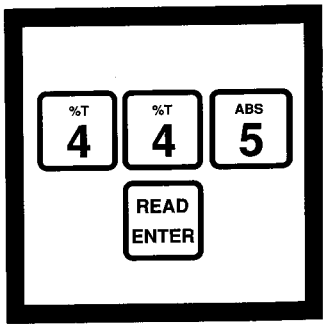
OXYGEN, DISSOLVED, HR (0 to 13.0 mg/L O_2)

HRDO Method



1. Enter the stored program number for dissolved oxygen.

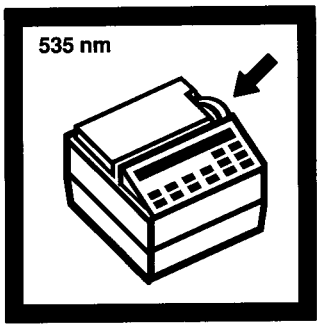
Press: **4 4 5 READ/ENTER**

The display will show:
DIAL nm TO 535

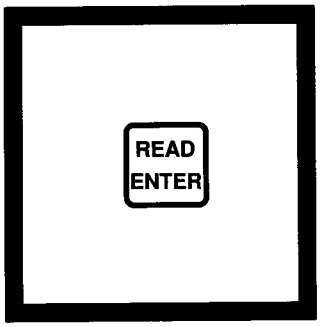
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *Samples must be analyzed on site and cannot be stored; see* Sampling and Storage *following these steps.*

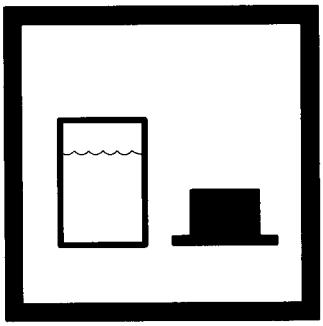


2. Rotate the wavelength dial until the small display shows:
535 nm

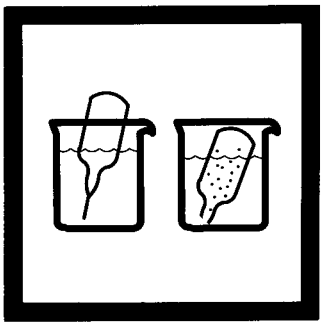


3. Press: **READ/ENTER**

The display will show:
mg/l O_2 HRDO

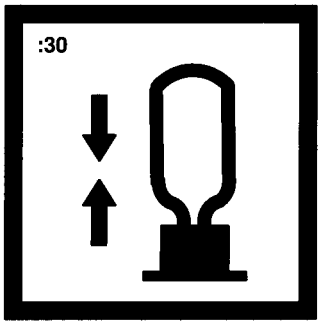


4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Fill a blue ampul cap with sample.



5. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

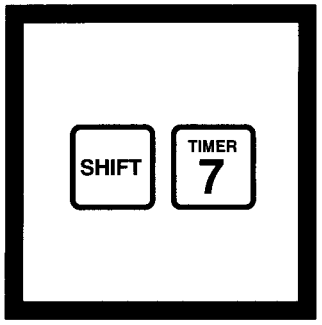
Note: *Keep the tip immersed while the ampul fills completely.*



6. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake the ampul for approximately 30 seconds.

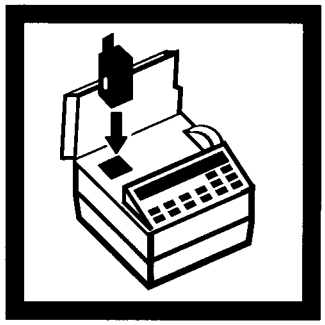
Note: *A small amount of the undissolved HRDO Reagent does not affect results.*

Note: *The cap prevents contamination with atmospheric oxygen.*



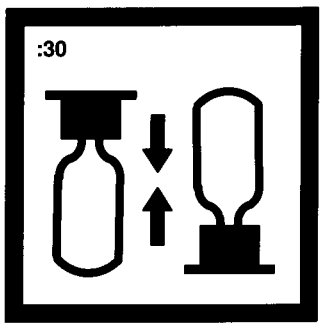
7. Press: **SHIFT TIMER**

A 2–minute reaction period enables oxygen, which was degassed during aspiration, to redissolve and react.

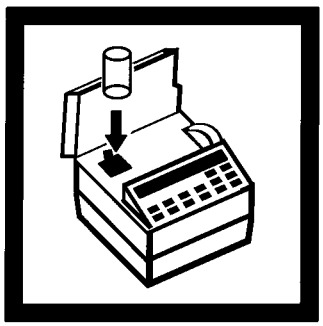


8. Place the AccuVac Vial Adapter into the cell holder.

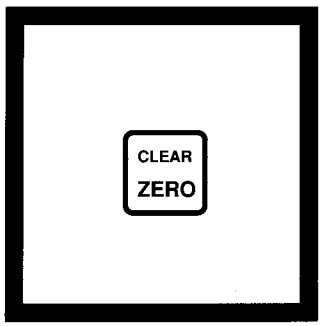
Note: *Place the grip tab at the rear of the cell holder.*



9. When the timer beeps, the display will show:
mg/l O_2 HRDO
Shake the ampul for 30 seconds.

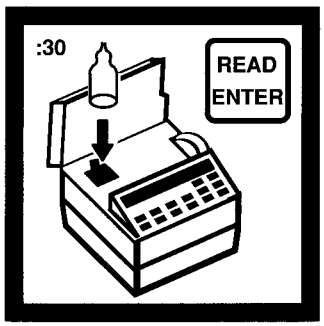


10. Place the blank into the cell holder. Close the light shield.



11. Press: **ZERO**

The display will show:
WAIT
then:
0.0 mg/l O_2 HRDO



12. Place the AccuVac ampul into the cell holder. Close the light shield. Wait approximately 30 seconds for the air bubbles to disperse from the light path.

Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L dissolved oxygen will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

SAMPLING AND STORAGE

The primary consideration in sampling with the High Range Dissolved Oxygen 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, the ampul 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.

ACCURACY CHECK

The results of this procedure may be compared with the results of a titrimetric procedure or dissolved oxygen meter.

PRECISION

In a single laboratory, using a standard solution of 7.22 mg/L O_2 determined by the Winkler method and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.20 mg/L O_2.

INTERFERENCES

The following do not interfere at a level of 10 mg/L which is in excess of naturally occurring levels: Cr^{3+}, Mn^{2+}, Fe^{2+}, Ni^{2+}, Cu^{2+} and NO_2^-.

SUMMARY OF METHOD

The High Range Dissolved Oxygen AccuVac ampul contains reagent that is vacuum sealed in a 12–mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
High Range Dissolved Oxygen AccuVac ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150–25

REQUIRED APPARATUS

AccuVac Dissolved Oxygen Sampler	1	each	24051–00
Adapter, AccuVac Vial	1	each	43784–00
Beaker, 50 mL	1	each	500–41
Caps, ampul, blue	varies	6/pkg	1731–06
Vial, zeroing	1	each	21228–00

OPTIONAL APPARATUS

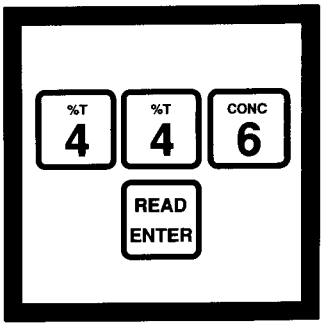
AccuVac Snapper Kit	each	24052–00
BOD bottle and stopper, 300 mL	each	621–00

Dissolved oxygen may also be determined by titrimetric methods. Request Publication 8042 for additional information.

