$  accuracy with YSI conductivity calibrator solutions.

**Temperature Error =  $\pm 1\%$  maximum**

The solution temperature error is the product of the temperature coefficient and the temperature offset from 25°C, expressed as a percentage of the reading that would have been obtained at 25°C. The error is not necessarily a linear function of temperature. The statement of error is derived from a 25°C temperature offset and a 3%/°C temperature coefficient.

**Total Error**

Considering only the above three factors, system accuracy under worst case conditions will be  $\pm 2\%$ , although the actual error will be considerably less if recommended and properly calibrated cells and instrument ranges are used. Additional errors, which can essentially be eliminated with proper handling, are described below.

**Cell Contamination**

This error is usually due to contamination of the solution being measured, which occurs when solution is carried-over from the last solution measured. Thus, the instrument might be correctly reporting the conductivity seen, but the reading does not accurately represent the value of the bulk solution. Errors will be most serious when low conductivity solutions are contaminated by carry-over from high conductivity solutions, and can then be of an order of magnitude or more.

Follow the cleaning instructions carefully before attempting low conductivity measurements with a cell of unknown history or one that has been previously used in higher value solutions.

An entirely different form of contamination sometimes occurs due to a buildup of foreign material directly on cell electrodes. While rare, such deposits have, on occasion, markedly reduced the effectiveness of the electrodes. The result is an erroneously low conductance reading.

**Electrical-Noise Errors**

Electrical noise can be a problem in any measurement range, but will contribute the most error and be the most difficult to eliminate when operating in the lowest ranges. The noise may be either line-conducted or radiated or both, and may require, grounding, shielding, or both.

**Galvanic and Miscellaneous Effects**

In addition to the error sources described above, there is another class of contributors that can be ignored for all but the most meticulous of laboratory measurements. These errors are always small and are generally completely masked by the error budget for cell-constant calibration, instrument accuracy, etc. Examples range from parasitic reactances associated with the solution container and its proximity to external objects to the minor galvanic effects resulting from oxide formation or deposition on electrodes. Only trial and error in the actual measurement environment can be suggested as an approach to reduce such errors. If the reading does not change as the setup is adjusted, errors due to such factors can be considered too small to see.

## 8. Troubleshooting

### Error Messages

The instrument performs a Power On Self Test each time it is turned on. The following error messages are provided to facilitate troubleshooting. They appear on the LCD when an error is detected.

Symptom	Possible Cause	Action
1. Instrument will not turn on	<ul style="list-style-type: none"> <li>• Low battery voltage</li> <li>• Batteries installed wrong</li> <li>• Meter requires service</li> </ul>	<ul style="list-style-type: none"> <li>• Replace batteries (pg 2)</li> <li>• Check battery polarity (pg 2)</li> <li>• Return system for service (pg 29)</li> </ul>
2. Instrument "locks up"	<ul style="list-style-type: none"> <li>• Instrument has received a shock</li> <li>• Batteries are low or damaged</li> <li>• System requires service</li> </ul>	<ul style="list-style-type: none"> <li>• Remove battery lid, wait 15 seconds for reset, replace lid. (pg 2)</li> <li>• Replace batteries (pg 2)</li> <li>• Return system for service (pg 29)</li> </ul>
3. Conductivity will not calibrate	<ul style="list-style-type: none"> <li>• Conductivity standards out of spec.</li> <li>• Conductivity cell is contaminated</li> </ul>	<ul style="list-style-type: none"> <li>• Recalibrate with known good standards (pg 11)</li> <li>• Clean conductivity cell (pg 20)</li> </ul>
4. pH will not calibrate due to unstable readings (decimal point keeps flashing)	<ul style="list-style-type: none"> <li>• pH sensor is fouled</li> <li>• pH sensor is bad</li> <li>• System requires service</li> </ul>	<ul style="list-style-type: none"> <li>• Clean pH sensor (pg 20)</li> <li>• Replace pH sensor (pg 4, 33)</li> <li>• Return system for service (pg 29)</li> </ul>
5. pH readings are inaccurate	<ul style="list-style-type: none"> <li>• Calibration is required</li> <li>• pH calibration buffers out of spec</li> <li>• Calibration procedure not correct</li> <li>• Sample temperature is over 20°C from calibration temperature</li> <li>• pH sensor is fouled or damaged</li> <li>• pH Sensor is bad</li> <li>• System requires service</li> </ul>	<ul style="list-style-type: none"> <li>• Recalibrate with known good standards (pg 6)</li> <li>• Calibrate within <math>\pm 20^{\circ}\text{C}</math> of sample temp (<math>\pm 10^{\circ}\text{C}</math> for best results)</li> <li>• Clean pH sensor (pg 20)</li> <li>• Replace pH sensor (pg 4, 33)</li> <li>• Return system for service (pg 29)</li> </ul>
6. Conductivity readings are inaccurate	<ul style="list-style-type: none"> <li>• Cell is contaminated</li> <li>• Calibration is required</li> <li>• Temperature coefficient is set incorrectly</li> <li>• Reference temperature is set incorrectly</li> <li>• Readings are or are not temperature compensated.</li> </ul>	<ul style="list-style-type: none"> <li>• Clean conductivity cell (pg 21)</li> <li>• See <i>Conductivity Calibration</i> (pg 11)</li> <li>• See <i>Changing The Temperature Coefficient</i> (pg 18)</li> <li>• See <i>Changing The Reference Temperature</i> (pg 19)</li> <li>• See <i>Making Measurements</i> (pg 12)</li> </ul>
7. LCD displays "LO BAT"	<ul style="list-style-type: none"> <li>• Batteries are low or damaged</li> <li>• System requires service</li> </ul>	<ul style="list-style-type: none"> <li>• Replace batteries (pg 2)</li> <li>• Return system for service (pg 29)</li> </ul>
8. Main Display reads "nOnE"	<ul style="list-style-type: none"> <li>• During recall, no data is currently stored in memory.</li> </ul>	<ul style="list-style-type: none"> <li>• Store data before attempting to recall (pg 14)</li> </ul>
9. pH Display reads "OVER"	<ul style="list-style-type: none"> <li>• When calibrating: pH level is over range allowed for the buffer value selected.</li> <li>• When measuring, pH level is <math>&gt; 14</math></li> </ul>	<ul style="list-style-type: none"> <li>• Recalibrate with known good standards (pg 6)</li> <li>• Clean pH sensor (pg 20)</li> <li>• Replace pH sensor (pg 4, 33)</li> <li>• Return system for service (pg 29)</li> </ul>

Symptom	Possible Cause	Action
10. Conductivity/Salinity Display reads "OVER"	<ul style="list-style-type: none"> <li>When calibrating: User cell constant cal K is &gt;5.25</li> <li>When measuring: Conductivity reading is &gt;200 mS</li> <li>Salinity reading is &gt;80 ppt</li> </ul>	<ul style="list-style-type: none"> <li>Recalibrate with known good standards (pg 11)</li> <li>Clean conductivity cell (pg 21)</li> <li>Return system for service (pg 29)</li> </ul>
11. pH Display reads "undr"	<ul style="list-style-type: none"> <li>When calibrating, pH level is under range allowed for the buffer value selected.</li> <li>When measuring, pH level is &lt; 0</li> </ul>	<ul style="list-style-type: none"> <li>Recalibrate with known good standards (pg 6)</li> <li>Clean pH sensor (pg 20)</li> <li>Replace pH sensor (pg 4, 33)</li> <li>Return system for service (pg 29)</li> </ul>
12. Conductivity Display reads "undr"	<ul style="list-style-type: none"> <li>User cell constant cal K is &lt;4.9</li> </ul>	<ul style="list-style-type: none"> <li>Recalibrate with known good standards (pg 11)</li> <li>Clean conductivity cell (pg 21)</li> <li>Return system for service (pg 29)</li> </ul>
13. Main Display reads "OVER" (Secondary display reads "ovr")	<ul style="list-style-type: none"> <li>Temperature reading is &gt;75°C</li> </ul>	<ul style="list-style-type: none"> <li>Measure samples at a temperature within the range of the system.</li> </ul>
14. Main Display reads "undr" (Secondary display reads "udr")	<ul style="list-style-type: none"> <li>Temperature reading is &lt;-5°C</li> </ul>	<ul style="list-style-type: none"> <li>Measure samples at a temperature within the range of the system.</li> </ul>
15. Main display reads "PErr"	<ul style="list-style-type: none"> <li>User cell constant cal K is 0.0</li> <li>Incorrect sequence of keystrokes.</li> </ul>	<ul style="list-style-type: none"> <li>See "Advanced Setup" chapter (pg 18)</li> <li>Refer to manual section for step by step instruction for the function you are attempting.</li> </ul>
16. Main display reads "LErr"	<ul style="list-style-type: none"> <li>In temperature compensated conductivity mode, temperature exceeds the values computed using user defined temperature coefficient and/or reference temperature.</li> <li>In cell constant cal mode, temperature exceeds the values computed using user defined temperature coefficient and/or reference temperature.</li> </ul>	<ul style="list-style-type: none"> <li>Adjust user defined temperature coefficient or reference temperature. (pg 18)</li> </ul>
17. Main display reads "Err" (Secondary display reads "ra")	<ul style="list-style-type: none"> <li>System has failed its RAM test check procedure.</li> </ul>	<ul style="list-style-type: none"> <li>Turn instrument OFF and back ON again.</li> <li>Return the system for service (pg 29)</li> </ul>
18. Main display reads "Err" (Secondary display reads "ro")	<ul style="list-style-type: none"> <li>System has failed its ROM test check procedure.</li> </ul>	<ul style="list-style-type: none"> <li>Turn instrument OFF and back ON again.</li> <li>Return the system for service (pg 29)</li> </ul>
19. Main display reads "FAIL" (Secondary display reads "eep")	<ul style="list-style-type: none"> <li>EEPROM has failed to respond in time.</li> </ul>	<ul style="list-style-type: none"> <li>Return the system for service (pg 29)</li> </ul>
20. Readings on main display don't change	<ul style="list-style-type: none"> <li>Meter is in recall mode.</li> </ul>	<ul style="list-style-type: none"> <li>Press MODE key to return to Normal Operation (pg 5, 12)</li> </ul>

## 9. Principles of Operation

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### 9.1 pH

The YSI Model 63 employs a field replaceable pH sensor for the determination of hydrogen ion concentration. The sensor is a combination electrode consisting of a proton selective glass reservoir filled with buffer at approximately pH 7 and a Ag/AgCl reference electrode which utilizes gelled electrolyte. A silver wire coated with AgCl is immersed in the buffer reservoir. Protons (H<sup>+</sup> ions) on both sides of the glass (media and buffer reservoir) selectively interact with the glass, setting up a potential gradient across the glass membrane. Since the hydrogen ion concentration in the internal buffer solution is invariant, this potential difference, determined relative to the Ag/AgCl reference electrode, is proportional to the pH of the media.

Our testing of the Model 63 pH sensor indicates that it should provide long life, good response time and accurate readings in most environmental waters, including fresh water of low ionic strength. No special sensor is required (nor offered) for water of low conductivity.

#### pH Calibration And Effect Of Temperature

The software of the YSI Model 63 calculates pH from the established linear relationship between pH and the millivolt output as defined by a variation of the Nernst equation:

$$E = E_0 + \frac{2.3RT}{nF} * \text{pH}$$

where E = millivolts output  
E<sub>0</sub> = a constant associated with the reference electrode  
T = temperature of measurement in degrees Kelvin  
R, n, and F are invariant constants

Thus, in simplified  $y = mx + b$  form, it is (mv output) = (slope)x(pH) + (intercept). In order to quantify this simple relationship, the instrument must be calibrated properly using buffers of known pH values. In this procedure, the millivolt values for two standard buffer solutions are experimentally established and used by the YSI Model 63 software to calculate the slope and intercept of the plot of millivolts vs. pH. Once this calibration procedure has been carried out, the millivolt output of the probe in any media can readily be converted by the YSI Model 63 software into a pH value, *as long as the calibration and the reading are carried out at the same temperature.* This last qualifier is almost never met in actual environmental measurements, thus, a mechanism must be in place to compensate for temperature or, in other words, to accurately convert the slope and intercept of the plot of pH vs. millivolts established at T<sub>c</sub> (temperature of calibration) into a slope and intercept at T<sub>m</sub> (temperature of measurement). Fortunately, the Nernst equation provides a basis for this conversion.

According to the Nernst equation as shown above, the slope of the plot of pH vs. millivolts is *directly proportional* to the absolute temperature in degrees Kelvin. Thus, if the slope of the plot is experimentally determined to be 59 mv/pH unit at 298 K (25 C), then the slope of the plot at 313 K (40 C) must be (313/298) \* 59 = 62 mv/pH unit. At 283 K (10 C), the slope is calculated to be 56 mv/pH unit ((283/298) \* 59). Determination of the slope of pH vs. mv plots at temperatures different from T<sub>c</sub> is thus relatively simple. In order to establish the intercept of the new plot, the point where plots of pH vs. mv at different temperatures intersect (the isopotential point) must be known. Using standard pH determination protocol, the YSI Model 63 software assigns the isopotential point as the mv reading at pH 7 and then calculates the intercept using

this assumption. Once the slope and intercept to the plot of pH vs. mv are assigned at the new temperature, the calculation of pH under the new temperature conditions is straightforward, and is automatically carried out by the software.

### Number of pH Calibration Points

When calibrating the YSI Model 63, you have the choice of 1-point 2-point, or 3-point calibration. **Perform a 2 or 3 point calibration at least once per day for accurate results.**

Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously (at least once per day), you can adjust the calibration by carrying out a 1-point calibration at pH 7 (or pH 6.86). This calibration procedure adjusts only the pH offset and leaves the previously-determined slope unaltered.

Select the **2-point** option to calibrate the pH probe using only two calibration standards. In this procedure, the pH sensor is calibrated using a pH 7 (or pH 6.86) buffer and *one additional* buffer. A two point calibration procedure (as opposed to a 3-point procedure) can save time if the pH of the sample is known to be either basic or acidic. For example, if the pH of a sample is known to vary between 5.5 and 7, a two point calibration with pH 7 and pH 4 buffers is appropriate. Three point calibration with an additional pH 10 buffer will not increase the accuracy of this measurement since the pH is not within this higher range.

Select the **3-point** option to calibrate the pH probe using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 (or pH 6.86) buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated.

## 9.2 Conductivity

The conductivity cell utilizes four pure nickel electrodes for the measurement of solution conductance. Two of the electrodes are current driven, and two are used to measure the voltage drop. The measured voltage drop is then converted into a conductance value in milli-Siemens (millimhos). To convert this value to a conductivity (specific conductance) value in milli-Siemens per cm (mS/cm), the conductance is multiplied by the cell constant which has units of reciprocal cm ( $\text{cm}^{-1}$ ). The cell constant for the Model 63 conductivity cell is  $5.0/\text{cm} \pm 4\%$ . For most applications, the cell constant is automatically determined (or confirmed) with each deployment of the system when the calibration procedure is followed. Solutions with conductivity's of 1.00, 10.0, 50.0, and 100.0 mS/cm, which have been prepared in accordance with recommendation 56-1981 of the Organisation Internationale de Métrologie Légale (OIML) are available from YSI. The instrument output is in  $\mu\text{S}/\text{cm}$  or  $\text{mS}/\text{cm}$  for both conductivity and specific conductance. The multiplication of cell constant times conductance is carried out automatically by the software.

### Temperature Effect On Conductivity

The conductivity of solutions of ionic species is highly dependent on temperature, varying as much as 3% for each change of one degree Celsius (temperature coefficient = 3%/C). In addition, the temperature coefficient itself varies with the nature of the ionic species present.

Because the exact composition of a natural media is usually not known, it is best to report a conductivity at a particular temperature, e.g. 20.2 mS/cm at 14 C. However, in many cases, it is also useful to compensate for the temperature dependence in order to determine at a glance if gross changes are occurring in the ionic content of the medium over time. For this reason, the Model 63 software also allows the user to output conductivity data in either raw or temperature compensated form. If "Conductivity" is selected, values of conductivity which are **NOT** compensated for temperature are output to the display. If "Specific Conductance" is selected, the Model 63 uses the temperature and raw conductivity values associated with each determination to generate a specific conductance value compensated to a user selected reference temperature (see *Advanced Setup*) between 15 C and 25 C. Additionally the user can select any temperature coefficient from 0% to 4% (see *Advanced Setup*). Using the Model 63 default reference temperature and temperature coefficient (25 C and 1.91%), the calculation is carried out as in the equation below:

$$\text{Specific Conductance (25°C)} = \frac{\text{Conductivity}}{1 + \text{TC} * (\text{T} - 25)}$$

As noted above, unless the solution being measured consists of pure KCl in water, this temperature compensated value will be somewhat inaccurate, but the equation with a value of TC = 0.0191 will provide a close approximation for solutions of many common salts such as NaCl and NH<sub>4</sub>Cl and for seawater.

### **9.3 Salinity**

Salinity is determined automatically from the Model 63 conductivity and temperature readings according to algorithms found in *Standard Methods for the Examination of Water and Wastewater* (ed. 1995). The use of the Practical Salinity Scale 1978 results in values which are unitless, since the measurements are carried out in reference to the conductivity of standard seawater at 15°C. However, the unitless salinity values are very close to those determined by the previously-used method where the mass of dissolved salts in a given mass of water (parts per thousand) was reported. Hence, the designation "ppt" is reported by the instrument to provide a more conventional output.