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

OXYGEN, DISSOLVED, LR (0 to 800 µg/L O_2)

Indigo Carmine Method (Using AccuVac Ampuls)



1. Enter the stored program number for low range dissolved oxygen.

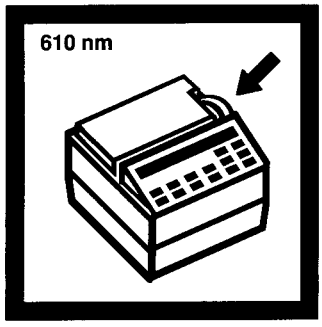
Press: **4 4 6 READ/ENTER**

The display will show:
DIAL nm TO 610

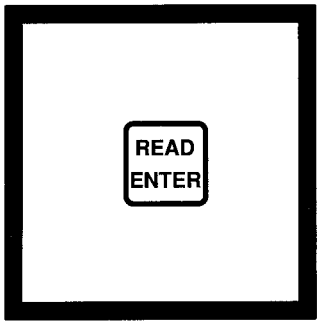
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *Samples must be analyzed on site and cannot be stored; see* Sampling and Storage *following this procedure.*



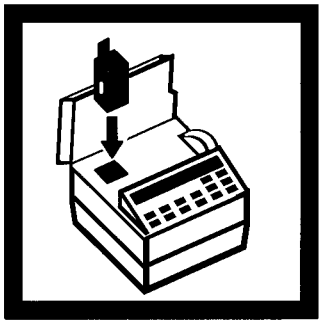
2. Rotate the wavelength dial until the small display shows:
610 nm



3. Press: **READ/ENTER**

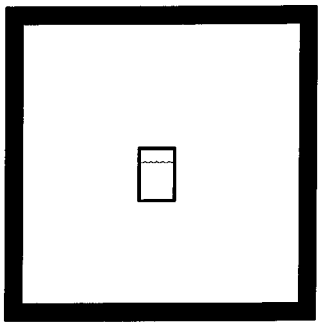
The display will show:
µg/l O2 LRDO AV

Note: *The ampuls will contain a small piece of wire to maintain reagent quality. The solution color will be yellow.*

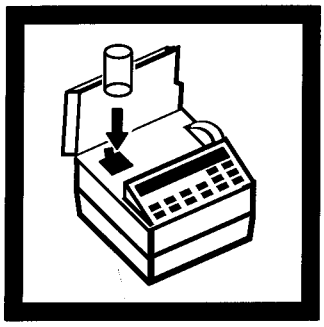


4. Place the AccuVac Vial Adapter into the cell holder.

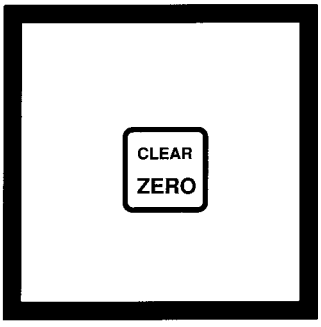
Note: *Place the grip tab at the rear of the cell holder.*



5. Fill a zeroing vial (the blank) with at least 10 mL of sample.

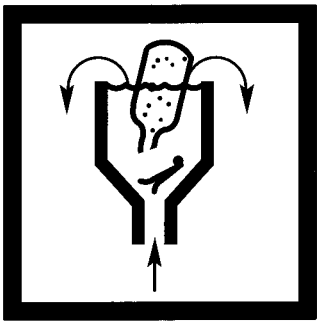


6. Place the blank into the cell holder. Close the light shield.



7. Press: **ZERO**

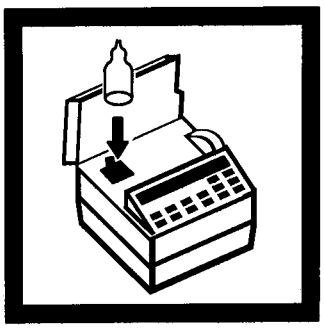
The display will show:
WAIT
then:
0. µg/l O2 LRDO AV



8. Fill a Low Range Dissolved Oxygen AccuVac Ampul with sample.

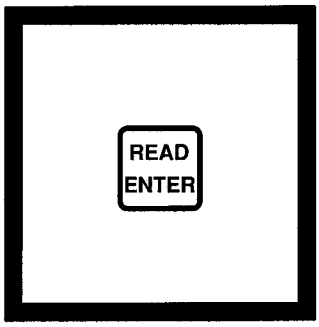
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *One measure of accuracy is to verify the zero concentration of the blank. Follow the steps given in the* Accuracy Check.



9. Immediately place the AccuVac ampul into the vial adapter. Close the light shield.

Note: Use the initial reading. The reading is stable for 30 seconds. After 30 seconds, the ampul solution will absorb oxygen from the air.



10. Press: **READ/ENTER**

The display will show:
WAIT
then the result in µg/L dissolved oxygen will be displayed.

Note: For a DR/2000 in the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

SAMPLING AND STORAGE

The primary 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. Aspirate the sample into the ampul.

c) Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0 ±1 µg/L.

INTERFERENCES

Excess amounts of sodium thioglycolate, sodium ascorbate, sodium ascorbate + sodium sulfite, sodium ascorbate + cupric sulfate, sodium nitrite, sodium sulfite, sodium thiosulfate, and hydroquinone will not reduce the oxidized form of the indicator solution and do not cause significant interference. A 100,000–fold excess of hydrazine will begin to reduce the oxidized form of the indicator solution.

Sodium hydrosulfite will reduce the oxidized form of the indicator solution and will cause a serious interference.

SUMMARY OF METHOD

The Low 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, the yellow solution will turn blue. The blue color development is proportional to the concentration of dissolved oxygen.

OXYGEN, DISSOLVED, LR, continued

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Low Range Dissolved Oxygen AccuVac Ampuls	1 ampul	25/pkg	25010–25

REQUIRED APPARATUS

Adapter, AccuVac Vial	1	each	43784–00
Vial, zeroing	1	each	21228–00

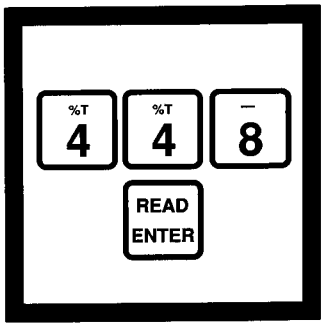
OPTIONAL REAGENTS AND APPARATUS

AccuVac Snapper Kit		each	34052–00
Beaker, low form, 50 mL		each	500–41
Sodium Hydrosulfite, technical grade		500 g	294–34

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

OXYGEN, DISSOLVED, SHR (0 to 45.0 mg/L O_2)

Super High Range Method



1. Enter the stored program number for dissolved oxygen.

Press: **4 4 8 READ/ENTER**

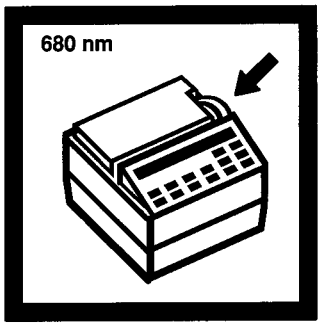
The display will show:
DIAL nm TO 680

Note: *Dr/2000s with software versions 3.0 and greater will display "P" and the program number.*

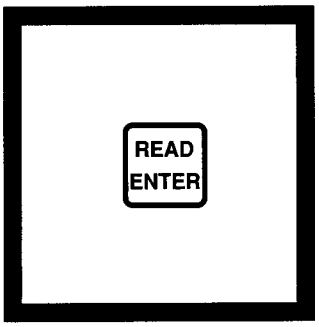
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *Samples must be analyzed on site and cannot be stored; see* Sampling and Storage *following these steps.*

Note: *For DR/2000s without this stored program, see* Instrument Setup *following these steps.*

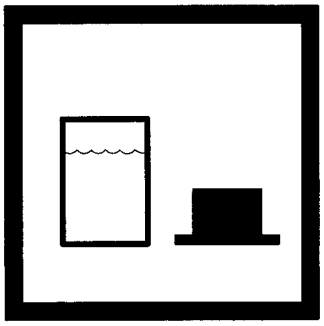


2. Rotate the wavelength dial until the small display shows:
680 nm

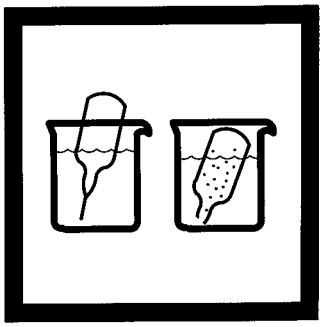


3. Press: **READ/ENTER**

The display will show:
mg/l O_2 SHRDO AV

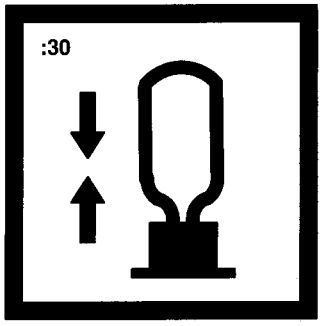


4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Fill a blue ampul cap with sample.



5. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

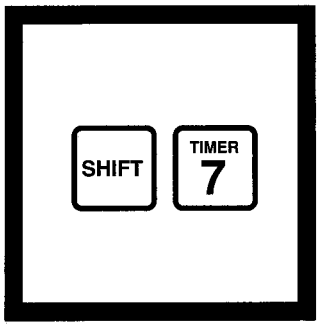
Note: Keep the tip immersed while the ampul fills completely.



6. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake the ampul for approximately 30 seconds.

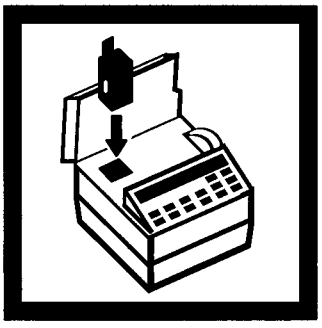
Note: A small amount of the undissolved reagent does not affect results.

Note: The cap prevents contamination with atmospheric oxygen.



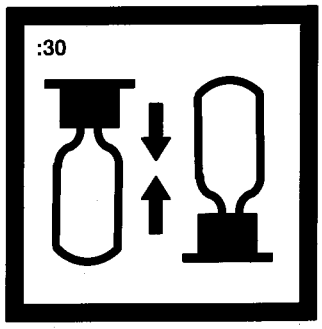
7. Press: **SHIFT TIMER**

A 2–minute reaction period enables oxygen, which was degassed during aspiration, to redissolve and react.

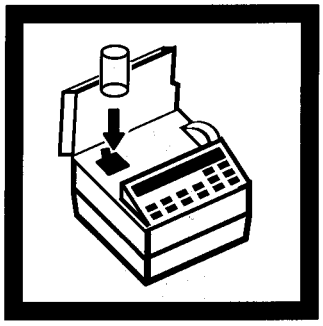


8. Place the AccuVac Vial Adapter into the cell holder.

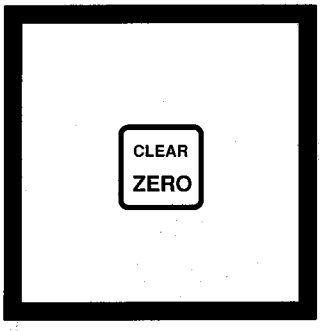
Note: Place the grip tab at the rear of the cell holder.



9. When the timer beeps, the display will show:
mg/l O2 SHRDO AV
Shake the ampul for 30 seconds.

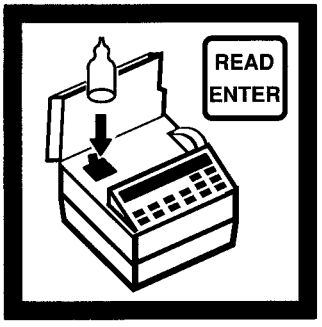


10. Place the blank into the cell holder. Close the light shield.



11. Press: **ZERO**

The display will show:
WAIT
then:
0.0 mg/l O_2 SHRDO AV



12. Place the AccuVac ampul into the cell holder. Close the light shield.

Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L dissolved oxygen will be displayed.

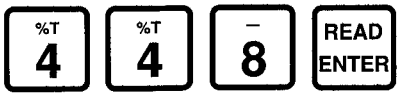
Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

INSTRUMENT SETUP

For a DR/2000 with software version 2.0 and 2.2 that do not have the Super High Range Dissolved Oxygen Method, Stored Method #448, enter the calibration as a Hach–entered program:

1. Press: 

2. Press: 

3. Press: 

4. Within 3 seconds press:

The display will show:

ENTER nm

If the display returns to the METHOD prompt, repeat the sequence.

5. Press: 

Note: If you make an error, press SHIFT CLEAR and re–enter the number. When the number is correct, press READ/ENTER. The display will show:

DECIMAL? 00.00

6. Use the arrow keys to correctly position the decimal point. For this method, press the **DOWN ARROW** key once. The display will show:

DECIMAL? 000.0

7. When the decimal point is correctly positioned, press **READ/ENTER.** The display will show:

UNITS?

8. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. The display will show:

mg/l

9. With the proper unit of measure displayed, press **READ/ENTER.** The display will show:

SYMBOL?

10. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key repeatedly until the display shows:

mg/l o

11. Press **SHIFT** to make the "o" uppercase. The display will show:

mg/l O

12. Press **READ/ENTER** to accept the capital O.

13. Press the 2 on the numeric key pad. The display will show:

mg/l O_2

14. Press the **READ/ENTER** key to accept the subscript 2.

15. Continue to construct the rest of the display:

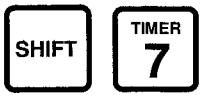
mg/l O_2 SHRDO AV

The space is the "character" displayed after one press of the **DOWN ARROW** key.

16. When the last character of the symbol is accepted by pressing the **READ/ENTER** key, the display will show:

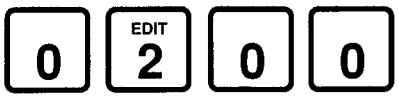
TIMER?