For further information on conductivity and the above standard information, refer to the ASTM document, *Standard Methods of Test for Electrical Conductivity of Water and Industrial Wastewater*, ASTM Designation D1125-82, and OIML *Recommendation Number 56*. ASTM symbols for conductivity, cell constant, and path length differ from those preferred in the general literature and also from those used in this manual.

### **9.4 Temperature**

The YSI Model 63 system utilizes a thermistor which changes predictably in resistance with temperature variation. The algorithm for conversion of resistance to temperature is built-in to the Model 63 software, and accurate temperature readings, in degrees Celsius, are provided automatically. No calibration or maintenance of the temperature sensor is required.

## 10. Warranty and Repair

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YSI Model 63 Meters are warranted for two years from date of purchase by the end user against defects in materials and workmanship. YSI Model 63 probes, cables and sensors are warranted for one year from date of purchase by the end user against defects in material and workmanship. Breakage of pH sensors is NOT covered under warranty. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

### **Limitation of Warranty**

This Warranty does not apply to any YSI product damage or failure caused by (i) failure to install, operate or use the product in accordance with YSI's written instructions, (ii) abuse or misuse of the product, (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure, (iv) any improper repairs to the product, (v) use by you of defective or improper components or parts in servicing or repairing the product, or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI's LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

### **YSI Authorized Service Centers**

**Please visit [www.ysi.com](http://www.ysi.com) or contact YSI Technical Support for the nearest authorized service center.**

YSI Incorporated • Technical Support • Phone: +1 937 767-7241 • 800 897-4151 • Fax: 937 767-1058 • Email: [environmental@ysi.com](mailto:environmental@ysi.com)

## **10.1 Cleaning Instructions**

**NOTE: Before they can be serviced, equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected.** Biological contamination is presumed for any instrument, probe, or other device that has been used with body fluids or tissues, or with waste water. Radioactive contamination is presumed for any instrument, probe or other device that has been used near any radioactive source.

If an instrument, probe, or other part is returned or presented for service without a Cleaning Certificate, and if in our opinion it represents a potential biological or radioactive hazard, our service personnel reserve the right to withhold service until appropriate cleaning, decontamination, and certification has been completed. We will contact the sender for instructions as to the disposition of the equipment. Disposition costs will be the responsibility of the sender.

When service is required, either at the user's facility or at YSI, the following steps must be taken to insure the safety of our service personnel.

- 1.** In a manner appropriate to each device, decontaminate all exposed surfaces, including any containers. 70% isopropyl alcohol or a solution of 1/4 cup bleach to 1 gallon tap water are suitable for most disinfecting. Instruments used with waste water may be disinfected with .5% Lysol if this is more convenient to the user.
- 2.** The user shall take normal precautions to prevent radioactive contamination and must use appropriate decontamination procedures should exposure occur.
- 3.** If exposure has occurred, the customer must certify that decontamination has been accomplished and that no radioactivity is detectable by survey equipment.
- 4.** Any product being returned to the YSI Repair Center, should be packed securely to prevent damage.
- 5.** Cleaning must be completed and certified on any product before returning it to YSI.

## 10.2 Packing Instructions

1. Clean and decontaminate items to insure the safety of the handler.
2. Complete and include the Cleaning Certificate.
3. Place the product in a plastic bag to keep out dirt and packing material.
4. Use a large carton, preferably the original, and surround the product completely with packing material.
5. Insure for the replacement value of the product.

<b>Cleaning Certificate</b>	
Organization _____	
Department _____	
Address _____	
City _____	State _____ Zip _____
Country _____	Phone _____
Model No. of Device _____ Lot Number _____	
Contaminant (if known) _____	
_____	
Cleaning Agent(s) used _____	
Radioactive Decontamination Certified?	
(Answer only if there has been radioactive exposure)	
___ Yes ___ No	
Cleaning Certified By _____	
	Name                  Date

## 11. Required Notice

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The Federal Communications Commission defines this product as a computing device and requires the following notice:

This equipment generates and uses radio frequency energy and if not installed and used properly, may cause interference to radio and television reception. There is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- re-orient the receiving antenna
- relocate the computer with respect to the receiver
- move the computer away from the receiver
- plug the computer into a different outlet so that the computer and receiver are on different branch circuits.

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet, prepared by the Federal Communications Commission, helpful: "How to Identify and Resolve Radio-TV Interference Problems." This booklet is available from the U.S. Government Printing Office, Washington, DC 20402, Stock No. 0004-000-00345-4.

## 12. Accessories and Replacement Parts

The following parts and accessories are available from YSI or any Franchise Dealer authorized by YSI.

YSI Order Number	Description
3161	Conductivity Calibration Solution 1,000 $\mu$ /cm (1 Quart)
3163	Conductivity Calibration Solution 10,000 $\mu$ /cm (1 Quart)
3165	Conductivity Calibration Solution 100,000 $\mu$ /cm (1 Quart)
3167	Conductivity Calibration Solution 1,000 $\mu$ /cm (8 pints)
3168	Conductivity Calibration Solution 10,000 $\mu$ /cm (8 pints)
3169	Conductivity Calibration Solution 50,000 $\mu$ /cm (8 pints)
3821	pH Buffer Solution, 4
3822	pH Buffer Solution, 7
3823	pH Buffer Solution, 10
5050	Carrying Case
031133	pH sensor
113165	Conductivity Probe/Cable Assembly (10 feet)
113166	Conductivity Probe/Cable Assembly (25 feet)
113157	Conductivity Probe/Cable Assembly (50 feet)
113158	Conductivity Probe/Cable Assembly (100 feet)
031163	Front Case Cover
055242	Rear Case Cover
055210	Battery Cover Kit
055204	Case Gasket and Screw
031129	Main Board Assembly
038213	Electrode Cleaning Brush, Conductivity
031189	Graduated Cylinder, 100 mL
060992	Container, Plastic (uses 060991 cap)
060991	Cap, Plastic Container (for 060992 container)

## 13. Appendix A - Specifications

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**Materials:** ABS, Stainless Steel, and other materials

**Dimensions:**

Height:	9.5 inches	(24.13 cm)
Thickness:	2.2 inches	(5.6 cm)
Width:	3.5 inches max.	(8.89 cm)
Weight:	1.7 pounds (w/ 10' cable)	(.77 kg)
Display:	2.3"W x 1.5"L	(5.8 cm W x 3.8 cm L)

**Power:** 6 AA-size Alkaline Batteries (included)

Approximately 100 hours operation from each new set of batteries

Automatic shutoff after 10 hours without a key press

**Water Tightness:** Meets or exceeds IP65 standards

**Probe Operating Environment**

Medium: fresh, sea, or polluted water and most other liquid solutions.

Temperature: -5 to +75 °C

Depth: 0 to 10, 0 to 25, 0 to 50, or 0 to 100 feet (depending on cable length)

**Meter Ambient Operating/Storage Temperature:** -5 to +45 °C

**System Performance Specifications**

Measurement	Range	Resolution	Accuracy
pH	0 to 14	0.01 unit	± 0.1 pH unit within ±10°C of calibration temperature <b>or</b> ± 0.2 pH unit within ±20°C of calibration temperature
Conductivity	0 to 499.9 µS/cm 0 to 4999 µS/cm 0 to 49.99 mS/cm 0 to 200.0 mS/cm	0.1 µS/cm 1.0 µS/cm 0.01 mS/cm 0.1 mS/cm	± 0.5% FS
Salinity	0 to 80 ppt	0.1 ppt	± 2%, or ± 0.1 ppt
Temperature	-5 to +75 °C	0.1 °C	± 0.1°C ±1 LSD

**Adjustable Conductivity Reference Temperature:** 15°C to 25°C

**Adjustable Temperature Compensation Factor for Conductivity:** 0% to 4%

**pH Response Time:** 3 sec for 95% of the change at 25°C

**Temperature Response Time:** 20 sec for 95% of the change

**Temperature Compensation:** Automatic

**Range:** User selected or Autoranging for Conductivity

**Data Storage:** 50 points with ID number.

## 14. Appendix B - Health and Safety

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### YSI pH 4, 7 & 10 Buffer Solutions: 3821, 3822, 3823

#### pH 4 Ingredients:

- ☞ Potassium Hydrogen Phthalate
- ☞ Formaldehyde
- ☞ Water

#### pH 7 Ingredients:

- ☞ Sodium Phosphate, Dibasic
- ☞ Potassium Phosphate, Monobasic
- ☞ Water

#### pH 10 Ingredients:

- ☞ Potassium Borate, Tetra
- ☞ Potassium Carbonate
- ☞ Potassium Hydroxide
- ☞ Sodium (di) Ethylenediamine Tetraacetate
- ☞ Water

**CAUTION - Avoid inhalation, skin contact, eye contact or ingestion. May affect mucous membranes.**

Inhalation may cause severe irritation and be harmful. Skin contact may cause irritation; prolonged or repeated exposure may cause Dermatitis. Eye contact may cause irritation or conjunctivitis. Ingestion may cause nausea, vomiting and diarrhea.

#### **FIRST AID:**

**INHALATION - Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.**

**SKIN CONTACT - Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.**

**EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.**

**INGESTION - If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.**

## 15. Appendix C - pH Buffer Values

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### YSI pH 4, 7 and 10 Buffer Solutions: 3821, 3822, 3823

The following table lists the values of YSI pH buffer solutions at various temperatures.

Temperature	pH 4	pH 7	pH 10
0°C	4.01	7.13	10.34
5°C	4.00	7.10	10.26
10°C	4.00	7.07	10.19
15°C	4.00	7.05	10.12
20°C	4.00	7.02	10.06
25°C	4.01	7.00	10.00
30°C	4.01	6.99	9.94
35°C	4.02	6.98	9.90
40°C	4.03	6.97	9.85
50°C	4.06	6.97	9.78
60°C	4.09	6.98	9.70

### NIST pH 4.01, 6.86 and 9.18 Buffers: SRM 185g, SRM 186-Ie/IIe, SRM 187c

The following table lists the values of NIST pH buffer solutions at various temperatures.

Temperature	pH 4.01	pH 6.86	pH 9.18
0°C	4.005	6.984	9.463
5°C	4.003	6.950	9.395
10°C	4.001	6.924	9.333
15°C	4.002	6.899	9.277
20°C	4.003	6.879	9.226
25°C	4.005	6.863	9.180
30°C	4.010	6.852	9.139
35°C	4.020	6.844	9.102
37°C	4.025	6.842	N/A
40°C	4.033	6.840	9.070
45°C	4.047	6.837	9.042
50°C	4.061	6.836	9.018

## 16. Appendix D - Temperature Correction Data

### Conductivity Temperature Correction Data for Typical Solutions

#### A. Potassium Chloride \*\* (KCl)

Concentration: 1 mole/liter			Concentration: $1 \times 10^{-1}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	65.10	1.67	0	7.13	1.78
5	73.89	1.70	5	8.22	1.80
10	82.97	1.72	10	9.34	1.83
15	92.33	1.75	15	10.48	1.85
20	101.97	1.77	20	11.65	1.88
25	111.90	1.80	25	12.86	1.90
			30	14.10	1.93
			35	15.38	1.96
			37.5	16.04	1.98
			40	16.70	1.99
			45	18.05	2.02
			50	19.43	2.04

Concentration: $1 \times 10^{-2}$ mole/liter			Concentration: $1 \times 10^{-3}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	0.773	1.81	0	0.080	1.84
5	0.892	1.84	5	0.092	1.88
10	1.015	1.87	10	0.105	1.92
15	1.143	1.90	15	0.119	1.96
20	1.275	1.93	20	0.133	1.99
25	1.412	1.96	25	0.147	2.02
30	1.553	1.99	30	0.162	2.05
35	1.697	2.02	35	0.178	2.07
37.5	1.771	2.03	37.5	0.186	2.08
40	1.845	2.05	40	0.194	2.09
45	1.997	2.07	45	0.210	2.11
50	2.151	2.09	50	0.226	2.13

\*\* Charts developed by interpolating data from *International Critical Tables*, Vol. 6, pp. 229-253, McGraw-Hill Book Co., NY.

## B. Sodium Chloride\* (NaCl)

Saturated solutions at all temperatures			Concentration: 0.5 mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	134.50	1.86	0	25.90	1.78
5	155.55	1.91	5	29.64	1.82
10	177.90	1.95	10	33.61	1.86
15	201.40	1.99	15	37.79	1.90
20	225.92	2.02	20	42.14	1.93
25	251.30	2.05	25	46.65	1.96
30	277.40	2.08	30	51.28	1.99
			35	56.01	2.01
			37.5	58.40	2.02
			40	60.81	2.02
			45	65.65	2.04
			50	70.50	2.05

Concentration: $1 \times 10^{-1}$ mole/liter			Concentration: $1 \times 10^{-2}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	5.77	1.83	0	0.632	1.87
5	6.65	1.88	5	0.731	1.92
10	7.58	1.92	10	0.836	1.97
15	8.57	1.96	15	0.948	2.01
20	9.60	1.99	20	1.064	2.05
25	10.66	2.02	25	1.186	2.09
30	11.75	2.04	30	1.312	2.12
35	12.86	2.06	35	1.442	2.16
37.5	13.42	2.07	37.5	1.508	2.17
40	13.99	2.08	40	1.575	2.19
45	15.14	2.10	45	1.711	2.21
50	16.30	2.12	50	1.850	2.24

Concentration: $1 \times 10^{-3}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)
0	0.066	1.88
5	0.076	1.93
10	0.087	1.98
15	0.099	2.02
20	0.111	2.07
25	0.124	2.11
30	0.137	2.15
35	0.151	2.19
37.5	0.158	2.20
40	0.165	2.22
45	0.180	2.25
50	0.195	2.29

\* Charts developed by interpolating data from the *CRC Handbook of Chemistry and Physics*, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.

### C. Lithium Chloride\* (LiCl)

Concentration: 1 mole/liter			Concentration: $1 \times 10^{-1}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	39.85	1.82	0	5.07	1.87
5	46.01	1.85	5	5.98	1.85
10	52.42	1.89	10	6.87	1.85
15	59.07	1.92	15	7.75	1.85
20	65.97	1.95	20	8.62	1.85
25	73.10	1.98	25	9.50	1.86
30	80.47	2.02	30	10.40	1.88
35	88.08	2.05	35	11.31	1.91
37.5	91.97	2.07	37.5	11.78	1.92
40	95.92	2.08	40	12.26	1.94
45	103.99	2.11	45	13.26	1.98
50	112.30	2.15	50	14.30	2.02

Concentration: $1 \times 10^{-2}$ mole/liter			Concentration: $1 \times 10^{-3}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	0.567	1.88	0	0.059	1.93
5	0.659	1.92	5	0.068	2.03
10	0.755	1.96	10	0.078	2.12
15	0.856	2.00	15	0.089	2.19
20	0.961	2.04	20	0.101	2.25
25	1.070	2.08	25	0.114	2.28
30	1.183	2.12	30	0.127	2.31
35	1.301	2.16	35	0.140	2.32
37.5	1.362	2.18	37.5	0.147	2.32
40	1.423	2.20	40	0.154	2.31
45	1.549	2.24	45	0.166	2.29
50	1.680	2.28	50	0.178	2.25

### D. Potassium Nitrate\*\* (KNO<sub>3</sub>)

Concentration: $1 \times 10^{-1}$ mole/liter			Concentration: $1 \times 10^{-2}$ mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	6.68	1.78	0	0.756	1.77
5	7.71	1.79	5	0.868	1.80
10	8.75	1.81	10	0.984	1.83
15	9.81	1.83	15	1.105	1.86
20	10.90	1.85	20	1.229	1.88
25	12.01	1.87	25	1.357	1.90
30	13.15	1.90	30	1.488	1.93
35	14.32	1.92	35	1.622	1.95
37.5	14.92	1.94	37.5	1.690	1.96
40	15.52	1.95	40	1.759	1.97
45	16.75	1.97	45	1.898	1.99
50	18.00	2.00	50	2.040	2.01

\* Charts developed by interpolating data from the *CRC Handbook of Chemistry and Physics*, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.

\*\* Charts developed by interpolating data from *International Critical Tables*, Vol. 6, pp. 229-253, McGraw-Hill Book Co., NY.

## E. Ammonium Chloride\* (NH<sub>4</sub>Cl)

Concentration: 1 mole/liter			Concentration: 1 x 10 <sup>-1</sup> mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	64.10	1.60	0	6.96	1.82
5	74.36	1.53	5	7.98	1.88
10	83.77	1.45	10	9.09	1.93
15	92.35	1.37	15	10.27	1.97
20	100.10	1.29	20	11.50	2.00
25	107.00	1.21	25	12.78	2.03
			30	14.09	2.06
			35	15.43	2.07
			37.5	16.10	2.08
			40	16.78	2.08
			45	18.12	2.09
			50	19.45	2.09

Concentration: 1 x 10 <sup>-2</sup> mole/liter			Concentration: 1 x 10 <sup>-3</sup> mole/liter		
°C	mS/cm	%/°C (to 25°C)	°C	mS/cm	%/°C (to 25°C)
0	0.764	1.84	0	0.078	1.88
5	0.889	1.86	5	0.092	1.90
10	1.015	1.88	10	0.105	1.91
15	1.144	1.91	15	0.119	1.93
20	1.277	1.94	20	0.133	1.95
25	1.414	1.97	25	0.148	1.98
30	1.557	2.02	30	0.162	2.01
35	1.706	2.06	35	0.178	2.04
37.5	1.782	2.08	37.5	0.186	2.06
40	1.860	2.10	40	0.194	2.07
45	2.020	2.14	45	0.210	2.11
50	2.186	2.18	50	0.227	2.15

\* Charts developed by interpolating data from the *CRC Handbook of Chemistry and Physics*, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.