17. There is one timer period for this method. Press:



The display will show:

MM:SS TIME 1?

18. To enter the correct timer interval of 2:00 minutes, press: 

The display shows:

02:00 TIME 1?

19. Press **READ/ENTER** to accept the timer value. The display will show:

MM:SS TIME 2?

20. A second time interval is not needed. Press **READ/ENTER** to exit. The display will show:

 # 1 Data

21. Enter the following 12 numbers as shown. Complete each number with the **READ/ENTER** key.

# 1 Data	**0**
# 2 Data	**11053**
# 3 Data	**11308**
# 4 Data	**11308**
# 5 Data	**11309**
# 6 Data	**11308**
# 7 Data	**65535**
# 8 Data	**65535**
# 9 Data	**65535**
# 10 Data	**6553**
# 11 Data	**512**
CHECKSUM	**2188**

The final number is a check value which is used to determine if the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1, and the entire sequence must be re–entered. If all the numbers are entered correctly, the display will return to the method prompt and the instrument is ready to use.

SAMPLING AND STORAGE

The foremost 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, the ampul 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.

ACCURACY CHECK

The results of this procedure may be compared with the results of a titrimetric procedure or dissolved oxygen meter.

PRECISION

In a single laboratory, using a standard solution of 33.0 mg/L O_2 determined by the Winkler method and three representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±1.3 mg/L O_2.

INTERFERENCES

The following do not interfere at a level of 10 mg/L which is in excess of naturally occurring levels: Cr^{3+}, Mn^{2+}, Fe^{2+}, Ni^{2+}, Cu^{2+} and NO_2^-.

Magnesium is commonly present in seawater and interferes. If the seawater contains more than 50% salt water, the true oxygen concentration will decrease by 25%. If the seawater contains less than 50% salt water, the interference will be less than 5%.

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, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150–25

REQUIRED APPARATUS

Adapter, AccuVac Vial	1	each	43784–00
Beaker, 50 mL	1	each	500–41
Vial, zeroing	1	each	21228–00

OXYGEN, DISSOLVED, SHR, continued

OPTIONAL REAGENTS

AccuVac Snapper Kit	each	24052–00
AccuVac Dissolved Oxygen Sampler	each	24051–00
BOD Bottle and Stopper, 300 mL	each	621–00
Caps, ampul, blue	6/pkg	1731–06
DO Sampler, w/ 60 mL bottle, samples to 3 m (10 ft)	each	1962–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

OXYGEN DEMAND, CHEMICAL

For water, wastewater and seawater

Reactor Digestion Method*; USEPA approved for reporting wastewater analysis** (0-150 and 0-1500 ranges)

DIGESTION

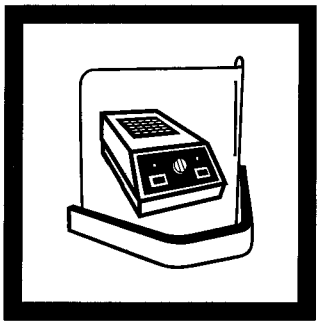
1. Homogenize 500 mL of sample for 2 minutes in a blender.

0 to 15,000 mg/L Note: *Homogenize 100 mL of sample. Pour the homogenized sample into a 250–mL beaker and stir with a magnetic stirrer.*

Note: *Blending ensures distribution of solids and improves accuracy and reproducibility.*

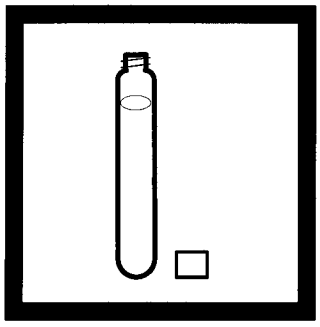
Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these procedures.*

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 accidently misused. Please read all warnings and the safety section of this manual. Appropriate eye protection and clothing should be used for adequate user protection. If contact occurs, flush the affected area with running water. Follow instructions carefully.*



2. Turn on the COD Reactor. Preheat to 150 °C. Place the plastic shield in front of the reactor.

Caution: *Ensure safety devices are in place to protect analyst from splattering should reagent leaking occur.*

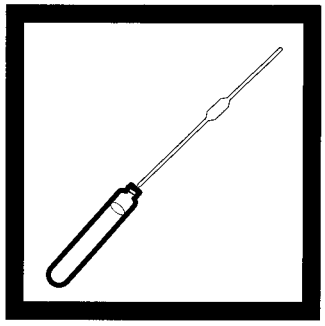


3. Remove the cap of a COD Digestion Reagent Vial for the appropriate range:

Sample Concentration Range (mg/L)	COD Digestion Reagent Vial Type
0 to 150	Low Range
0 to 1,500	High Range
0 to 15,000	High Range Plus

Use the cap tool provided to loosen the High Range Plus vials caps.

Note: *The reagent mixture is light–sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The amount of 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.

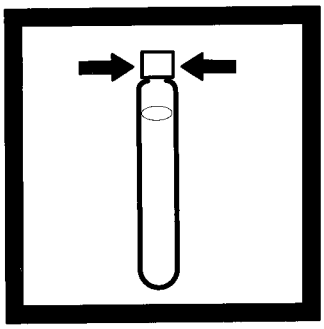
0 to 15,000 mg/L Note: *Pipet only 0.20 mL of sample, not 2.00 mL, using a TenSette Pipet. For greater accuracy a minimum of three replicates should be analyzed and the results averaged.*

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 contact occurs, wash with running water.*

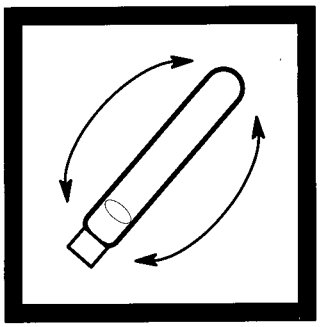
Note: *For proof of accuracy, use COD standard solutions (preparation given in the* Accuracy Check*) in place of the sample.*

*Jirka, A.M.; Carter, M.J. *Analytical Chemistry*, 1975, 47(8). 1397

**Federal Register*, April 21, 1980, 45(78), 26811–26812

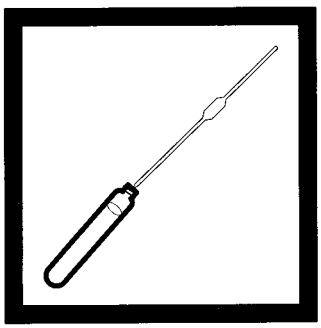


5. Replace the vial cap tightly. Use the cap tool provided, if necessary. Rinse the COD vial with demineralized water and wipe the vial clean with a paper towel.



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 COD Reactor.

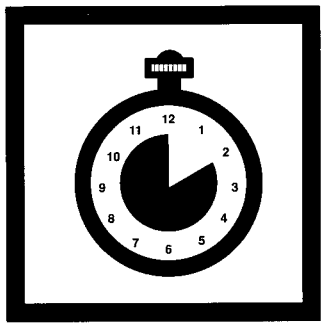
Note: The vial will become very hot during mixing.



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) demineralized water for the sample.

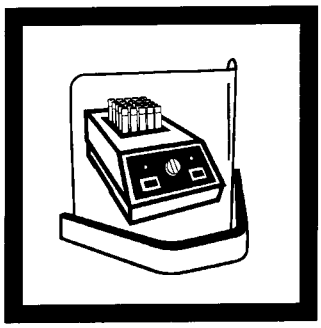
Note: Be sure the pipet is well rinsed, or use a clean pipet.

Note: One blank must be run with each set of samples. All tests (samples and blank) should be run with the same lot of vials. The lot number appears on the container label.

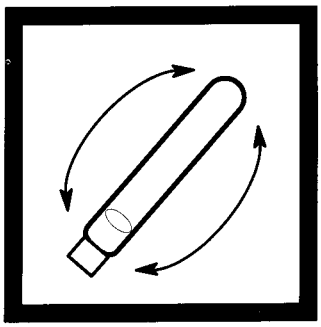


8. Heat the vials for 2 hours.

Note: Many wastewater samples containing easily oxidized materials are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until it remains unchanged. At this point, the sample is completely digested. Cool the vials to room temperature for final measurement.

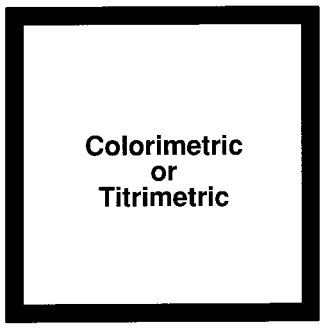


9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.



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, the reagent capacity may have been exceeded. Measure the COD and, if necessary, repeat the test with a diluted sample.

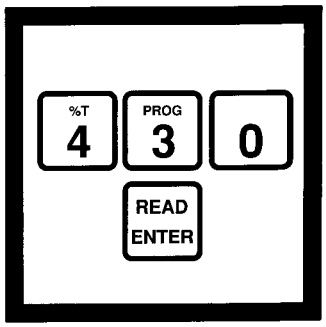


11. Use one of the following analytical techniques to determine the sample concentration:

Colorimetric determination, 0 to 150 mg/L COD
Colorimetric determination, 0 to 1,500 mg/L COD
Colorimetric determination, 0 to 15,000 mg/L COD
Buret titration

OXYGEN DEMAND, CHEMICAL, continued

COLORIMETRIC DETERMINATION, 0 to 150 mg/L COD



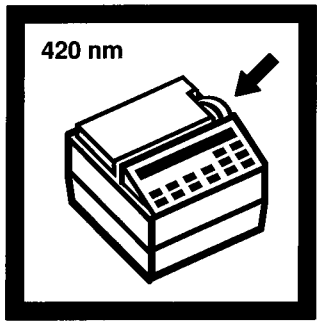
1. Enter the stored program number for chemical oxygen demand (COD), low range.

Press: **4 3 0 READ/ENTER**

The display will show:
DIAL nm TO 420

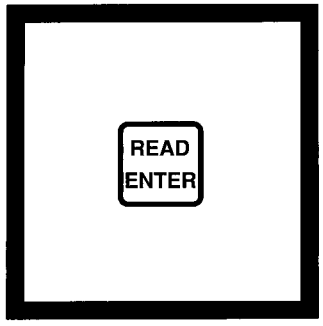
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



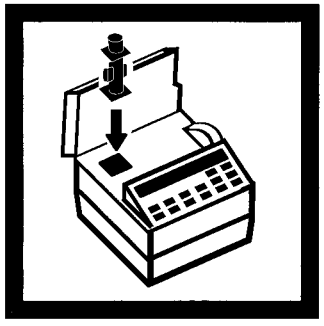
2. Rotate the wavelength dial until the small display shows:
420 nm

Note: *Approach the wavelength setting from the higher to lower values.*

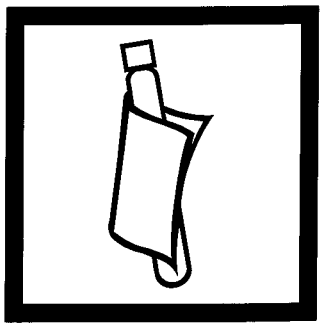


3. Press: **READ/ENTER**

The display will show:
mg/l COD L

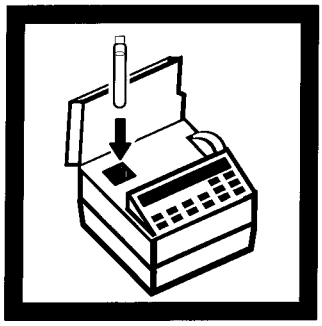


4. Place the COD Vial Adapter into the cell holder with the marker to the right.



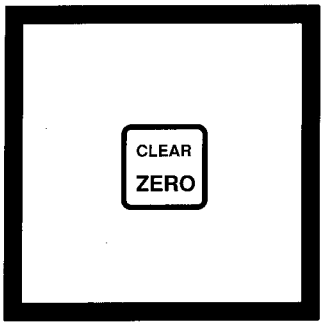
5. Clean the outside of the blank with a towel.

Note: *Wiping with a damp towel, followed by a dry one will remove fingerprints or other marks.*



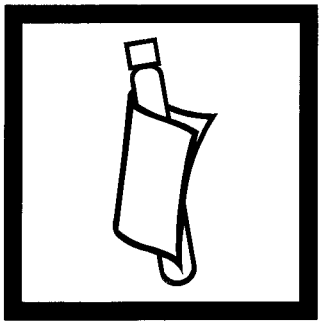
6. Place the blank into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.

Note: *The blank is stable when stored in the dark; see* Blanks for Colorimetric Determination *following these procedures.*

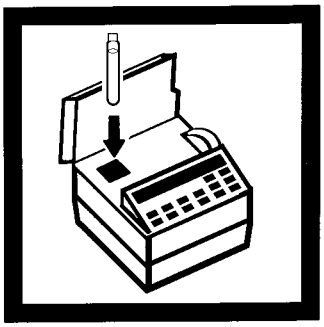


7. Press: **ZERO**

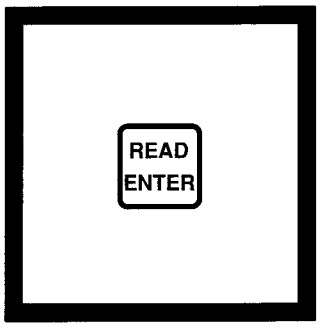
The display will show:
WAIT
then:
0. mg/l COD L



8. Clean the outside of the sample vial with a towel.



9. Place the sample vial into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



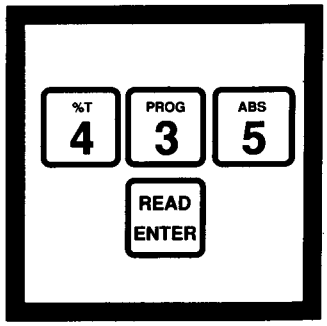
10. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L COD will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

Note: *For most accurate results with samples near 150 mg/L COD, repeat the analysis with a diluted sample.*

COLORIMETRIC DETERMINATION, 0 to 1,500 and 0 to 15,000 mg/L COD



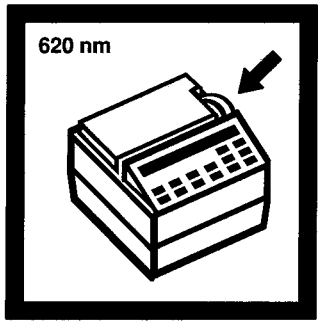
1. Enter the stored program number for chemical oxygen demand, high range.