*YSI incorporated*



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031178  
A31178C - Web  
January 07

# Ammonia CHEMets® Kit

K-1510/R-1501: 0 - 1 & 1 - 10 ppm N

## Safety Information

Read SDS (available at [www.chemetrics.com](http://www.chemetrics.com)) before performing this test procedure. Wear safety glasses and protective gloves.

## Non-Seawater Test Procedure

1. Fill the sample cup to the 25 mL mark with the sample to be tested (fig. 1).
2. Add 2 drops of A-1500 Stabilizer Solution (fig. 2). Stir to mix the contents of the cup.
3. Place the CHEMet ampoule, tip first, into the sample cup. Snap the tip. The ampoule will fill leaving a bubble for mixing (fig. 3).
4. To mix the ampoule, invert it several times, allowing the bubble to travel from end to end.
5. Dry the ampoule and wait **1 minute** for color development.
6. Obtain a test result using the appropriate comparator.

### a. Low Range Comparator (fig. 4):

Place the ampoule, flat end first, into the comparator. Hold the comparator up toward a source of light and view from the bottom. Rotate the comparator until the best color match is found.

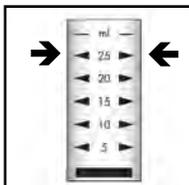


Figure 1



Figure 2

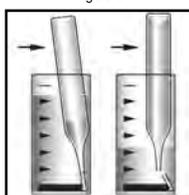


Figure 3



Figure 4

- b. High Range Comparator (fig. 5): Place the ampoule between the color standards until the best color match is found.

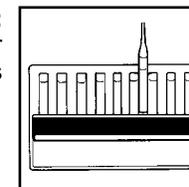


Figure 5

## Seawater Test Procedure

1. Using the syringe, add 1.0 mL of A-1501 Stabilizer Solution to the sample cup.
2. Fill the sample cup to the 25 mL mark with the seawater sample to be tested (fig 1).
3. Perform the Test Procedure above, beginning with Step 3.

## Test Method

The Ammonia CHEMets®<sup>1</sup> test kit employs direct nesslerization.<sup>2,3</sup> In a strongly alkaline solution, ammonia reacts with Nessler Reagent ( $K_2HgI_4$ ) to produce a yellow-colored complex in direct proportion to the ammonia concentration.

This method is applicable to drinking water, clean surface water, good quality nitrified wastewater effluent and seawater. Other types of samples may require a preliminary distillation step. Ketones, alcohols, and aldehydes may cause off-color test results. Glycine and hydrazine will cause high test results. Aromatic and aliphatic amines, iron, sulfide, calcium and magnesium may cause turbidity.

1. CHEMets is a registered trademark of CHEMetrics, Inc. U.S. Patent No. 3,634,038
2. APHA Standard Methods, 18th ed., Method 4500-NH<sub>3</sub> C - 1988
3. ASTM D 1426 - 08, Ammonia Nitrogen in Water, Test Method A

Visit [www.chemetrics.com](http://www.chemetrics.com) to view product demonstration videos.  
Always follow the test procedure above to perform a test.



4295 Catlett Road, Midland, VA 22728 U.S.A.  
Phone: (800) 356-3072; Fax: (540) 788-4856  
E-Mail: [orders@chemetrics.com](mailto:orders@chemetrics.com)  
Feb. 18, Rev. 13



# Ammonia Nitrogen Test Kit

NI-SA (2428700)

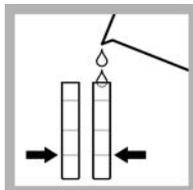
DOC326.98.00007

## Test preparation

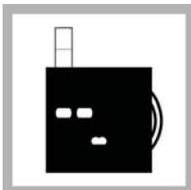
**CAUTION:** ⚠ *Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.*

- Put the color disc on the center pin in the color comparator box (numbers to the front).
- Use sunlight or a lamp as a light source to find the color match with the color comparator box.
- Rinse the tubes with sample before the test. Rinse the tubes with deionized water after the test.
- If the color match is between two segments, use the value that is in the middle of the two segments.
- If the color disc becomes wet internally, pull apart the flat plastic sides to open the color disc. Remove the thin inner disc. Dry all parts with a soft cloth. Assemble when fully dry.
- To verify the test accuracy, use a standard solution as the sample.
- This test kit is for seawater. If used for brackish or fresh water, the test kit gives a higher than actual value. The error in brackish water is usually less than 10%. The error in low salinity or fresh water is a maximum 16%.
- This test is very sensitive to contamination. Try to get the same result on a second test. Fully rinse the tubes with fresh sample before the second test. The reagents clean the tubes during the first test.
- To increase the range of this test to 4 mg/L NH<sub>3</sub>-N, dilute the sample as follows. Use a 3-mL syringe to add 2.5 mL of sample to each tube. Dilute the sample to the 5-mL mark with deionized water. Use the diluted sample in the test procedure and multiply the result by 2.

## Test procedure—Ammonia-nitrogen (0–2.0 mg/L NH<sub>3</sub>-N)



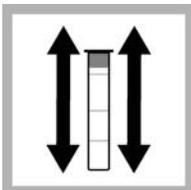
1. Fill two tubes to the first line (5 mL) with sample.



2. Put one tube into the left opening of the color comparator box.



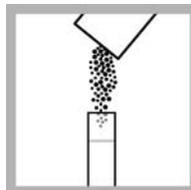
3. Add one Ammonia Salicylate Reagent Powder Pillow to the second tube.



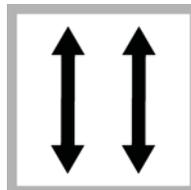
4. Put a stopper on the tube. Shake until the powder fully dissolves.



5. Wait 3 minutes.



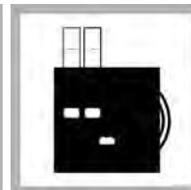
6. Add one Ammonia Cyanurate Reagent Powder Pillow to the same tube. Put a stopper on the tube.



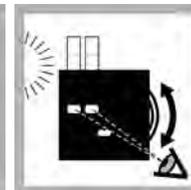
7. Shake until the powder fully dissolves.



8. Wait 15 minutes. A green color develops.



9. Put the second tube into the color comparator box.



10. Hold the color comparator box in front of a light source. Turn the color disc to find the color match.



11. Read the result in mg/L in the scale window.

## Replacement items

Description	Unit	Item no.
Ammonia Salicylate Reagent Powder Pillows, 5 mL	50/pkg	2395266
Ammonia Cyanurate Reagent Powder Pillows, 5 mL	50/pkg	2395466
Color disc, ammonia nitrogen, salicylate, 0–2.0 mg/L	each	9261300
Color comparator box	each	173200
Glass viewing tubes, glass, 18 mm	6/pkg	173006
Stoppers for 18-mm glass tubes and AccuVac Ampuls	6/pkg	173106

## Optional items

Description	Unit	Item no.
Nitrogen ammonia standard solution, 1.0 mg/L NH <sub>3</sub> -N	500 mL	189149
Water, deionized	500 mL	27249
Syringe, Luer-Lok® Tip, 3 mL	each	4321300

### Calculate the mg/L NH<sub>3</sub> and mg/L NH<sub>4</sub><sup>+</sup>

Ammonia in water is in the form of the ammonium ion (NH<sub>4</sub><sup>+</sup>) and un-ionized ammonia (NH<sub>3</sub>). NH<sub>3</sub> is toxic to fish. Table 1 shows that the percent of NH<sub>3</sub> increases as the pH and temperature increase. This test kit measures both NH<sub>4</sub><sup>+</sup> and NH<sub>3</sub> as ammonia nitrogen (NH<sub>3</sub>-N).

To calculate the mg/L NH<sub>3</sub> in the sample, refer to Table 1 and the equation that follows.

$$\text{mg/L NH}_3 = ((\text{mg/L NH}_3\text{-N} \times \text{percent NH}_3 \text{ from Table 1}) \div 100) \times 1.2$$

**Example:** The test result was 1.6 mg/L NH<sub>3</sub>-N. The sample pH was 7.6 and the sample temperature was 16 °C. The mg/L NH<sub>3</sub> is  $((1.6 \times 1.16) \div 100) \times 1.2 = 0.02 \text{ mg/L NH}_3$ .

To calculate the mg/L NH<sub>4</sub><sup>+</sup> in the sample, refer to Table 1 and the equation that follows.

$$\text{mg/L NH}_4^+ = ((\text{mg/L NH}_3\text{-N} \times (100 - \text{percent NH}_3 \text{ from Table 1})) \div 100) \times 1.3$$

**Example:** The test result was 1.6 mg/L NH<sub>3</sub>-N. The sample pH was 7.6 and the sample temperature was 16 °C. The mg/L NH<sub>4</sub><sup>+</sup> is  $((1.6 \times (100 - 1.16)) \div 100) \times 1.3 = 2.056 \text{ mg/L NH}_4^+$ .

**Table 1 Percent of NH<sub>3</sub> in water**

pH	16 °C	18 °C	20 °C	22 °C	24 °C	26 °C	28 °C	30 °C	32 °C
7.0	0.29	0.34	0.39	0.46	0.52	0.60	0.69	0.80	0.91
7.2	0.46	0.54	0.62	0.82	0.83	0.96	1.10	1.26	1.44
7.4	0.73	0.85	0.98	1.14	1.31	1.50	1.73	1.98	2.26
7.6	1.16	1.34	1.55	1.79	2.06	2.36	2.71	3.10	3.53
7.8	1.82	2.11	2.44	2.81	3.22	3.70	4.23	4.82	5.48
8.0	2.86	3.30	3.81	4.38	5.02	5.74	6.54	7.43	8.42
8.2	4.45	5.14	5.90	6.76	7.72	8.80	9.98	11.29	12.72
8.4	6.88	7.90	9.04	10.31	11.71	13.26	14.95	16.78	18.77
8.6	10.48	11.97	13.61	15.41	17.37	19.50	21.78	24.22	26.80
8.8	15.66	17.73	19.98	22.41	25.00	27.74	30.62	33.62	36.72
9.0	22.73	25.46	28.36	31.40	34.56	37.83	41.16	44.53	47.91
9.2	31.80	35.12	38.55	42.04	45.57	49.09	52.58	55.99	59.31
9.4	42.49	46.18	49.85	53.48	57.02	60.45	63.73	66.85	69.79
9.6	53.94	57.62	61.17	64.56	67.77	70.78	73.58	76.17	78.55
9.8	64.99	68.31	71.40	74.28	76.92	79.33	81.53	83.51	85.30
10.0	74.63	77.35	79.83	82.07	84.08	85.88	87.49	88.92	90.19
10.2	82.34	84.41	86.25	87.88	89.33	90.60	91.73	92.71	93.58





# Ammonia Nitrogen Test Kit

NI-8 (224100)

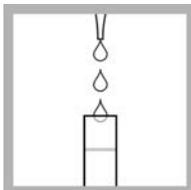
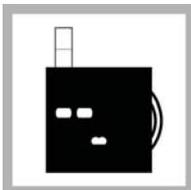
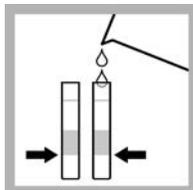
DOC326.97.00070

## Test preparation

**CAUTION:** ⚠ *Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.*

- Put the color disc on the center pin in the color comparator box (numbers to the front).
- Use sunlight or a lamp as a light source to find the color match with the color comparator box.
- Rinse the tubes with sample before the test. Rinse the tubes with deionized water after the test.
- If the color match is between two segments, use the value that is in the middle of the two segments.
- If the color disc becomes wet internally, pull apart the flat plastic sides to open the color disc. Remove the thin inner disc. Dry all parts with a soft cloth. Assemble when fully dry.
- To verify the test accuracy, use a standard solution as the sample.
- The recommended sample temperature is 20 °C. Warmer temperatures cause high results. Colder temperatures cause low results.
- More than 6 gpg hardness causes a white precipitate to develop. To remove the interference, add 1 drop of Rochelle salt reagent to the sample tube before the reagent is added.
- To measure very low quantities of ammonia, add ammonia-free water to one tube. Add 3 drops of the Nessler reagent. Put this tube in the left opening of the color comparator box as the reagent blank.
- To record the test result as mg/L NH<sub>3</sub>, multiply the test result by 1.2. To record the test result as mg/L NH<sub>4</sub><sup>+</sup>, multiply the test result by 1.3.

## Test procedure—Ammonia-nitrogen (0–3.0 mg/L NH<sub>3</sub>-N)



1. Fill two tubes to the first line (5 mL) with sample.

2. Put one tube into the left opening of the color comparator box.

3. Add 3 drops of Nessler reagent to the second tube.

4. Swirl to mix. A yellow color develops.

5. Wait 1 minute. Read the result within 5 minutes.

6. Put the second tube into the color comparator box.

7. Hold the color comparator box in front of a light source. Turn the color disc to find the color match.

8. Read the result in mg/L in the scale window.

## Replacement items

Description	Unit	Item no.
Nessler reagent	100 mL MDB	2119432
Color disc, ammonia nitrogen, Nessler, 0–3.0 mg/L	each	9262600
Color comparator box	each	173200
Plastic viewing tubes, 18 mm, with caps	4/pkg	4660004

## Optional items

Description	Unit	Item no.
Caps for plastic viewing tubes (4660004)	4/pkg	4660014
Water, deionized	500 mL	27249
Glass viewing tubes, glass, 18 mm	6/pkg	173006
Rochelle salt solution	29 mL DB	172533
Stoppers for 18-mm glass tubes and AccuVac Ampuls	6/pkg	173106



## Hach Test Strips



Obtain quick, quantitative answers in the field or lab

Test strips are one of the easiest methods of testing water. Simply dip the strip in water, following the instructions and compare the color of the strip to determine the result. Use test strips when a general range is sufficient. Test strips should not be used when an exact measurement is required.

With Hach Water Quality Test Strips, technicians in the field can test many samples in only a few minutes, and make immediate evaluations on-site. No measuring, set-up, clean-up, or chemical handling are necessary. Hach test strips are also used in laboratories all over the world for pre-test screening-to detect the presence of materials that might interfere with lab testing.

- Easy to use
- Disposable
- Inexpensive



### How to Use Test Strips

Test strips are one of the simplest types of tests to use. Simply dip the strip into the water according to directions on the bottle or package. The test strip will change color. Then compare the color of the test strip to the chart provided in the package to determine the test result. For common questions about test strips, see our Hach FAQ page at <https://www.hach.com/kb-productfaq#28>.



# Detergents CHEMets Kit

K-9400/R-9400: 0 - 3 ppm

## Test Procedure

1. Rinse the reaction tube with the sample to be tested, and then fill it to the 5 mL mark with the sample.
2. While holding the double-tipped ampoule in a vertical position, snap the upper tip using the tip breaking tool (fig. 1).
3. Invert the ampoule and position the open end over the reaction tube. Snap the upper tip and allow the contents to drain into the reaction tube (fig. 1).
4. Cap the reaction tube and shake it vigorously for **30 seconds**. Allow the tube to stand undisturbed for **1 minute**.
5. Make sure that the flexible tubing is firmly attached to the CHEMet ampoule tip.
6. Insert the CHEMet assembly (tubing first) into the reaction tube making sure that the end of the flexible tubing is at the bottom of the tube. Break the tip of the CHEMet ampoule by gently pressing it against the side of the reaction tube (fig. 2). The ampoule should draw in fluid only from the organic phase (bottom layer).
7. When filling is complete, remove the CHEMet assembly from the reaction tube.
8. Remove the flexible tubing from the CHEMet ampoule and wipe all liquid from the exterior of the ampoule. Place an ampoule cap firmly onto the tip of the CHEMet ampoule. Invert the ampoule several times, allowing the bubble to travel from end to end.

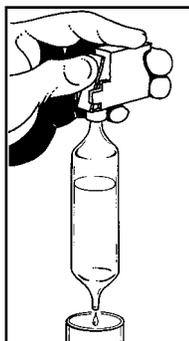


Figure 1

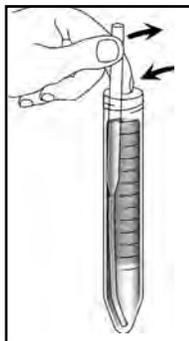


Figure 2

9. Obtain a test result by placing the ampoule, flat end first, into the comparator. Hold the comparator up toward a source of light and view from the bottom. Rotate the comparator until the best color match is found (fig. 3).

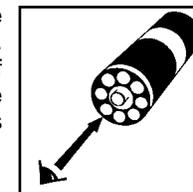


Figure 3

## Tip Breaker

The tip breaker opens for easy disposal of the glass tips (pull lever away from body of tip breaker or pull open the side wall). The tip breaker will work most effectively if the tips are emptied out frequently.

## Test Method

The Detergents CHEMets®<sup>1</sup> test kit employs the methylene blue extraction method<sup>2,3,4</sup>. Anionic detergents react with methylene blue to form a blue complex that is extracted into an immiscible organic solvent. The intensity of the blue color is directly related to the concentration of "methylene blue active substances (MBAS)" in the sample. Anionic detergents are one of the most prominent methylene blue active substances. Test results are expressed in ppm (mg/Liter) linear alkylbenzene sulfonate (equivalent weight 325).

1. CHEMets is a registered trademark of CHEMetrics, Inc. U.S. Patent No. 3,634,038
2. APHA Standard Methods, 22nd ed., Method 5540 C - 2000
3. EPA Methods for Chemical Analysis of Water and Wastes, Method 425.1 (1983)
4. ASTM D 2330-02, Methylene Blue Active Substances

## Safety Information

Read SDS (available at [www.chemetrics.com](http://www.chemetrics.com)) before performing this test procedure. Wear safety glasses and protective gloves.