Press: **4 3 5 READ/ENTER**

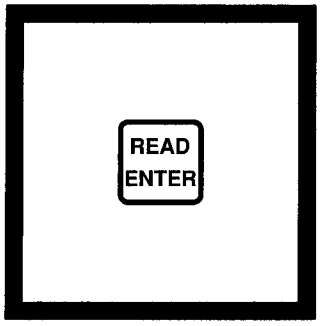
The display will show:
DIAL nm TO 620

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

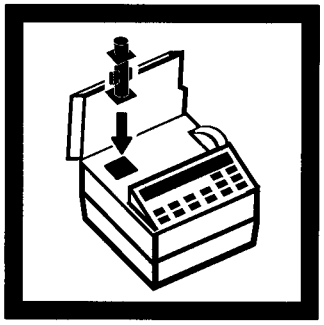


2. Rotate the wavelength dial until the small display shows:
620 nm

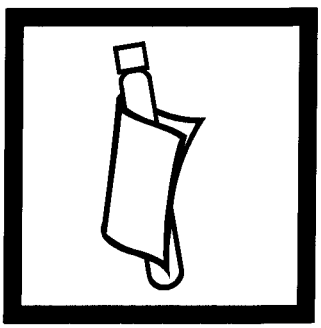


3. Press: **READ/ENTER**

The display will show:
mg/l COD H

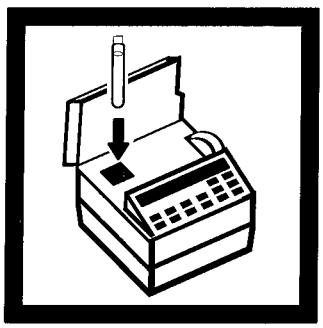


4. Place the COD Vial Adapter into the cell holder with the marker to the right.



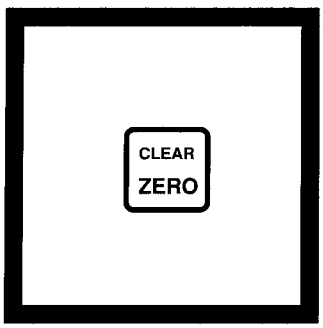
5. Clean the outside of the blank with a towel.

Note: *Wiping with a damp towel followed by a dry one will remove fingerprints or other marks.*



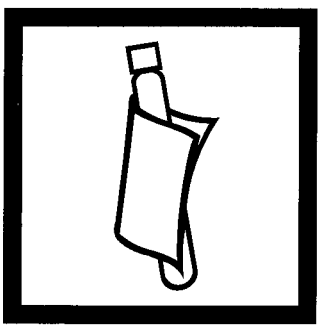
6. Place the blank into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.

Note: *The blank is stable when stored in the dark. See* Blanks for Colorimetric Determination *following these procedures.*

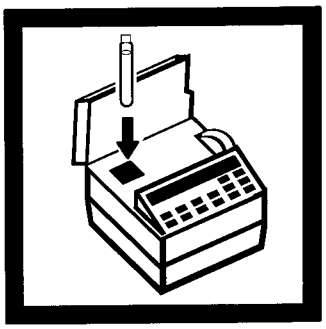


7. Press: **ZERO**

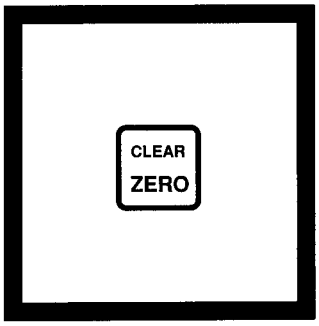
The display will show:
WAIT
then:
0. mg/l COD H



8. Clean the outside of the sample vial with a towel.



9. Place the sample vial in the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



10. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L COD will be displayed.

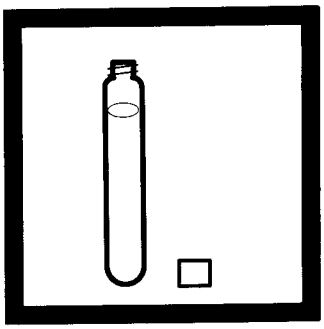
0 to 15,000 mg/L Note: *When High Range Plus COD Digestion Reagent Vials are used, multiply the displayed value by ten.*

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

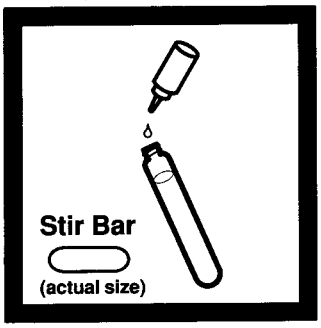
Note: *For most accurate results with samples near 1,500 or 15,000 mg/L COD, repeat the analysis with a diluted sample.*

OXYGEN DEMAND, CHEMICAL, continued

BURET TITRATION, 0 to 150, 0 to 1,500 and 0 to 15,000 mg/L COD

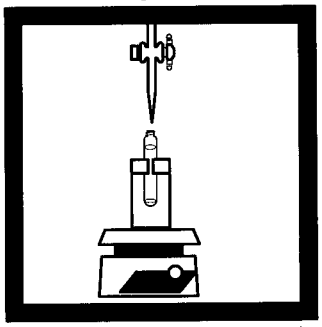


1. Carefully remove the cap of a vial. Rinse the inside walls with less than 1 mL of demineralized water.

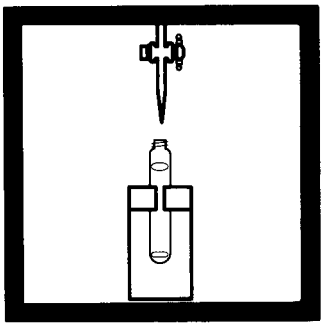


2. Add a small Teflon–coated stirring bar and one drop of the appropriate Ferroin Indicator Solution. When using the Low Range COD Digestion Reagent Vials, use Low Range Ferroin Indicator Solution. When using the High Range or High Range Plus COD Digestion Reagent Vials, use High Range Ferroin Indicator Solution.

Note: If the color of the prepared sample changes from blue–green to orange–brown, the COD value is out of range. Dilute the sample and repeat the digestion.



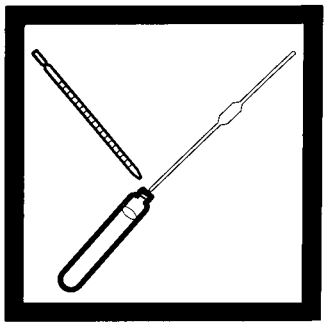
3. Place the vial on the titration stand. Turn on the magnetic stirrer.



4. Titrate with the appropriate Ferrous Ammonium Sulfate Standard Solution (FAS) until the sample color changes sharply from greenish–blue to orange–brown. When using the Low Range COD Digestion Reagent Vials, use 0.0125 N FAS. When using the High Range or High Range Plus COD Digestion Reagent Vials, use 0.125 N FAS. Record the mL of titrant required. The mL required for the prepared sample is value B. The mL required for the blank is value A.

Note: Mix the FAS solution well before using.

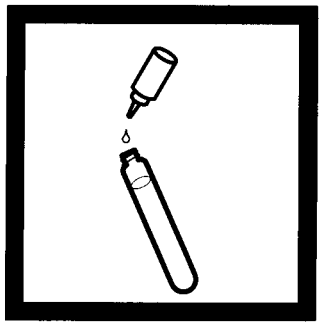
Note: Values A and B are used in Step 8.



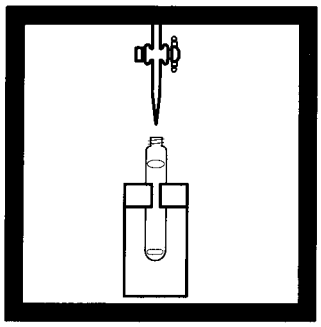
5. Pipet 2.00 mL of Potassium Dichromate Standard Solution into an empty vial. When using the Low Range COD Digestion Reagent Vials, use a 0.025 N solution. When using High Range or High Range Plus COD Digestion Reagent Vials, use a 0.25 N solution.

Add 3 mL of Sulfuric Acid to the vial. Swirl to mix. Wait for the solution to cool until the vial is comfortable to touch.

Note: Do Steps 5 through 7 daily because the FAS deteriorates over time.



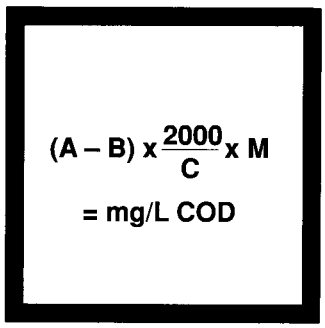
6. Add one drop of the Ferroin Indicator Solution selected in Step 2.



7. Add a stir bar and titrate with the Ferrous Ammonium Sulfate Standard Solution selected in Step 4 until the color changes from greenish–blue to orange–brown. Record the number of ml required. This is value C in the following equation.

Note: Mix the FAS solution well before using.

Note: To remove the stir bar from the vial, tip the vial at an angle in one hand and hold the stir bar retriever in the other. Place the retriever near the bottom of the vial on the OUTSIDE. Move the retriever up the wall to the top of the vial.



8. Determine the mg/L COD according to the following equation:

$$\text{COD mg/L} = (A - B) \times \frac{2000}{C} \times M$$

Where:
A = mL used in titration of reagent blank
B = mL used in titration of prepared sample
C = mL used in titration of standard solution in Step 7 above
M = 0.1 when using Low Range COD Digestion Reagent vials

1 when using High Range COD Digestion Reagent vials

10 when using High Range Plus COD Digestion Reagent vials

For example when using Low Range COD Reagent vials:
A = 3.95 mL
B = 2.00 mL
C = 4.00 mL
M = 0.1

$$\text{COD mg/L} = (3.95 - 2.00) \times \frac{2000}{4.00} \times 0.1$$
$$= 97.5$$

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 in Section I).*

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 demineralized water. Use 2 mL as the sample volume. The expected result will be 100 mg/L COD. Or, dilute 10 mL of 1000–mg/L COD Standard Solution to 100 mL to produce 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. Use 2 mL of one of these solutions as the sample volume; the expected result will be 300 or 1000 mg/L COD respectively. Or, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with demineralized water.

- 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 demineralized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

PRECISION FOR COLORIMETRIC DETERMINATION

In a single laboratory, using standard solutions of 100 mg/L COD and 500 mg/L COD and two lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±2.7 mg/L COD, ±18 mg/L COD and ±100 mg/L COD for 0 to 150, 0 to 1,500 and 0 to 15,000 mg/L ranges, respectively.

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 the following table. Dilute samples with higher chloride concentrations. Dilute the sample enough to reduce the chloride concentration to the level given in column 2.

Vial Type Used	(1) Maximum Cl^- concentration in sample (mg/L)	(2) Maximum Cl^- concentration of diluted samples (mg/L)	(3) Maximum Cl^- concentration in sample when 0.50 $HgSO_4$ added (mg/L)
Low Range	2000	1000	8000
High Range	2000	1000	4000
High RangePlus	20,000	10,000	40,000

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_4$) 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.

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 620 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of demineralized water and measure the absorbance of the blank. Record the value. Prepare a 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_2 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 ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0–150 mg/L colorimetric or titrimetric method is used, the amount of Cr^{6+} remaining is determined. When the 0–1,500 mg/L or 0–15,000 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences.

REQUIRED REAGENTS (For Colorimetric Analysis)

Description	Quantity Required Per Test	Units	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, demineralized	varies	4 L	272–56

REQUIRED REAGENTS (For Buret Titration)*

Select one or both Potassium Dichromate Standard Solutions:			
0.025 N	2 mL	500 ml	164–49
0.25 N	2 mL	1000 mL	1809–53
Sulfuric Acid, ACS	3 mL	500 mL**	979–49
Water, demineralized	varies	4 L	272–56
Select the appropriate COD Digestion Reagent Vial:			
Low Range	1 to 2 vials	25/pkg**	21258–25
High Range	1 to 2 vials	25/pkg**	21259–25
High Range Plus	1 to 2 vials	25/pkg**	24159–25
Select one or both Ferroin Indicator Solutions:			
Low Range	1 to 2 drops	29 mL	20551–33
High Range	1 to 2 drops	29 mL DB	1812–33
Select one or both Ferrous Ammonium Sulfate Standard Solutions*:**			
0.0125 N	varies	1000 mL	14237–53
0.125 N	varies	500 mL	20548–49

REQUIRED APPARATUS (For Colorimetric Analysis)

Cap Tool, COD	1	each	45587–00
COD Reactor, 120/240 Vac	1	each	45600–00
COD Vial Adapter, DR/2000	1	each	44799–00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700–01
Pipet, volumetric, Class A, 2 mL	1	each	14515–36
Pipet Filler, safety bulb	1	each	14651–00
Test Tube Rack	1 to 2 racks	each	18641–00

REQUIRED APPARATUS (For Buret Titration)

Bottle, wash, 500 mL	1	each	620–11
Buret clamp, double	1	each	328–00
Buret, automatic, Class A, 5.00 mL	1	each	20550–37
Cap Tool, COD	1	each	45537–00
COD Reactor, 120/240 Vac	1	each	45600–00
Pipet, volumetric, Class A, 2.00 mL	1	each	14515–36
Pipet, Mohr, 5.00 mL	1	each	20934–37
Pipet Filler, safety bulb	1	each	14651–00
Stir Bar	1	each	20549–59
Stir Bar Retriever	1	each	15232–00
Support Stand, 5 X 8"	1	each	563–00
Test Tube Rack, 8 place	1 (2 recommended)	each	18641–00
Titration Stand, test tube	1	each	18642–00
Select one based on available voltage:			
Stirrer, magnetic, 120 Vac, 50/60 Hz	1	each	23444–00
Stirrer, magnetic, 240 Vac, 50/60 Hz	1	each	23444–02

*Does not include reagents or apparatus for reagent blanks or standardization.
**Contact Hach for larger sizes.
***Ferrous ammonium sulfate standard solutions, as prepared by Hach, have a length of cadmium wire in each bottle. The cadmium wire will help preserve the standard solution. Before filling the buret, the bottle should be swirled to bring the upper layer of solution in contact with the wire. When titrating these solutions, do not return unused portions from the buret to the bottle or allow solution to stand in the buret for long periods of time. Do NOT use an automatic buret with a reservoir that holds more solution than can be used in one day.