[www.chemetrics.com](http://www.chemetrics.com)  
4295 Catlett Road, Midland, VA 22728 U.S.A.  
Phone: (800) 356-3072; Fax: (540) 788-4856  
E-Mail: [orders@chemetrics.com](mailto:orders@chemetrics.com)

Feb. 18, Rev. 10

# Detergents CHEMets® Kit

K-9404/R-9404: 0 - 1400 ppm

## Safety Information

Read MSDS (available at [www.chemetrics.com](http://www.chemetrics.com)) before performing this test procedure. Wear safety glasses and protective gloves.

## Test Procedure

1. Rinse the reaction tube with **detergent free water**, then fill it to the 5 mL mark with **detergent free water**.
2. Place a yellow pipette tip firmly onto the end of the orange MiniPet®<sup>5</sup> (fig. 1).  
**NOTE:** The pipette tips are not designed for re-use. Use a new tip for each sample dilution.
3. Depress the plunger on the minipet. Immerse the tip in the sample to be tested and release the plunger. When the plunger is released, a portion of the test sample will be drawn into the yellow tip (fig. 2).  
**NOTE:** The end of the yellow tip must not be touching the side or bottom of the sample container.
4. Holding the minipet over the reaction tube, depress the plunger to dispense the sample (fig. 3).
5. Cap the reaction tube and invert it to mix the contents.
6. While holding the double-tipped ampoule in a vertical position, snap the upper tip using the tip breaking tool (fig. 4).
7. Invert the ampoule and position the open end over the reaction tube. Snap the upper tip and allow the contents to drain into the reaction tube (fig. 4).
8. Cap the reaction tube and shake it vigorously for **30 seconds**. Allow the tube to stand undisturbed for **1 minute**.
9. Make sure that the flexible tubing is firmly attached to the CHEMet ampoule tip.
10. Insert the CHEMet assembly (tubing first) into the reaction tube making sure that the end of the flexible tubing is at the bottom of the tube. Break the tip of the CHEMet ampoule by gently pressing it against the side of the reaction tube (fig. 5). The ampoule should draw in fluid only from the organic phase (bottom layer).
11. When filling is complete, remove the CHEMet assembly from the reaction tube.
12. Remove the flexible tubing from the CHEMet ampoule and wipe all liquid from the exterior of the ampoule. Place an ampoule cap firmly onto the tip of the CHEMet ampoule. Invert the ampoule several times, allowing the bubble to travel from end to end.
13. Obtain a test result by placing the ampoule between the color standards until the best color match is found (fig 6).



Figure 1



Figure 2



Figure 3

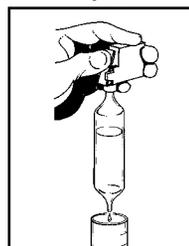


Figure 4



Figure 5

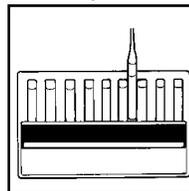


Figure 6

## Tip Breaker

The tip breaker opens for easy disposal of the glass tips (pull lever away from body of tip breaker or pull open the side wall). The tip breaker will work most effectively if the tips are emptied out frequently.

## Test Method

The Detergents CHEMets®<sup>1</sup> test kit employs the methylene blue extraction method<sup>2,3,4</sup>. Test results are expressed in ppm (mg/Liter) linear alkylbenzene sulfonate (equivalent weight 325).

1. CHEMets is a registered trademark of CHEMetrics, Inc. U.S. Patent No. 3,634,038
2. APHA Standard Methods, 21st ed., method 5540 C (2005)
3. EPA Methods for Chemical Analysis of Water and Wastes, method 425.1 (1983)
4. ASTM D 2330-02, Methylene Blue Active Substances
5. MiniPet is a registered trademark of Tricontinent Scientific, Inc.



[www.chemetrics.com](http://www.chemetrics.com)  
4295 Catlett Road, Midland, VA 22728 U.S.A.  
Phone: (800) 356-3072; Fax: (540) 788-4856  
E-Mail: [orders@chemetrics.com](mailto:orders@chemetrics.com)

Oct. 12, Rev. 3



# Detergents Test Kit

DE-2 (143203)

DOC326.97.00058

## Test preparation

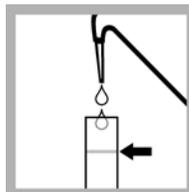
**CAUTION:** Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

- Put the color disc on the center pin in the color comparator box (numbers to the front).
- Use sunlight or a lamp as a light source to find the color match with the color comparator box.
- Rinse the tubes with sample before the test. Rinse the tubes with deionized water after the test.
- If the color match is between two segments, use the value that is in the middle of the two segments.
- If the color disc becomes wet internally, pull apart the flat plastic sides to open the color disc. Remove the thin inner disc. Dry all parts with a soft cloth. Assemble when fully dry.
- Use the filtration procedure for samples that contain turbidity.
- If the test result is more than the maximum limit, dilute the sample as follows. Use the dropper to add 1 mL of sample to a tube. Dilute the sample to the 20-mL line with deionized water. Use the diluted sample in the test procedure. Multiply the value in the scale window by 20 to get the test result in mg/L.
- To use the included demineralizer bottle, fill the bottle with tap water and shake to mix. Use this water as deionized water in the test procedure. Fill the bottle again when empty. Replace the resin after the bottle is filled approximately 100 times.
- Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

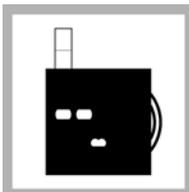
## Replacement items

Description	Quantity	Item no.
Chloroform, ACS grade	500 mL	1445849
Detergents Reagent	100 mL MDB	105932
Wash Water Buffer Solution	500 mL	99949
Color comparator box	each	173200
Color disc, detergents, 0–1.2 mg/L	each	9265700
Demineralizer bottle, 177-mL capacity	each	1429900
Dropper, glass, 0.5- and 1.0-mL marks	5/pkg	1419705
Filter thimble	1	51200
Glass viewing tubes, 5-mL and 20-mL marks	6/pkg	173606
Glass wool	5 g	252074
Pipet bulb	1	178600
Stoppers for viewing tubes, No. 2 plastic	6/pkg	1448001
Test tube, 13 x 100 mm	10/pkg	56510
Transfer pipet	1	221800

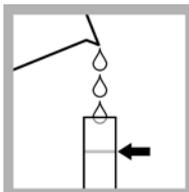
## Test procedure—Detergents (0–1.2 mg/L as LAS (linear alkylate sulfonate) and/or ABS (alkyl benzene sulfonate))



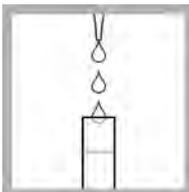
1. Fill a tube to the top line (20 mL) with deionized water.



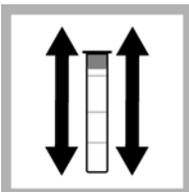
2. Put the tube into the left opening of the color comparator box.



3. Fill a second tube to the top line (20 mL) with sample.



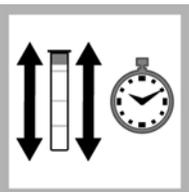
4. Add 12 drops of Detergents Reagent.



5. Put the stopper on the second tube. Shake to mix.



6. Add chloroform to the first line (5 mL). Chloroform is heavier than water and goes to the bottom of the tube.



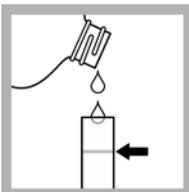
7. Put the stopper on the tube. Shake vigorously for 30 seconds.



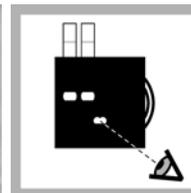
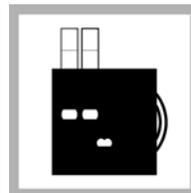
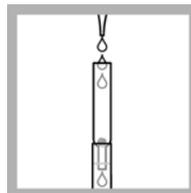
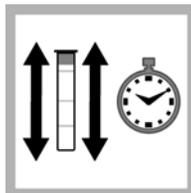
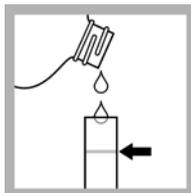
8. Do not touch the tube for 1 minute to let the chloroform separate from the sample.



9. Use the transfer pipet to remove the top water layer from the tube. Discard the water.



10. Add Wash Water Buffer Solution to the top mark (20 mL).



**11.** Use the transfer pipet to remove the Wash Water Buffer. Discard the buffer. This step removes the remaining water sample.

**12.** Add Wash Water Buffer to the top mark (20 mL).

**13.** Put the stopper on the tube. Shake vigorously for 30 seconds.

**14.** Do not touch the tube for 1 minute to let the chloroform separate.

**15.** If the sample contains turbidity, complete the [Filtration procedure for turbid samples](#) on page 2.

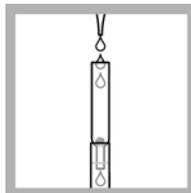
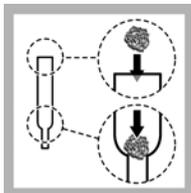
**16.** Put the second tube into the color comparator box.

**17.** Hold the color comparator box in front of a light source. Turn the color disc to find the color match.

**18.** Read the result in mg/L in the scale window.

### Filtration procedure for turbid samples

If the sample contains turbidity, pour the chloroform layer through a filter during the test procedure after step 15.



**1.** Put a small ball (the size of a large pea) of glass wool in the filter thimble.

**2.** Use the transfer pipet to remove the chloroform layer from the viewing tube.

**3.** Put the filter thimble on a clean test tube. Add the chloroform to the filter thimble.

**4.** Use the filtered chloroform to continue the test procedure after step 15.



- **Free and Total Chlorine Test Kit**
- **Trousse d'analyse chlore libre et total**
- **Test Kit auf freies und Gesamtchlor**
- **Kit para cloro libre y total**

0-3.5 mg/L

•Mod. CN-66/-66F/-66T

•# 2231-01,-02,-03

- To ensure accurate results, read carefully before proceeding.
- Pour obtenir des résultats exacts, lire attentivement le mode d'emploi avant d'utiliser la trousse.
- Um genaue Ergebnisse zu gewährleisten, lesen Sie das Folgende bitte aufmerksam durch, bevor Sie fortfahren.
- Para obtener resultados precisos, lea detenidamente las instrucciones antes de proceder al análisis.



#### WARNING

*Handling chemical samples, standards, and reagents can be dangerous. Review the Material Safety Data Sheets before handling any chemicals.*

#### ATTENTION

*La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les fiches de données de sécurité des produits avant de manipuler tout produit chimique.*

#### WARNUNG

*Die Handhabung chemischer Proben, Standards und Reagenzien kann gefährlich sein. Bitte gehen Sie die Material sicherheitsdatenblätter durch, bevor Sie Chemikalien handhaben.*

#### ADVERTENCIA

*El manejo de sustancias químicas, patrones y reactivos, puede resultar peligroso. Lea las fichas de informaciones de seguridad de materiales antes de manipular cualquier producto químico.*

## Measuring Hints and General Test Information

- Wash all labware between tests. Contamination may alter test results. Clean with a non-abrasive detergent or a solvent such as isopropyl alcohol. Use a soft cloth for wiping or drying. Do not use paper towels or tissue on plastic tubes as this may scratch them. Rinse with clean water (preferably deionized water).
- Rinse all viewing tubes thoroughly with the sample water before testing.
- To open PermaChem® Powder Pillows:
  1. Tap the bottom of the pillow on a hard surface.
  2. Tear open the pillow along the dashed line.
  3. Open the pillow and form a spout by squeezing the side edges.
  4. Pour the contents into the sample.
- Accuracy is not affected by undissolved powder.
- Read the result of the Free Chlorine Test within one minute of the addition of the powder.
- Read the result of the Total Chlorine Test between three and six minutes after addition of the powder.
- Hach strongly recommends that, for optimum test results, reagent accuracy be checked with each new lot of reagents. Use the standard solution included in this kit or listed in the *OPTIONAL REAGENTS AND EQUIPMENT* section. Follow the instructions included with each standard solution.

## Conseils pour les mesures et informations générales sur l'analyse

- Laver toute la verrerie entre les analyses. La contamination peut fausser les résultats d'analyses. Laver avec un détergent non abrasif ou un solvant tel que l'isopropanol. Utiliser un tissu doux pour essuyer ou sécher. Ne pas utiliser de tissu ou papier d'essuyage sur les tubes en plastique pour ne pas les rayer. Rincer à l'eau propre (de préférence de l'eau désionisée).
- Rincer soigneusement tous les tubes colorimétriques avec l'échantillon d'eau avant l'analyse.
- Pour ouvrir les sachets PermaChem®:
  1. Taper le bas du sachet sur une surface dure.
  2. Déchirer le sachet en suivant le pointillé.
  3. Ouvrir le sachet et former un bec en rapprochant les bords latéraux.
  4. Verser le contenu dans l'échantillon.
- L'exactitude n'est pas affectée par la poudre non dissoute.
- Lire le résultat de la mesure du chlore libre en moins d'une minute après l'addition du réactif.
- Lire le résultat de la mesure du chlore total entre trois et six minutes après l'addition du réactif.
- Pour de meilleurs résultats, Hach recommande vivement de vérifier la validité du réactif pour chaque nouveau lot de réactifs. Utiliser la solution étalon contenue dans cette trousse ou listée dans la partie *REACTIFS ET EQUIPEMENTS OPTIONNELS*. Suivre les instructions fournies avec chaque solution étalon.

## Meßtips und allgemeine Testinformationen

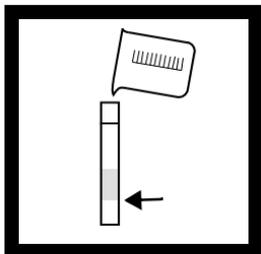
- Waschen Sie alle Laborartikel zwischen den Tests. Verunreinigung kann die Testergebnisse verfälschen. Reinigen Sie sie mit einem nicht scharfen Detergent oder einem Lösungsmittel wie zum Beispiel Isopropylalkohol. Verwenden Sie für das Abwischen oder Abtrocknen ein weiches Tuch. Verwenden Sie bei den Plastikröhrchen keine Papierhandtücher oder Tissue-Papier, da dieses sie zerkratzen kann. Spülen Sie mit sauberem Wasser (vorzugsweise entsalztes Wasser).
- Spülen Sie alle Prüfröhrchen vor dem Test gründlich mit dem Probenwasser.
- Öffnen der PermaChem<sup>®</sup>-Pulverkissen:
  1. Klopfen Sie mit dem Boden des Kissens auf eine harte Oberfläche.
  2. Öffnen Sie das Kissen und bilden Sie durch Drücken der Seitenkanten einen Ausgießer.
  3. Schütten Sie den Inhalt in die Probe.
- Die Genauigkeit wird durch unaufgelöstes Pulver nicht beeinträchtigt.
- Lesen Sie das Ergebnis des Tests auf freies Chlor innerhalb von 1 Minute nach der Zugabe des Pulvers ab.
- Lesen Sie das Ergebnis des Gesamtchlor-Tests zwischen drei und sechs Minuten nach der Zugabe des Pulvers ab.
- Hach empfiehlt dringend, für optimale Testergebnisse die Genauigkeit des Reagenzes bei jeder neuen Charge von Reagenzien zu überprüfen. Verwenden Sie dazu die diesem Kit beiliegende Standardlösung oder die im Abschnitt *ZUSÄTZLICHE REAGENZIEN UND ZUBEHÖR* aufgeführte Standardlösung. Befolgen Sie die Anweisungen, die jeder Standardlösung beiliegen.

## Consejos para la medición e información general sobre el análisis

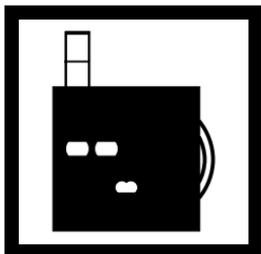
- Lavar todo el material de laboratorio entre los análisis. La contaminación puede alterar los resultados. Limpiar con detergentes no abrasivos o con un disolvente como el alcohol isopropílico. Utilizar un paño suave para limpiar o secar. No utilizar ni toallitas ni pañuelos de papel para limpiar los tubos de plástico para no rayarlos. Aclarar con agua limpia (preferentemente agua desionizada).
- Enjuagar todos los tubos para colorimetría abundantemente con la muestra de agua antes de realizar el análisis.
- Para abrir las Cápsulas de Reactivo PermaChem<sup>®</sup> proceda del siguiente modo:
  1. Golpee ligeramente la parte inferior de la cápsula contra una superficie dura.
  2. Tire de la línea de puntos para abrir.
  3. Abra la cápsula y presione sobre los laterales de la misma hasta que se forme un pico.
  4. Vierta el contenido en la muestra.
- La exactitud del análisis no se verá afectada por restos de polvos de reactivo sin disolver.
- No deje transcurrir más de un minuto entre la adición de los polvos de reactivo y la lectura del resultado de la determinación de cloro libre.
- Lea el resultado de la determinación de cloro libre entre un minuto, después de haber añadido los polvos de reactivo.
- Lea el resultado de la determinación de cloro total entre tres y seis minutos, después de haber añadido los polvos de reactivo.
- Para obtener mejores resultados, Hach recomienda encarecidamente comprobar la validez del reactivo con cada nuevo lote. Utilice para ello la solución patrón incluida en este kit o relacionada en la sección de *REACTIVOS Y EQUIPAMIENTO OPCIONALES*. Siga las instrucciones que se incluyen en cada solución patrón.

• **Free Chlorine Test • Technique chlore libre**  
• **Test Auf Freies Chlor • Determinación de cloro libre**

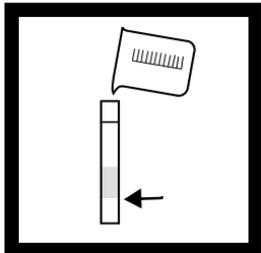
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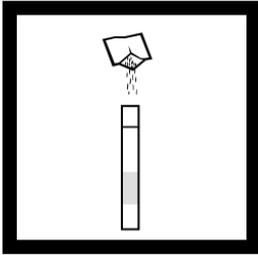
- 1.** Fill a viewing tube to the first (5-mL) line with sample water. This is the blank.
- Remplir un tube colorimétrique jusqu'au premier trait (5-mL) avec l'échantillon d'eau. Ceci est le blanc.
  - Füllen Sie ein Prüfröhrchen bis zur ersten (5-mL) Linie mit Probenwasser. Dieses ist die Blindprobe.
  - Llene un tubo para colorimetría hasta la primera marca (5-mL) con la muestra de agua. Esto constituye el blanco.



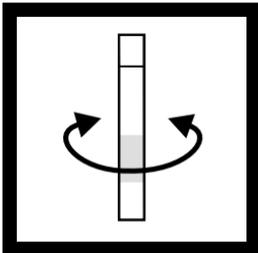
- 2.** Place this tube in the top left opening of the color comparator.
- Placer ce tube dans l'ouverture supérieure gauche du comparateur.
  - Stellen Sie dieses Röhrchen in die obere linke Öffnung des Farbkomparators.
  - Coloque este tubo en la abertura superior izquierda del comparador.



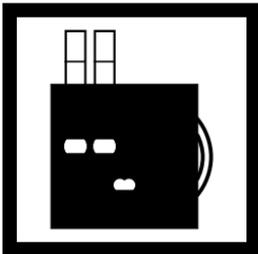
- 3.** Fill another viewing tube to the first (5-mL) line with sample water.
- Remplir un autre tube jusqu'au premier trait (5-mL) avec l'échantillon d'eau.
  - Füllen Sie ein weiteres Prüfröhrchen bis zur ersten (5-mL) Linie mit Probenwasser.
  - Llene otro tubo para colorimetría hasta la primera marca (5-mL) con la muestra de agua.



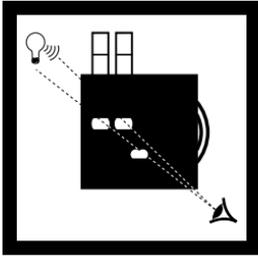
- 4.** Add the contents of one DPD Free Chlorine Reagent Powder Pillow to the second tube. Complete the test and read the result within one minute of the addition of the powder.
- Ajouter le contenu d'un sachet de réactif DPD chlore libre au second tube. Terminer l'essai et lire le résultat en moins d'une minute après l'addition du réactif.
  - Geben Sie den Inhalt eines DPD-freien Chlorreagenz-Pulverkissens in das zweite Röhrchen. Beenden Sie den Test und lesen Sie das Ergebnis innerhalb von einer Minute nach der Zugabe des Pulvers ab.
  - Vierta el contenido de una de las cápsulas de reactivo de cloro libre DPD en el segundo tubo de los preparados anteriormente. Realice el análisis y lea el resultado en el curso de un minuto tras la adición del polvo de reactivo.



- 5.** Swirl to mix.
- Agiter pour mélanger.
  - Schwenken Sie zum Vermischen.
  - Agite para mezclar.

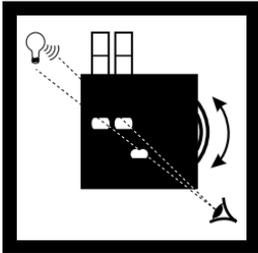


- 6.** Place the second tube in the top right opening of the color comparator.
- Placer le second tube dans l'ouverture supérieure droite du comparateur.
  - Setzen Sie das zweite Röhrchen in die obere rechte Öffnung des Farbkomparators.
  - Coloque el segundo tubo en la abertura superior derecha del comparador.



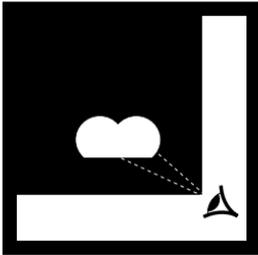
7. Hold comparator up to a light source such as the sky, a window or a lamp. Look through the openings in front.

- Tenir le comparateur face à une surface uniformément éclairée (ciel, lampe, fenêtre) et regarder par les ouvertures de la face antérieure du comparateur.
- Halten Sie den Komparator gegen eine Lichtquelle, wie zum Beispiel den Himmel, ein Fenster oder eine Lampe. Sehen Sie durch die Öffnungen vorn.
- Oriente el comparador hacia una fuente de luz, tal como el cielo, una ventana o una lámpara. Mire a través de las aberturas frontales del comparador.



8. Rotate the color disc until the color matches in the two openings.

- Tourner le disque jusqu'à égalité des teintes dans les deux ouvertures.
- Drehen Sie die Farbscheibe, bis die Farbe in den beiden Öffnungen übereinstimmt.
- Haga girar el disco de color hasta que el color coincida en ambas aberturas.



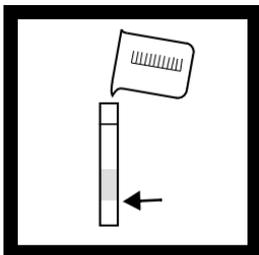
9. Read the mg/L free chlorine in the scale window.

- Lire la concentration du chlore libre en mg/L dans la fenêtre de l'échelle.
- Lesen Sie die mg/L freies Chlor in dem Skalenfenster ab.
- Lea los mg/L de cloro libre en la ventanilla de la escala.

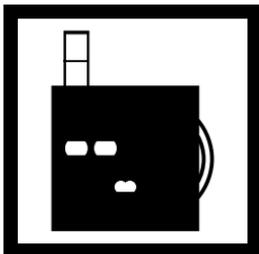
## • Total Chlorine Test • Technique chlore total

## • Gesamtchlor-Test • Determinación de cloro total

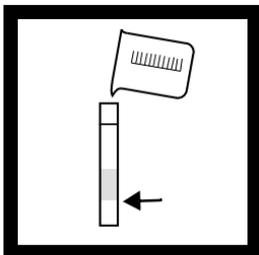
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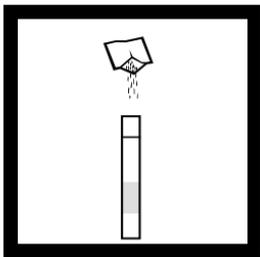
1. Fill a viewing tube to first (5-mL) line with sample water. This is the blank.
  - Remplir un tube colorimétrique jusqu'au premier trait (5-mL) avec l'échantillon d'eau. Ceci est le blanc.
  - Füllen Sie ein Prüfröhrchen bis zur ersten (5-mL) Linie mit Probenwasser. Dieses ist die Blindprobe.
  - Llène un tubo para colorimetría hasta la primera marca (5-mL) con la muestra de agua. Esto constituye el blanco.



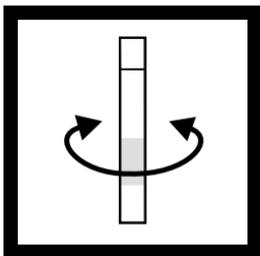
2. Place this tube in the top left opening of the color comparator.
  - Placer ce tube dans l'ouverture supérieure gauche du comparateur.
  - Stellen Sie dieses Röhrchen in die obere linke Öffnung des Farbkomparators.
  - Coloque este tubo en la abertura superior izquierda del comparador.



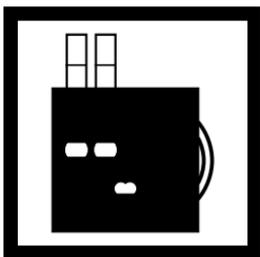
3. Fill another viewing tube to the first (5-mL) line with sample water.
  - Remplir un autre tube jusqu'au premier trait (5-mL) avec l'échantillon d'eau.
  - Füllen Sie ein weiteres Prüfröhrchen bis zur ersten (5-mL) Linie mit Probenwasser.
  - Llène otro tubo para colorimetría hasta la primera marca (5-mL) con la muestra de agua.



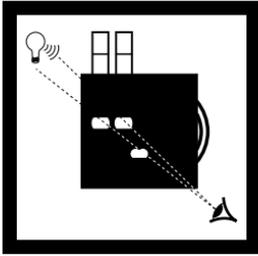
- 4.** Add the contents of one DPD Total Chlorine Reagent Powder Pillow to the second tube.
- Ajouter le contenu d'un sachet de réactif DPD chlore total au second tube.
  - Geben Sie den Inhalt eines DPD-Gesamtchlorreagenz-Pulverkissens in das zweite Röhrchen.
  - Vierta el contenido de una de las Cápsulas de Reactivo de Cloro Total DPD en el segundo tubo de los preparados anteriormente.



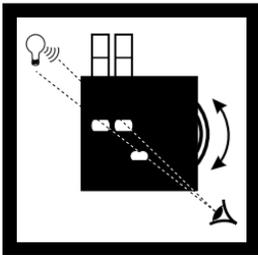
- 5.** Swirl to mix. Wait three minutes. The result of the test must be read within six minutes of the addition of the powder.
- Agiter pour mélanger. Attendre trois minutes. Lire le résultat en moins de six minutes après l'addition du réactif.
  - Schwenken Sie es zum Vermischen. Warten Sie drei Minuten lang. Das Testergebnis muß innerhalb von sechs Minuten nach der Zugabe des Pulvers abgelesen werden.
  - Agite hasta mezclar. Espere tres minutos. El resultado del análisis debe leerse antes de transcurridos seis minutos desde la adición del polvo de reactivo.



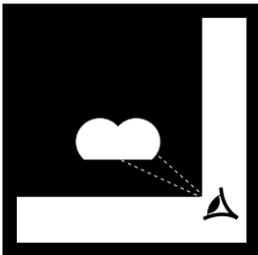
- 6.** Place the second tube in the top right opening of the color comparator.
- Placer le second tube dans l'ouverture supérieure droite du comparateur.
  - Stellen Sie das zweite Röhrchen in die obere rechte Öffnung des Farbkomparators.
  - Coloque el segundo tubo en la abertura superior derecha del comparador de colores.



- 7.** Hold comparator up to a light source such as the sky, a window or a lamp. Look through the openings in front.
- Tenir le comparateur face à une surface uniformément éclairée (ciel, lampe, fenêtre) et regarder par les ouvertures de la face antérieure du comparateur.
  - Halten Sie den Komparator gegen eine Lichtquelle, wie zum Beispiel den Himmel, ein Fenster oder eine Lampe. Sehen Sie durch die Öffnungen vorn.
  - Oriente el comparador hacia una fuente de luz, tal como el cielo, una ventana o una lámpara. Mire a través de las aberturas frontales del comparador.



- 8.** Rotate the color disc until the color matches in the two openings.
- Tourner le disque jusqu'à égalité des teintes dans les deux ouvertures.
  - Drehen Sie die Farbscheibe, bis die Farbe in den beiden Öffnungen übereinstimmt.
  - Haga girar el disco de color hasta que el color coincida en ambas aberturas.



- 9.** Read the mg/L total chlorine in the scale window.
- Lire la concentration du chlore total en mg/L dans la fenêtre de l'échelle.
  - Lesen Sie die mg/L Gesamtchlor im Skalenfenster ab.
  - Lea los mg/L de cloro total en la ventanilla de la escala.

## REPLACEMENTS

<b>Description</b>	<b>Unit</b>	<b>Cat. No.</b>
Color Comparator Box .....	each.....	1732-00
Color Disc, DPD Chlorine, 0–3.5 mg/L.....	each.....	21988-00
Color Viewing Tube, plastic, with cap .....	4/pkg.....	46600-04
DPD Free Chlorine Reagent Powder Pillows .....	100/pkg.....	14077-99
DPD Total Chlorine Reagent Powder Pillows .....	100/pkg.....	14076-99

## REACTIFS ET PIÈCES DE RECHANGE

<b>Désignation</b>	<b>Unité</b>	<b>Réf. N°</b>
Comparteur .....	1.....	1732-00
Disque coloré chlore DPD, 0–3,5 mg/L.....	1.....	21988-00
Tube colorimétrique en plastique avec bouchon .....	4/paq.....	46600-04
Réactif DPD chlore libre, 5 mL .....	100/paq.....	14077-99
Réactif DPD chlore total, 5 mL.....	100/paq.....	14076-99

## VERBRAUCHSMATERIAL UND ERSATZTEILE

<b>Beschreibung</b>	<b>Einheit</b>	<b>Kat. Nr.</b>
Farbkomparator .....	1.....	1732-00
Farbscheibe, DPD-Chlor, 0–3,5 mg/L.....	1.....	21988-00
Farbprüfröhrchen, Plastik, mit Kappe .....	4/Stck.....	46600-04
DPD freies Chlorreagenz-Pulverkissen.....	100/Stck.....	14077-99
DPD Gesamtchlorreagenz-Pulverkissen .....	100/Stck.....	14076-99

## REACTIVOS Y MATERIALES

<b>Descripción</b>	<b>Unidad</b>	<b>Nº Ref.</b>
Comparador de Colores.....	1.....	1732-00
Disco de Color Cloro DPD, 0–3.5 mg/L.....	1.....	21988-00
Tubo Para Colorimetría de plástico, con tapa protectora .....	4/lote.....	46600-04
Cápsulas de Reactivo de Cloro Libre DPD.....	100/lote.....	14077-99
Cápsulas de Reactivo de Cloro Total DPD .....	100/lote.....	14076-99

## OPTIONAL REAGENTS AND EQUIPMENT

<b>Description</b>	<b>Unit</b>	<b>Cat. No.</b>
Caps, for plastic Color Viewing Tubes 46600-04 .....	4/pkg.....	46600-14
Chlorine Standard Solution, 50–75 mg/L, 2-mL PourRite Ampule....	20/pkg.....	14268-20
Color Viewing Tube, glass .....	6/pkg.....	1730-06
Instructions, Color Viewing Tube .....	each.....	46600-88
Stoppers, for glass Color Viewing Tubes 1730-06.....	6/pkg.....	1731-06

## REACTIFS ET EQUIPEMENTS OPTIONNELS

<b>Désignation</b>	<b>Unité</b>	<b>Réf. N°</b>
Bouchons pour tubes en plastique 46600-04.....	4/paq.....	46600-14
Solution étalon chlore, 50–75 mg/L, ampoule PourRite 2 mL .....	20/paq.....	14268-20
Tube colorimétrique en verre .....	6/paq.....	1730-06
Instructions pour tubes colorimétriques .....	1.....	46600-88
Bouchons pour tubes en verre 1730-06.....	6/paq.....	1731-06

## ZUSÄTZLICHE REAGENZIEN UND ZUBEHÖR

<b>Beschreibung</b>	<b>Einheit</b>	<b>Kat. Nr.</b>
Kappe für Plastik-Prüfröhrchen 46600-04 .....	4/Stck.....	46600-14
Chlorstandardlösung, 50–75 mg/L, 2 mL-PourRite-Ampulle .....	20/Stck.....	14268-20
Prüfröhrchen, Glas .....	6/Stck.....	1730-06
Gebrauchsanweisung für Farbprüfröhrchen .....	1.....	46600-88
Stopfen für Glas-Prüfröhrchen 1730-06.....	6/Stck.....	1731-06

## REACTIVOS Y EQUIPAMIENTO OPCIONALES

<b>Descripción</b>	<b>Unidad</b>	<b>Nº Ref.</b>
Tapones para tubos de plástico 46600-04.....	4/lote.....	46600-14
Solución patrón de cloro, 50–75 mg/L ampollas PourRite de 2 mL... ..	20/lote.....	14268-20
Tubos para colorimetría de vidrio .....	6/lote.....	1730-06
Instrucciones para los tubos para colorimetría.....	1.....	46600-88
Tapones para tubos de vidrio 1730-06 .....	6/lote.....	1731-06

- 
- **Pour assistance technique, informations de prix ou informations pour commander, contactez HACH Company ou votre distributeur HACH.**
  - **Technische Unterstützung, aktuelle Preisankünfte und Bestellhilfe erhalten Sie bei Ihrer HACH Vertretung.**
  - **Para obtener asistencia técnica así como información sobre los precios y pedidos, ponerse en contacto con HACH Company o la agencia local de distribución.**
- 



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Loveland, Colorado 80539-0389  
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Chaussée de Namur, 1  
B-5150 Floriffoux (Namur), Belgium  
Telephone : (32) (81) 44.71.71  
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---

**FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:**  
In the U.S.A. - **Call 800-227-4224 toll-free for more information.**  
Outside the U.S.A. - **Contact the HACH office or distributor serving you.**

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# Pro 1030



## USER MANUAL

English



a xylem brand

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Item #605182  
Rev A, January 2013  
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## WARRANTY

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The YSI Professional 1030 instrument (Pro1030) is warranted for three (3) years from date of purchase by the end user against defects in materials and workmanship, exclusive of batteries and any damage caused by defective batteries. Pro1030 cable assemblies are warranted for two (2) years from date of purchase by the end user against defects in material and workmanship. Pro1030 pH and ORP sensors are warranted for one (1) year from date of purchase by the end user against defects in material and workmanship. Pro1030 instruments, cables & sensors are warranted for one (1) year from date of purchase by the end user against defects in material and workmanship when purchased by rental agencies for rental purposes. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio at +1 937 767-7241, 800-897-4151 or visit [www.YSI.com](http://www.YSI.com) for a Product Return Form. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

### LIMITATION OF WARRANTY

This Warranty does not apply to any YSI product damage or failure caused by:

1. Failure to install, operate or use the product in accordance with YSI's written instructions;
2. Abuse or misuse of the product;
3. Failure to maintain the product in accordance with YSI's written instructions or standard industry procedure;
4. Any improper repairs to the product;
5. Use by you of defective or improper components or parts in servicing or repairing the product;
6. Modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI'S LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

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## INTRODUCTION

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Thank you for purchasing the YSI Pro1030, an instrument from the YSI *Professional Series* product family. The Pro1030 measures conductivity, temperature and either pH or ORP in water. The Pro1030 features an impact resistant and waterproof (IP-67) case, a rugged MS-8 (military-spec) cable connector, backlit display, user-selectable sensor options, 50 data set memory and a rubber over-mold case.

The Pro1030 provides valuable instructions and prompts near the bottom of the display that will guide you through operation and use; however, reading the entire manual is recommended for a better understanding of the instrument's features.



*The Pro1030 cannot communicate to a PC via a ProComm communications saddle.*

## GETTING STARTED

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### INITIAL INSPECTION

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Carefully unpack the instrument and accessories and inspect for damage. Compare received parts with items on the packing list. If any parts or materials are damaged or missing, contact YSI Customer Service at 800-897-4151 (+1 937 767-7241) or the authorized YSI distributor from whom the instrument was purchased.

### BATTERY INSTALLATION

---

The instrument requires 2 alkaline C-cell batteries. Under normal conditions, the average battery life is 425 hours at room temperature without using the back light. A battery symbol  will blink in the lower, left corner of the display to indicate low batteries when approximately 1 hour of battery life remains.

To install or replace the batteries:

1. Turn the instrument off and flip over to view the battery cover on the back.
2. Unscrew the four captive battery cover screws.
3. Remove the battery cover and remove the old batteries if necessary.

4. Install the new batteries, ensuring correct polarity alignment (figure 1).
5. Place the battery cover on the back of the instrument and tighten the four screws. Do not over-tighten.

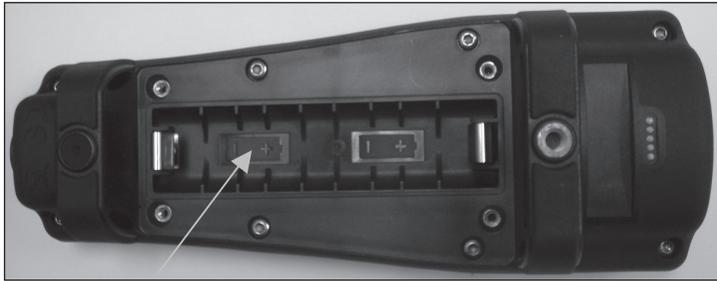


Figure 1. Pro1030 with battery cover removed. Notice battery symbols indicating polarities.

**i** The waterproof instrument case is sealed at the factory and is not to be opened, except by factory-authorized service technicians. Do not attempt to separate the two halves of the instrument case as this may damage the instrument, break the waterproof seal, and will void the warranty.

### KEY PAD

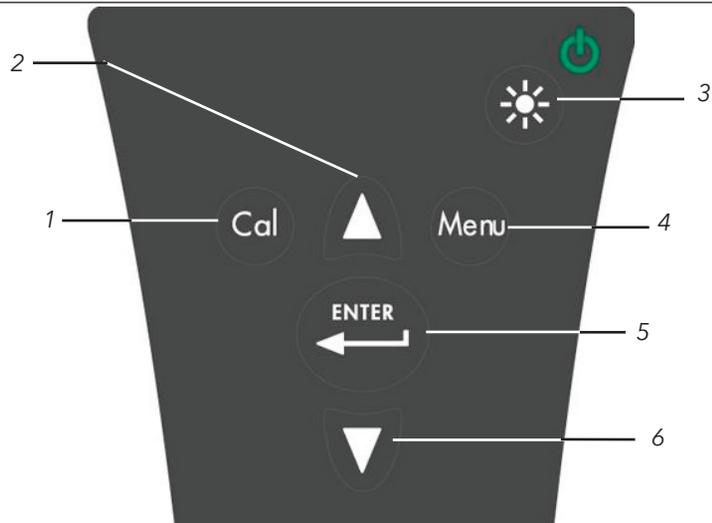


Figure 2, Keypad

Number	Key	Description
1		<b>Calibrate</b> Press and hold for 3 seconds to calibrate. Opens Calibrate menu from the Run screen.
2		<b>Up Arrow</b> Use to navigate through menus, to navigate through box options along the bottom of the Run screen and to increase numerical inputs.
3		<b>Power and Backlight</b> Press once to turn instrument on. Press a second time to turn backlight on. Press a third time to turn backlight off. Press and hold for 3 seconds to turn instrument off.
4		<b>Menu</b> Press to enter the System Setup menu from the Run screen.
5		<b>Enter</b> Press to confirm entries and selections.
6		<b>Down Arrow</b> Use to navigate through menus, to navigate through box options at the bottom of the Run screen and to decrease numerical inputs.

### CONNECTING THE SENSOR AND CABLE

“Bulkhead” refers to the single-pin connector at the end of the probe/cable assembly where an ISE sensor, either pH or ORP, is installed (figure 3). The conductivity and temperature sensors are located above and next to the bulkhead and are not replaceable.



When an ISE sensor is not installed in the cable, the bulkhead connector is not water-proof. Do not submerge the cable without a sensor installed. Submerging the cable without a sensor installed may cause permanent damage to the cable that is not covered under warranty.

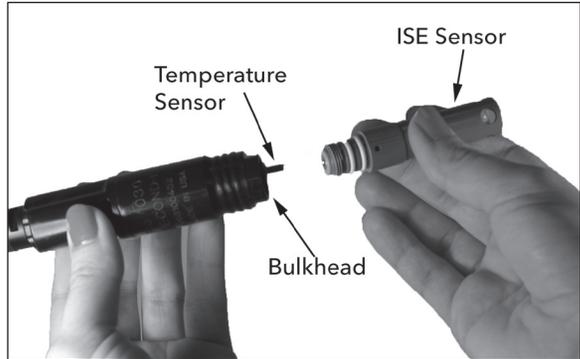


Figure 3

## INSTALLING THE ISE SENSOR

The Pro1030 has three compatible ISE sensors: pH (model #1001), pH-amplified (model #1001A) and ORP (model #1002).

1. Remove the plastic plug from the cable's bulkhead port by pulling it straight out of the port. This can be discarded.
2. Remove the red plastic plug from the sensor's connector by pulling it straight off the sensor. This can be discarded.
3. Ensure both the sensor connector and bulkhead connector are clean and dry.
4. Grasp the sensor with one hand and the cable bulkhead in the other.
5. Push the sensor into the connector on the cable until it is firmly seated with only 1 o-ring visible. Failure to properly seat the sensor may result in damage.
6. Twist the sensor clockwise to engage the threads and finger tighten. Do NOT use a tool. This connection is water-tight.

The ISE sensor is shipped with the tip in a storage bottle. To remove, twist the bottle off the lid and remove the bottle from the sensor. Next, remove the o-ring and slide the lid off the sensor.

## CONNECTING THE PROBE/CABLE ASSEMBLY TO THE INSTRUMENT

To connect the cable, align the keys on the cable connector to the slots on the instrument connector. Push together firmly and then twist the outer ring until it locks into place (figure 4). This connection is water-proof.



Figure 4, Note the keyed connector.

## RUN SCREEN

Press the power/backlight key  to turn the instrument on. The instrument will run through a self test and briefly display a splash screen with system information before displaying the main Run screen (figure 5). A language selection menu will display the first time the Pro1030 is powered on. See the First Power On section of this manual for more information.

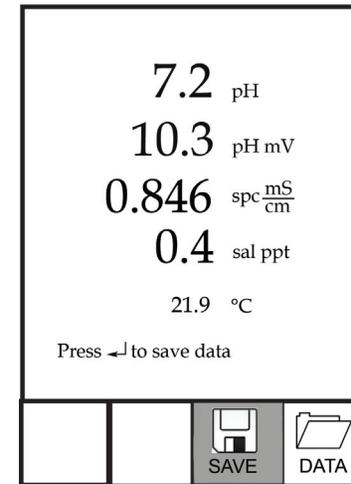


Figure 5, Run screen.

## BACKLIGHT

Once the instrument is powered on, pressing the power/backlight key  will turn on the display backlight. The backlight will remain on until the key is pressed again or after two minutes of not pressing any key on the keypad.

## POWERING OFF

To turn the instrument off, press and hold the power/backlight key  for three seconds.

## NAVIGATION

The up  and down  arrow keys allow you to navigate through the functions of the Pro1030.

### NAVIGATING THE RUN SCREEN

When in the Run screen, the up  and down  arrow keys will move the highlighted box along the bottom options. Once a box is highlighted, press enter to access the highlighted option.

Description of Run screen box functions from left to right:

Option	Description
 SAVE	Highlight and press enter to save displayed data to memory.
 DATA	Highlight and press enter to view and/or erase saved data.

### NAVIGATING THE SYSTEM SETUP MENU

When in the System Setup menu, the up and down arrow keys will move the highlighted bar up and down the system setup options. See the System Setup menu section of this manual for more information about these options.

## FIRST POWER ON

The instrument will step through an initial configuration when powered on for the first time. This will set the language. Use the up or down arrow keys to highlight the appropriate language, then press enter to confirm (figure 6).

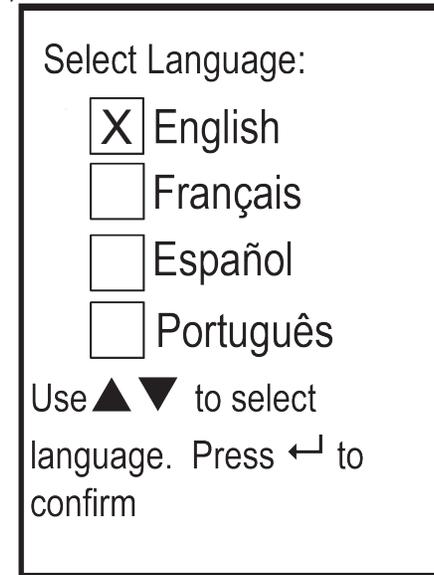


Figure 6, Select language

After selecting a language, the Run screen will be displayed. The next time the instrument is powered up, the Run screen will display immediately after the splash screen.

## SYSTEM SETUP MENU

Press the menu  key to access the System Setup menu. The System Setup menu contains two screens notated as 'pages'. The current page is indicated near the bottom of the display (figure 7).

Use the up and down arrow keys to scroll through menu options and menu pages.

### EXITING THE SYSTEM SETUP MENU

To exit the System Setup menu, press the down arrow key until the ESC - Exit box is highlighted, then press enter to return to the Run screen.

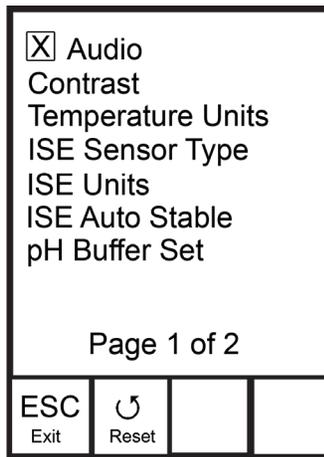


Figure 7, page 1 of System Setup menu.

## AUDIO

Audio can be enabled by highlighting Audio and pressing enter. When enabled, there will be an 'X' in the box next to Audio.

When Audio is enabled, the Pro1030 will beep twice to indicate stability when Auto Stable is enabled. The instrument will also beep when a key is pressed. When Audio is disabled, the Pro1030 will not beep.

## CONTRAST

To adjust the display Contrast, use the up or down arrow keys to highlight Contrast, then press enter. Next, use the up or down arrow keys to adjust the contrast. The up arrow key will darken the contrast and the down arrow key will lighten the contrast. After adjusting the contrast, press enter to save and exit the Contrast adjustment function.

### ALTERNATE CONTRAST ADJUSTMENT OPTION

If necessary, there is an alternate method of adjusting the contrast. To adjust the contrast, press and hold the menu key, then press the up arrow key to darken the contrast or press the down arrow key to lighten the contrast.

## TEMPERATURE UNITS

Highlight Temperature Units and press enter to open a submenu that allows you to change the temperature units displayed on the Run

screen. Highlight the desired unit (Celsius or Fahrenheit) and press enter to enable. The enabled temperature unit will have an 'X' in the box next to it. Only one unit may be enabled at a time. Highlight the ESC-Exit box and press enter to save any changes and to close the Temperature Units submenu.

## ISE SENSOR TYPE

ISE Sensor Type sets the type of ISE sensor being used; either pH (model #1001) or ORP (model #1002).

Use the up or down arrow keys to highlight ISE Sensor Type, then press enter to open a submenu. Highlight the sensor type corresponding to the sensor installed on the cable and press enter to confirm. The enabled sensor type will have an 'X' in the box next to it. Next, use the down arrow key to highlight the ESC - Exit, then press enter to save changes and to close the sensor submenu.

## ISE UNITS

Highlight ISE Units and press enter to open a submenu that allows you to select the ISE units to be displayed on the Run screen. Highlight a unit and press enter to enable or disable it. An enabled ISE unit will have an 'X' in the box next to it. Highlight the ESC-Exit box along the bottom of the display and press enter to save any changes and to close the ISE Units submenu.

When pH is enabled in the ISE Sensor Type menu, there are two selectable measurement units: pH and pH mV. pH mV is the sensor's electrical measurement signal before being converting into pH units. pH mVs can help you determine if you are performing a good calibration and the condition of the pH electrode.

When ORP is enabled in the ISE Sensor Type menu, only ORP mVs can be enabled as the ISE unit.

## AUTO STABLE

Auto Stable utilizes preset values to indicate when a reading is stable. The preset values are adjustable in the System Setup menu. The user can input a % change in measurement reading over 'x' amount of time in seconds. There are two separate Auto Stable controls, one for ISE readings (ISE Auto Stable) and one for conductivity readings (Cond. Auto Stable). ISE Auto Stable is located on the first page of the System Setup menu. Cond. Auto Stable is located on the second page of the System Setup menu.

When Auto Stable is enabled, an **AS** symbol will display next to the reading on the Run screen and blink during stabilization. When the ISE and/or conductivity reading stabilizes based on the Auto Stable settings, the **AS** symbol will display steadily and the instrument will beep twice if Audio is turned on.

ISE Auto Stable can be set to a % change of 0.0 to 9.9% over 3 to 19 seconds. The auto stable criteria is applied to the pH measurement or the ORP mV reading depending on which sensor is enabled in the ISE Sensor menu.

Conductivity Auto Stable can be set to a % change of 0.0 to 1.9% over 3 to 19 seconds. The conductivity auto stable criteria is applied to the conductivity reading, but the AS symbol will display next to all enabled conductivity units.

To enable Auto Stable, highlight either ISE Auto Stable or Cond. Auto Stable, then press enter to open the submenu. Next, use the up or down arrow keys to highlight the % change or seconds (secs) input field, then press enter to make the highlighted field adjustable. Use the up or down arrow keys to adjust the selected value, then press enter to confirm changes. Once you have confirmed any changes, highlight the ESC-Exit box along the bottom of the display and press enter to close the Auto Stable submenu. To disable Auto Stable, set the % Change input to 0.0.

## **pH BUFFER SET**

---

Highlight pH Buffer Set and press enter to open a submenu that allows you to select the Buffer Set used for auto buffer recognition during a pH calibration. There are two buffer set options: USA (4, 7 and 10) and NIST (4.01, 6.86 and 9.18). Highlight the buffer set and press enter to enable. The enabled buffer set will have an 'X' in the box next to it. Highlight the ESC-Exit box and press enter to save any changes and to close the submenu.

## **CONDUCTIVITY UNITS (COND. UNITS)**

---

Highlight Cond. Units (Conductivity Units) and press enter to open a submenu that allows you to select the conductivity units to be displayed on the Run screen. Highlight a unit and press enter to enable or disable it. An enabled conductivity unit will have an 'X' in the box next to it. Highlight the ESC-Exit box along the bottom of the display and press enter to save any changes and to close the conductivity units submenu.

There are seven options for displaying conductivity. Only two units can be enabled at the same time:

- COND-mS/cm displays conductivity in milliSiemens per centimeter.
- COND-uS/cm displays conductivity in microSiemens per centimeter.
- SPC-mS/cm displays Specific Conductance in milliSiemens per centimeter. Specific Conductance is temperature compensated conductivity.
- SPC-uS/cm displays Specific Conductance in microSiemens per centimeter. Specific Conductance is temperature compensated conductivity.
- Sal ppt displays salinity in parts per thousand. The salinity reading is calculated from the instrument's conductivity and temperature values using algorithms found in *Standard Methods for the Examination of Water and Wastewater*.
- TDS g/L displays Total Dissolved Solids in grams per liter. TDS is calculated from conductivity compensated to 25°C using a user-selectable TDS constant.
- TDS mg/L displays Total Dissolved Solids in milligrams per liter. TDS is calculated from conductivity compensated to 25°C using a user-selectable TDS constant.

Note: 1 S = 1 mho.

1 milliSiemen = 1,000 microSiemens.

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## **SPECIFIC CONDUCTANCE**

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The conductivity of a sample is highly dependent on temperature, varying as much as 3% for each change of one degree Celsius (temperature coefficient = 3%/°C). In addition, the temperature coefficient itself varies with the nature of the ionic species present in the sample. Therefore, it is useful to compensate for this temperature dependence in order to quickly compare conductivity readings taken at different temperatures.

The Pro1030 can display non-temperature compensated conductivity as well as temperature compensated Specific Conductance. If Specific Conductance is enabled, the Pro1030 uses the temperature and conductivity values associated with each measurement to calculate a specific conductance value that is temperature compensated based on a user-selected temperature coefficient (0 to 4%) and reference temperature (15 to 25°C).

Using the Pro1030's default reference temperature and temperature coefficient (25 °C and 1.91%), the calculation is carried out as follows:

$$\text{Specific Conductance (25°C)} = \frac{\text{Conductivity of sample}}{1 + 0.0191 * (T - 25)}$$

T = Temperature of the sample in °C

### **SPECIFIC CONDUCTANCE REFERENCE TEMPERATURE (SPC REF. TEMP.)**

---

SPC Ref. Temp. (Specific Conductance Reference Temperature) is the reference temperature used to calculate Specific Conductance. The reference temperature range is 15 and 25°C. The default value is 25°C.

To change the reference temperature, highlight SPC Ref. Temp. and press enter to open the submenu. With the reference temperature highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. Press enter to save the new reference temperature. Next, highlight the ESC-Exit box and press enter to close the submenu.

### **SPECIFIC CONDUCTANCE TEMPERATURE COEFFICIENT (SPC %/°C)**

---

SPC %/°C (Specific Conductance Temperature Coefficient) is the temperature coefficient used to calculate Specific Conductance. The coefficient range is 0.00 to 4.00. The default value is 1.91% which is based on KCl standards.

To change the temperature coefficient, highlight SPC %/°C and press enter to open the submenu. With the temperature coefficient highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. Press enter to save the new coefficient. Next, highlight the ESC-Exit box and press enter to close the submenu.

### **TDS CONSTANT**

---

TDS Constant is a multiplier used to calculate an estimated TDS (Total Dissolved Solids) value from conductivity. The multiplier is used to convert Specific Conductance in mS/cm to TDS in g/L. The Pro1030's default value is 0.65. This multiplier is highly dependent on the nature of the ionic species present in the water sample. To be assured of moderate accuracy for the conversion, you must determine a multiplier

for the water at your sampling site. Use the following procedure to determine the multiplier for a specific sample:

1. Determine the specific conductance of a water sample from the site;
2. Filter a sample of water from the site;
3. Completely evaporate the water from a carefully measured volume of the filtered sample to yield a dry solid;
4. Accurately weigh the remaining solid;
5. Divide the weight of the solid (in grams) by the volume of water used (in liters) to yield the TDS value in g/L for this site;
6. Divide the TDS value in g/L by the specific conductance of the water in mS/cm to yield the conversion multiplier. Be certain to use the correct units.

If the nature of the ionic species at the site changes between sampling studies, the TDS values will be in error. TDS cannot be calculated accurately from specific conductance unless the make-up of the chemical species in the water remains constant.

To change the TDS Constant in the Pro1030, highlight TDS Constant and press enter to open the submenu. With the TDS Constant highlighted, press enter to make the field adjustable. Next, use the up or down arrow key to increase or decrease the value. The input range is 0.30 to 1.00. Press enter to save the new TDS Constant. Next, highlight the ESC-Exit box and press enter to close the submenu.

### **LANGUAGE**

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Highlight Language and press enter to open a submenu that allows you to change the language. Highlight the desired language (English, Spanish, Portuguese, or French) and press enter to enable. The enabled language will have an 'X' in the box next to it. Highlight ESC-Exit box and press enter to save any changes and to close the Language submenu.

The text in the boxes along the bottom of the Run screen will always be displayed in English regardless of the language enabled in the System Setup menu.

### **AUTO SHUTOFF**

---

Auto Shutoff allows you to set the instrument to turn off automatically after a period of time. In the setup menu, use the up or down arrow keys to highlight Auto Shutoff, then press enter to open the submenu. Press enter while the minute field is highlighted to make it adjustable.

Next, use the up or down arrow keys to adjust the shut off time from 0 to 60 minutes. Press enter to save the new shutoff time. Next, highlight the ESC-Exit box and press enter to close the submenu.

To disable Auto Shutoff, set the Time in Minutes to 0 (zero).

## CELL CONSTANT

The Cell Constant displays the cell constant of the conductivity cell. The cell constant is calculated and updated each time a conductivity calibration is performed. The cell constant range is 4.0 to 6.0. Resetting the System Menu resets the cell constant to 5.0.

## RESETTING THE SYSTEM SETUP MENU AND CELL CONSTANT TO FACTORY DEFAULT

To reset the Pro1030 settings and conductivity cell constant back to factory default, press the down arrow key while in the System Setup menu until the Reset -  box is highlighted, then press enter. The instrument will prompt you to confirm the reset. Highlight Yes and press enter to continue with the reset or highlight No and press enter to cancel the reset. A Factory Reset will not affect data saved in the instrument's memory.

The following will be set in the Pro1030 after performing a reset:

<i>Parameter</i>	<i>Reset Defaults</i>
Audio	On
Contrast	Set to mid range
Temperature Units	°C
ISE Sensor Type	pH
ISE Units	pH
ISE Auto Stable	Off (0.0 % Change and 10 seconds)
pH Buffer Set	USA
Conductivity Units	<i>cond mS/cm and spc mS/cm</i>

<i>Parameter</i>	<i>Reset Defaults</i>
Conductivity Auto Stable	Off (0.0 % Change and 10 seconds)
SPC Reference Temperature	25°C
SPC Temperature Coefficient	1.91%/°C
TDS Constant	0.65
Language	English
Auto Shutoff	30 minutes
Conductivity Cell Constant	5.0
pH Calibration	Factory default

## CALIBRATION

### TEMPERATURE

All Pro1030 cables have built-in temperature sensors. Temperature calibration is not required nor is it available.

### pH CALIBRATION

The Pro1030 pH sensor can be calibrated by performing a 1, 2 or 3-point calibration. At least one of the calibration points must be done with pH buffer 7 or 6.86. For auto buffer recognition to work properly with an older or dirty sensor, calibrate in buffer 7 or 6.86 first. For highest accuracy, use fresh, traceable pH buffers and ensure the sensor and calibration vessel are clean.

#### 1-POINT CALIBRATION

1. Place the sensor in pH buffer 7 or 6.86 and allow the temperature and pH readings to stabilize.
2. Press and hold Cal  for three seconds.
3. Highlight pH and press enter. If pH is not listed as an option, check the System Setup menu to ensure pH is enabled in the ISE Sensor Type menu.
4. Highlight 1 point and press enter.
5. If necessary, use the up and down arrow keys to adjust the pH buffer value. Note the pH mV reading which ideally should be between -50 and +50 in buffer 7.
6. Press enter to complete the calibration or press Cal  to cancel.

7. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
8. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting guide for possible solutions.

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## 2-POINT CALIBRATION

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1. Place the sensor in pH buffer 7 or 6.86 and allow the temperature and pH readings to stabilize.
2. Press and hold Cal  for three seconds.
3. Highlight pH and press enter. If pH is not listed as an option, check the System Setup menu to ensure pH is enabled in the ISE Sensor Type menu.
4. Highlight 2 point and press enter.
5. If necessary, use the up and down arrow keys to adjust the pH buffer value. Note the pH mV reading which ideally should be between -50 and +50 in buffer 7.
6. Press enter to continue to second point.
7. Rinse the sensor and place it in the second pH buffer (4/4.01 or 10/9.18).
8. If necessary, use the up and down arrow keys to adjust the pH buffer value.
9. Wait approximately 30 to 60 seconds for the pH sensor to stabilize and for the temperature reading to stabilize. Note the pH mV reading. pH mVs in buffer 4 should be +159 to 180 mV from the previous buffer 7 pH mV value. pH mVs in buffer 10 should be -159 to 180 mV from the previous buffer 7 pH mV value.
10. Press enter to complete the calibration or press Cal  to cancel.
11. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
12. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting section of this manual for possible solutions.

---

## 3-POINT CALIBRATION

---

1. Place the sensor in pH buffer 7 or 6.86 and allow the temperature and pH readings to stabilize.
2. Press and hold Cal  for three seconds.

3. Highlight pH and press enter. If pH is not listed as an option, check the System Setup menu to ensure pH is enabled in the ISE Sensor Type menu.
4. Highlight 3 point and press enter.
5. If necessary, use the up and down arrow keys to adjust the pH buffer value. Note the pH mV reading which should be between -50 and +50 in buffer 7.
6. Press enter to continue to second point.
7. Rinse the sensor and place it in the second pH buffer (4/4.01 or 10/9.18). If necessary, use the up and down arrow keys to adjust the pH buffer value.
8. Wait approximately 30 to 60 seconds for the pH sensor to stabilize and for the temperature reading to stabilize. Note the pH mV reading. pH mVs in buffer 4 should be +159 to 180 mV from the previous buffer 7 pH mV value. pH mVs in buffer 10 should be -159 to 180 mV from the previous buffer 7 pH mV value.
9. Rinse the sensor and place it in the third pH buffer (4/4.01 or 10/9.18). If necessary, use the up and down arrow keys to adjust the pH buffer value.
10. Wait approximately 30 to 60 seconds for the pH sensor to stabilize and for the temperature reading to stabilize. Note the pH mV reading. pH mVs in buffer 4 should be +159 to 180 mV from the previous buffer 7 pH mV value. pH mVs in buffer 10 should be -159 to 180 mV from the previous buffer 7 pH mV value.
11. Press enter to complete the calibration or press Cal  to cancel.
12. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
13. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting section of this manual for possible solutions.

---

## ORP CALIBRATION

---

1. Place the clean sensor in ORP calibration solution. Wait for the ORP and temperature readings to stabilize.
2. Press and hold Cal  for three seconds.
3. Highlight ORP and press enter. If ORP is not listed as an option, check the System Setup menu to ensure ORP is enabled in the ISE Sensor Type menu.
4. Use the up and down arrow keys to adjust the ORP calibration solution value.

5. Wait for the temperature reading to stabilize, then press enter to complete the calibration or press Cal  to cancel.
6. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
7. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting section of this manual for possible solutions.

---

## CONDUCTIVITY CALIBRATION

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Ensure the conductivity sensor is clean and dry before performing a conductivity, specific conductance or salinity calibration.

It is not necessary to calibrate conductivity, specific conductance and salinity. Calibrating one of these parameters will simultaneously calibrate the others. YSI recommends calibrating specific conductance for greatest ease.

Always calibrate with fresh, traceable calibration solution with a value of 1000 uS or more.

Note: 1 mS = 1000 uS

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### CALIBRATING SPECIFIC CONDUCTANCE (SPC) OR CONDUCTIVITY

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Note: When calibrating Specific Conductance, the Pro1030 uses the factory default values for the Specific Conductance Reference Temperature and the Specific Conductance Temperature Coefficient regardless of what is configured in the System Setup Menu. The default value for the Reference Temperature is 25°C and the default value for the Temperature Coefficient is 1.91%/°C. It is important to note that the Temperature Coefficient of a calibration solution is dependent on the contents of the solution. Therefore, for highest accuracy, YSI recommends using a traceable calibration solution made of KCl (potassium chloride) when calibrating Specific Conductance since these solutions typically have a Temperature Coefficient of 1.91%/°C. Additionally, be sure to enter the value of the solution as it is listed for 25°C when calibrating Specific Conductance.

1. Place the sensor into the solution. The solution must cover the holes of the conductivity sensor that are closest to the cable

(figure 8). Ensure the entire conductivity sensor is submerged in the solution or the instrument will read approximately half the expected value. Gently move the probe up and down to remove any air bubbles from the conductivity sensor.

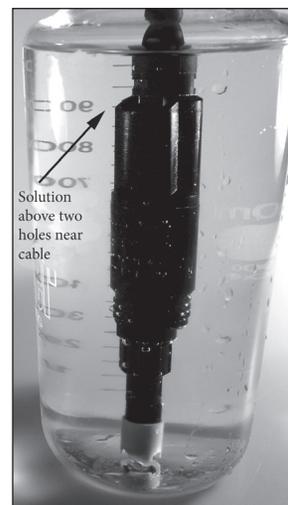


Figure 8, solution above two holes near cable.

2. Turn the instrument on and allow the conductivity and temperature readings to stabilize. Press and hold the Cal key for 3 seconds. Highlight Conductivity and press enter. Next, highlight the desired calibration method, Sp. Conductance or Conductivity, and press enter.
3. Highlight the units you wish to calibrate, either uS/cm or mS/cm, and press enter. 1 mS = 1,000 uS.
4. Use the up or down arrow key to adjust the value on the display to match the value of the conductivity calibration solution. Most conductivity solutions are labeled with a value at 25°C. If calibrating specific conductance, enter the value listed for 25°C. If calibrating conductivity, look up the value of the solution at the solution's current temperature and enter that value into the Pro1030. Press and holding either the up or down arrow key for 5 seconds will move the changing digit one place to the left. The Pro1030 will remember the entered calibration value and display it the next time a conductivity calibration is performed.
5. Press enter to complete the calibration or press Cal to cancel.
6. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.

7. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting section of this manual for possible solutions.

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## CALIBRATING IN SALINITY

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1. Place the sensor into the solution. The solution must cover the holes of the conductivity sensor that are closest to the cable (figure 8). Ensure the entire conductivity sensor is submerged in the solution or the instrument will read approximately half the expected value. Gently move the probe up and down to remove any air bubbles from the conductivity sensor.
2. Turn the instrument on and allow the conductivity and temperature readings to stabilize. Press and hold the Cal key for 3 seconds. Highlight Conductivity and press enter. Next, highlight Salinity and press enter.
3. Use the up or down arrow key to adjust the value on the display to match the value of the salinity solution. Press and holding either the up or down arrow key for 5 seconds will move the changing digit one place to the left. The Pro1030 will remember the entered calibration value and display it the next time a salinity calibration is performed.
4. Press enter to complete the calibration. Or, press Cal to cancel the calibration and return to the Run screen.
5. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and then the instrument will return to the Run screen.
6. If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the Run screen. See the Troubleshooting section of this manual for possible solutions.

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## TAKING MEASUREMENTS

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Before taking measurements, be sure the instrument has been calibrated to ensure the most accurate readings. Install the sensor guard to protect the pH or ORP sensor. Place the probe in the sample to be measured and give the probe a quick shake to release any air bubbles.

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## CONDUCTIVITY

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The conductivity sensor will provide quick readings as long as the entire sensor is submerged and no air bubbles are trapped in the sensor area. Immerse the probe into the sample so the sensors are completely submerged and then shake the probe to release any air bubbles. Occasional cleaning of the sensor may be necessary to maintain accuracy and increase the responsiveness. To clean the sensor, use the soft bristle cleaning brush provided with the instrument and a mild detergent.

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## pH/ORP

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pH and ORP readings are typically quick and accurate. However, it may take the sensors a little longer to stabilize if they become coated or fouled. To improve the response time of a sensor, follow the cleaning steps in the Maintenance section of this manual.

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## SAVING AND VIEWING DATA

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The Pro1030 can store 50 data sets in non-volatile memory for later viewing. A data set includes the values currently on the display, i.e. temperature, dissolved oxygen and two conductivity parameters. Each data point is referenced with a data set number, 01 through 50.

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## SAVING DATA

---

From the Run screen, use the up or down arrow keys to highlight the Save box and press enter to save the current readings. The instrument will indicate the data set is saved and display the saved data set's number (figure 9).

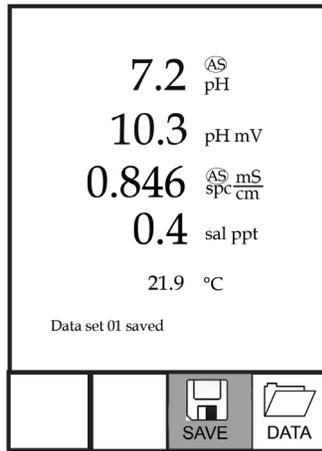


Figure 9, data set saved.

The instrument will display 'Memory Full' if all 50 data sets have been saved and you attempt to save another data set.

## VIEWING AND ERASING SAVED DATA

Data mode allows you to view and erase saved data. From the Run screen, use the up or down arrow keys to highlight Data and press enter to access Data mode. Note that the function boxes at the bottom of the display are different in Data mode (figure 10).

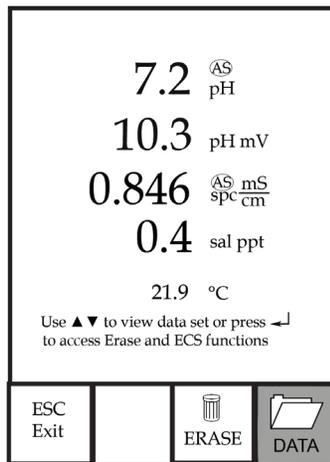


Figure 10, Data mode.

## VIEWING DATA

Once in Data mode, use the up and down arrow keys to view saved data sets in sequential order or press enter to access the bottom functions. After accessing the bottom functions, highlight the Data box and press enter to regain access to viewing data. The data set displayed is indicated by the data set number, 01 through 50.

## ERASING DATA

While viewing saved data, press the enter key to access the function boxes at the bottom of the display. Next, use the up or down arrow keys to highlight Erase, then press enter. The instrument will give you the option to erase one data set or all data sets (figure 11).

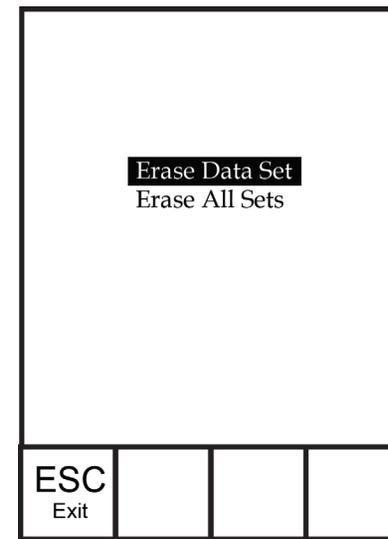


Figure 11, Erase data mode.

Use the up or down arrow key to select Erase Data Set, Erase All Sets or the ESC-Exit function box, then press enter to confirm.

Select ESC-Exit and press enter to exit Erase mode without erasing any data.

Select Erase Data Set and press enter to erase the data set that was displayed before entering Erase mode. For example, if data set 12 was displayed before entering erase mode, and Erase Data Set is selected, Data Set 12 will be erased from memory and the data sets AFTER that number will move up to keep them sequential. For example, if there are 15 records and number 12 is erased then 13 becomes 12, 14 becomes

13, and 15 becomes 14. The instrument will return to Data mode after erasing one data set.

Select Erase All Data Sets and press enter to clear the Pro1030 memory and return to Data mode.

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## EXITING DATA MODE

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While in Data mode, press enter to access the bottom functions. Next, highlight the ESC-Exit box and press enter to return to the Run screen.

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# CARE, MAINTENANCE AND STORAGE

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This section describes the proper procedures for care, maintenance and storage of the sensors. The goal is to maximize their lifetime and minimize down-time associated with improper sensor usage.

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## GENERAL MAINTENANCE

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### GENERAL MAINTENANCE - GASKET AND O-RINGS

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The instrument utilizes a gasket and o-rings as seals to prevent water from entering the battery compartment and the sensor port. Following the recommended procedures will help keep the instrument functioning properly.

If the gasket, o-rings and sealing surfaces are not maintained properly, it is possible that water can enter the battery compartment and/or sensor port of the instrument. If water enters these areas, it can damage the battery terminals or sensor port causing loss of battery power, false readings and corrosion to the sensors or battery terminals. Therefore, when the battery compartment lid is removed, the gasket that provides the seal should be carefully inspected for contamination (i.e. debris, grit, etc.) and cleaned with water and mild detergent if necessary.

The same inspection should be made of the o-rings associated with the ISE sensor connector when replacing the ISE sensor. The o-rings should be free of dirt or debris before installing the sensor onto the cable.

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### GENERAL MAINTENANCE - ISE SENSOR PORT

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It is important that the entire sensor connector end be dry when installing, removing or replacing the sensor. This will prevent water

from entering the port. Once the ISE sensor is removed, examine the connector inside the port. If any moisture is present, use compressed air to completely dry the connector or let it air dry. If the connector is corroded, contact YSI Technical Support or the YSI authorized dealer where you purchased the instrument.

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## SENSOR MAINTENANCE

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*Typical working life for pH and ORP sensors is approximately 12-24 months depending on usage, storage and maintenance. Proper storage and maintenance generally extends the sensor's working life.*

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### SENSOR MAINTENANCE - TEMPERATURE

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You must keep the temperature sensor free of build up. No additional maintenance is required. A toothbrush can be used to scrub the temperature sensor if needed.

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### SENSOR MAINTENANCE - CONDUCTIVITY

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The openings that allow sample access to the conductivity electrodes should be cleaned regularly. The small cleaning brush included in the Maintenance Kit is intended for this purpose. Dip the brush in clean water and insert it into each hole 10 to 12 times. In the event that deposits have formed on the electrodes, it may be necessary to use a mild detergent (laboratory grade soap or bathroom foaming tile cleaner) with the brush. Rinse thoroughly with clean water, then check the response and accuracy of the conductivity cell with a calibration solution.

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### SENSOR MAINTENANCE - pH AND ORP

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Cleaning is required whenever deposits or contaminants appear on the glass and/or platinum sensor surfaces or when the sensor's response slows. The cleaning can be chemical and/or mechanical.

Removing the sensor from the cable may make cleaning easier. Initially, use clean water and a soft clean cloth, lens cleaning tissue, or cotton swab to remove all foreign material from the glass bulb and/or platinum button. Then use a moistened cotton swab to carefully remove any material that may be blocking the reference electrode junction of the sensor.

If good pH and/or ORP response is not restored, perform the following additional procedure:

1. Soak the sensor for 10-15 minutes in clean water containing a few drops of commercial dish washing liquid.
2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the cleaning solution.
3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then rerinse with clean water.

If good pH and/or ORP response is still not restored, perform the following additional procedure:

1. Soak the sensor for 30-60 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most lab supply distributors. Be sure to follow the safety instructions included with the acid.
2. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water (not DI water), and then rerinse with clean water. To be certain that all traces of the acid are removed from the sensor crevices, soak the sensor in clean water for about an hour with occasional stirring.

If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

1. Soak the sensor for approximately 1 hour in a 1:1 dilution of commercially-available chlorine bleach.
2. Rinse the sensor with clean water and then soak for at least 1 hour in clean water with occasional stirring to remove residual bleach from the junction. (If possible, soak the sensor for a period of time longer than 1 hour in order to be certain that all traces of chlorine bleach are removed.) Then rerinse the sensor with clean water and retest.



**CAUTION:** When using a cotton swab, be careful NOT to wedge the swab between the guard and the glass sensor. If necessary, remove cotton from the swab tip, so that the cotton can reach all parts of the sensor tip without stress. You can also use a pipe cleaner for this operation if more convenient.



Dry the port and sensor connector with compressed air and apply a very thin coat of o-ring lubricant to all o-rings before reinstallation.

If this procedure is unsuccessful, as indicated by improper sensor performance, contact YSI Technical Support or the YSI authorized dealer where you purchased the instrument.

## SENSOR STORAGE

### SHORT TERM STORAGE

The instrument is supplied with a grey storage sleeve that slides over the probe guard. The sleeve is used for short-term storage (less than 2 weeks). Be sure to keep a small amount of moisture (clean tap water) on the sponge in the sleeve during storage. The moistened sponge in the sleeve provides a 100% water saturated air environment which is ideal for short-term sensor storage.

### LONG TERM STORAGE

The conductivity sensor should be stored long term in a dry state while the ISE sensor should be stored in solution. When storing for more than 30 days, place the ISE sensor in the storage bottle that was originally included with the sensor. This can be filled with buffer 4 solution. If you no longer have the storage bottle, simply place the sensor in a buffer 4 solution. Ensure the conductivity sensor is clean and dry.

Long Term Storage Temperature: -5 to 70°C (23 to 158°F) without pH  
0 to 30°C (32 to 86°F) with pH\*

\*Operating temperature range for pH sensor is -5 to 60°C (23 to 140°C).

## TROUBLESHOOTING

Symptom	Possible Solution
Instrument will not turn on, a battery symbol appears, or "Critical Shutdown" displays on the screen.	<ol style="list-style-type: none"> <li>1. Low battery voltage, replace batteries.</li> <li>2. Batteries installed incorrectly, check battery polarity.</li> <li>3. Return system for service.</li> </ol>
Temperature values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Sample temperature is less than -5° C or more than +55°C. Increase or decrease the sample temperature to bring within the allowable range.</li> <li>2. Contact YSI Tech Support.</li> </ol>

<i>Symptom</i>	<i>Possible Solution</i>
Instrument will not calibrate pH or ORP; instrument displays "Calibration Over", "Calibration Under", or "Unstable Reading" during calibration.	<ol style="list-style-type: none"> <li>1. Verify correct sensor type selection in the System Setup menu.</li> <li>2. Verify the calibration solution is accurate.</li> <li>3. If calibrating pH, make sure you are calibrating buffer 7 first.</li> <li>4. Clean the pH or ORP sensor.</li> <li>5. Contact YSI Tech Support.</li> </ol>
pH or ORP readings are inaccurate.	<ol style="list-style-type: none"> <li>1. Verify correct sensor type selection in the System Setup menu.</li> <li>2. Verify temperature readings are accurate.</li> <li>3. Recalibrate the pH or ORP sensor.</li> <li>4. Clean the pH or ORP sensor.</li> <li>5. Contact YSI Tech Support.</li> </ol>
pH values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Verify correct sensor type selection in the System Setup menu.</li> <li>2. Sample pH value is outside the measurement range of 0 to 14.</li> <li>3. Verify temperature readings are accurate.</li> <li>4. Recalibrate the pH sensor.</li> <li>5. Clean the pH sensor and recalibrate.</li> <li>6. Contact YSI Tech Support.</li> </ol>
ORP values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Verify correct sensor type selection in the System Setup menu.</li> <li>2. Sample ORP value is outside the measurement range of -1500 to 1500 mV.</li> <li>3. Verify temperature readings are accurate.</li> <li>4. Recalibrate the ORP sensor.</li> <li>5. Clean the ORP sensor and recalibrate.</li> <li>6. Contact YSI Tech Support.</li> </ol>

<i>Symptom</i>	<i>Possible Solution</i>
Instrument will not calibrate the Conductivity sensor; instrument displays "Calibration Over", "Calibration Under", or "Unstable Reading" during calibration.	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the calibration solution is above the two holes near the cable, see figure 8.</li> <li>3. Verify the calibration solution is not expired or contaminated. Try a new bottle of solution.</li> <li>4. Ensure you are entering in the correct value for the solution according to the measurement units. 1 mS = 1,000 uS.</li> <li>5. Allow sufficient stabilization time for conductivity and temperature AND wait at least 3 seconds before confirming a calibration.</li> <li>6. Contact YSI Tech Support.</li> </ol>
<i>Conductivity readings are inaccurate.</i>	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the sample is above the two holes near the cable, see figure 8.</li> <li>3. Verify calibration.</li> <li>4. Verify temperature readings are accurate.</li> <li>5. Verify the correct units are setup in the System Setup menu, i.e. uS vs mS and Conductivity vs. Specific Conductance.</li> <li>6. Contact YSI Tech Support.</li> </ol>
Conductivity values display Over or Undr on Run screen.	<ol style="list-style-type: none"> <li>1. Ensure the conductivity sensor is clean. Follow the cleaning procedures in the Care, Maintenance and Storage section of this manual.</li> <li>2. Verify the sample is above the two holes near the cable, see figure 8</li> <li>3. Verify calibration.</li> <li>4. Verify temperature readings are accurate.</li> <li>5. Sample conductivity is outside the measurement range of the instrument, i.e. 0-200 mS.</li> <li>6. Contact YSI Tech Support.</li> </ol>

## SPECIFICATIONS

These specifications represent typical performance and are subject to change without notice. For the latest product specification information, please visit YSI's website at [ysi.com](http://ysi.com) or contact YSI Tech Support.

<i>Parameter</i>	<i>Range</i>	<i>Resolution</i>	<i>Accuracy</i>
<i>Temperature</i>	-5 to 55°C	0.1°C	± 0.2°C
<i>pH</i>	0 to 14 pH units	0.01	Instrument with cable and sensor: +/- 0.2
<i>ORP</i>	-1500 to 1500 mV	1 mV	Instrument with cable and sensor: +/-20 mV
<i>Conductivity</i>	0-500 uS/cm 0-5 mS/cm 0-50 mS/cm 0-200 mS/ cm (auto ranging)	0.0001 to 0.1 mS/cm; 0.1 to 0 uS/ cm (range dependent)	Instrument only: ± 0.5% of the reading or 1 uS/ cm, whichever is greater. Instrument with 1 or 4 meter cables: ± 1.0% of the reading or 1 uS/cm, whichever is greater. Instrument with 10, 20, or 30 meter cables: ± 2.0% of the reading or 1 uS/cm, whichever is greater.
<i>Salinity</i>	0 to 70 ppt	0.1 ppt	± 1.0% of the reading or ± 0.1 ppt, whichever is greater.
<i>Total Dissolved Solids (TDS)</i>	0 to 100 g/L. TDS Constant range: 0.3 to 1.00 (0.65 default)	0.0001 to 0.1 g/L (range dependent)	Dependent on accuracy of temperature, conductivity and TDS Constant.

## ACCESSORIES / PART NUMBERS

<i>Part Number</i>	<i>Description</i>
6051030	Pro1030 Instrument
6261030-1, -4, -10, -20, or -30	1, 4, 10, 20, 30-meter cable assembly* (3.2, 13, 32.8, 65.6, 98.4-feet)
605101	pH Sensor
605102	ORP Sensor
603077	Flow cell
603056	Flow cell mounting spike
603075	Carrying case, soft-sided
603074	Carrying case, hard-sided
603069	Belt clip for clipping instrument onto belt
063517	Ultra clamp for instrument for clamping instrument to lab counter or other surface
063507	Tripod for instrument
603062	Cable management kit, included with all cables longer than 1 meter
605978	Cable weight, 4.9 oz, stackable
603070	Shoulder strap
038213	Soft bristle brush for cleaning conductivity cell
003821	pH 4 Buffer, box of 6 pints
003822	pH 7 Buffer, box of 6 pints
003823	pH 10 Buffer, box of 6 pints
603824	pH Buffer, assorted case, 2 pints each of buffer 4, 7 and 10
060907	Conductivity Calibration Solution, 1,000 µS/cm. 1 box of 8 pints.
060911	Conductivity Calibration Solution, 10,000 µS/cm. 1 box of 8 pints.
060660	Conductivity Calibration Solution, 50,000 µS/cm. 1 box of 8 pints.
065274	Conductivity Calibration Solution, 100,000 µS/ cm. 1 box of 8 pints.

\*All cables include a temperature and conductivity sensor. The pH or ORP sensor is sold separately.

## DECLARATION OF CONFORMITY

The undersigned hereby declares on behalf of the named manufacturer under our sole responsibility that the listed product conforms to the requirements for the listed European Council Directive(s) and carries the CE mark accordingly.

<i>Manufacturer:</i>	YSI Incorporated 1725 Brannum Lane Yellow Springs, OH 45387 USA
<i>Product Name:</i>	Pro1030 Water Quality Instrument
<i>Model Numbers</i>	
<i>Instrument/ Accessory:</i>	Pro1030 (6051030)
<i>Probe/Cable Assemblies:</i>	6051030-1, -4, -10, -20, and -30
<i>Conforms to the following:</i>	
<i>Directives:</i>	EMC 2004/108/EC RoHS 2011/65/EU WEEE 2002/96/EC
<i>Harmonized Standards:</i>	<ul style="list-style-type: none"> <li>• EN61326-1:2006 (IEC 61326-1:2005)</li> <li>• IEC 61000-3-2:2005</li> <li>• IEC 61000-3-3:2005</li> </ul>
<i>Supplementary Information:</i>	All performance met the operation criteria as follows: 1. ESD, IEC 61000-4-2:2001 2. Radiated Immunity, IEC 61000-4-3:2006 3. Electrical Fast Transient (EFT), IEC 61000-4-4:2004, +Corr. 1:2006 + Corr. 2:2007 4. Radio Frequency, Continuous Conducted Immunity, IEC61000-4-6:2006 5. IEC 6100-4-8:2001
<i>Authorized EU Representative</i>	Xylem Analytics UK Ltd Unit 2 Focal Point, Lacerta Court, Works Road Letchworth, Hertfordshire, SG6 1FJ UK



Signed: Lisa M. Abel  
Title: Director of Quality

Date: 31 Jan 2013

## RECYCLING

YSI is committed to reducing the environmental footprint in the course of doing business. Even though materials reduction is the ultimate goal, we know there must be a concerted effort to responsibly deal with materials after they've served a long, productive life-cycle. YSI's recycling program ensures that old equipment is processed in an environmentally friendly way, reducing the amount of materials going to landfills.

- Printed Circuit Boards are sent to facilities that process and reclaim as much material for recycling as possible.
- Plastics enter a material recycling process and are not incinerated or sent to landfills.
- Batteries are removed and sent to battery recyclers for dedicated metals.

When the time comes for you to recycle, follow the easy steps outlined at [www.ysi.com](http://www.ysi.com).

### BATTERY DISPOSAL

The Pro1030 is powered by alkaline batteries which the user must remove and dispose of when the batteries no longer power the instrument. Disposal requirements vary by country and region, and users are expected to understand and follow the battery disposal requirements for their specific locale.

## CONTACT INFORMATION

### ORDERING AND TECHNICAL SUPPORT

Telephone: 800 897 4151 (USA)  
+1 937 767 7241 (Globally)  
Monday through Friday, 8:00 AM to 5:00 ET

Fax: +1 937 767 9353 (orders)  
+1 937 767 1058 (technical support)

Email: [environmental@ysi.com](mailto:environmental@ysi.com)

Mail: YSI Incorporated  
1725 Brannum Lane  
Yellow Springs, OH 45387 USA

Internet: [ysi.com](http://ysi.com)

When placing an order please have the following available:

- 1.) YSI account number (if available)
- 2.) Name and phone number
- 3.) Purchase Order or Credit Card number
- 4.) Model Number or brief description
- 5.) Billing and shipping addresses
- 6.) Quantity

## **SERVICE INFORMATION**

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YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit [ysi.com](http://ysi.com) and click 'Support' or contact YSI Technical Support directly at 800-897-4151 (+1 937-767-7241).

When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for a YSI Service Center to accept the instrument for service. The form may be downloaded from [ysi.com](http://ysi.com) by clicking on the 'Support'.

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