OPTIONAL REAGENTS

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	236 mL	12186–31
COD Standard Solution, 1000 mg/L	236 mL	22539–31
Potassium Acid Phthalate, ACS	500 g	315–34
Sulfuric Acid, ACS	500 mL*	979–49
Mercuric Sulfate, ACS	28 g*	1915–20
Potassium Dichromate Standard Solution, 0.250 N	1000 mL*	1809–53

OPTIONAL APPARATUS

Beaker, 250 mL	each	500–46
Cylinder, graduated, 5 mL	each	508–37
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 Indicator Paper, 1 to 11 pH	5 rolls/pkg	391–33
Pipet, serological, 5 mL	each	532–37
Pipet Tips, for 19700–01 TenSette Pipet	50/pkg*	21856–96
Pipet, volumetric, Class A, 10 mL	each	14515–38
Safety shield, for COD reactor	each	23810–00
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

RELATED LITERATURE – Ask for your copy by literature code number.

Title	Literature Code No.
COD Disposal Information Brochure	4144

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

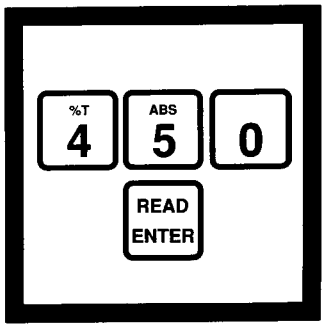
*Contact Hach for larger sizes.

TenSette is a Hach Company trademark.

OZONE (0 to 1.40 mg/L)

DPD Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



1. Enter the stored program number for ozone (O_3)–powder pillows.

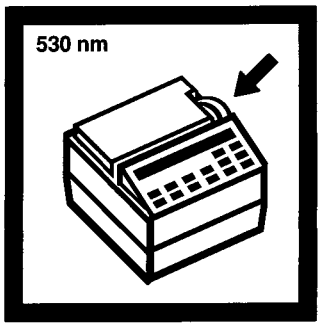
Press: **4 5 0 READ/ENTER**

The display will show:
DIAL nm TO 530

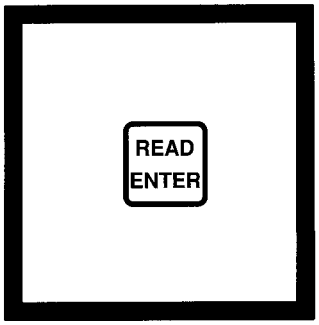
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

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

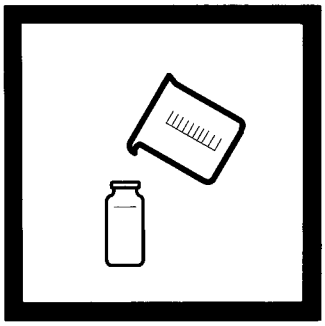


2. Rotate the wavelength dial until the small display shows:
530 nm



3. Press: **READ/ENTER**

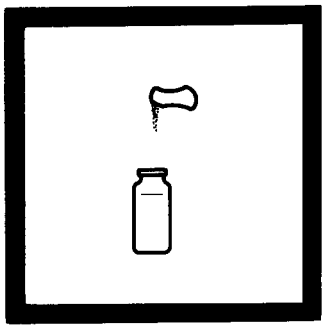
The display will show:
mg/l O_3 DPD



4. Fill a sample cell with 25 mL of sample.

Note: *The pour–Thru Cell can be used with this procedure if it is rinsed shortly after each analysis with demineralized water.*

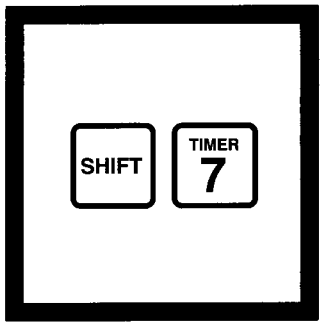
*Adapted from Palin, A.T., *J. Inst. Water Eng.*, 21 (6) 537–547 (1967)



5. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

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

Note: *Accuracy is not affected by undissolved powder.*



6. Press: **SHIFT TIMER**

A 3–minute reaction period will begin.

7. When the timer beeps, the display will show:

mg/l O_3 DPD

Fill a second sample cell (the blank) with 25 mL of sample. Place it into the cell holder. Close the light shield.

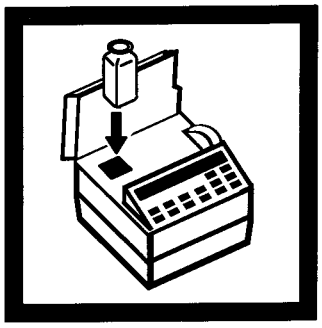
8. Press: **ZERO**

The display will show:

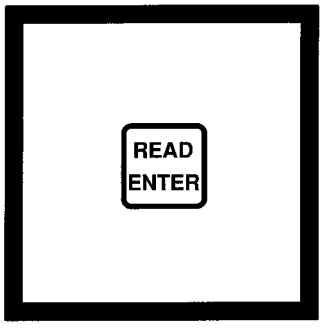
WAIT

then:

0.00 mg/l O_3 DPD



9. Within three minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.



10. Press: **READ/ENTER**

The display will show:

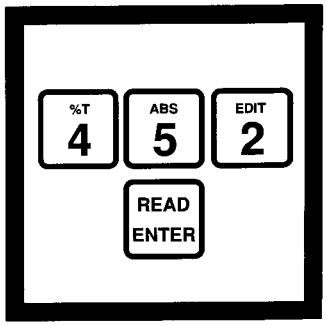
WAIT

then the result in mg/L O_3 will be displayed.

Note: *If the sample temporarily turns yellow after sample addition, or shows OVER–RANGE, dilute a fresh sample and repeat the test. A slight loss of ozone may occur because of the dilution. Multiply the the results by the appropriate dilution factor.*

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

USING ACCUVAC AMPULS



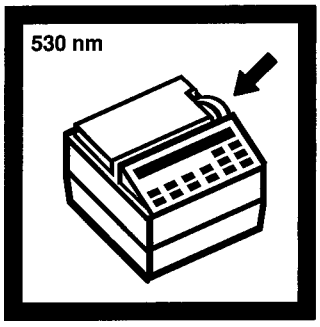
1. Enter the stored program number for ozone (O_3)–AccuVac ampuls.

Press: **4 5 2 READ/ENTER**

The display will show:
DIAL nm TO 530

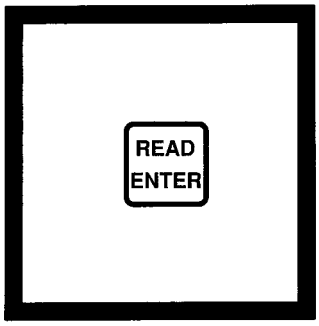
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



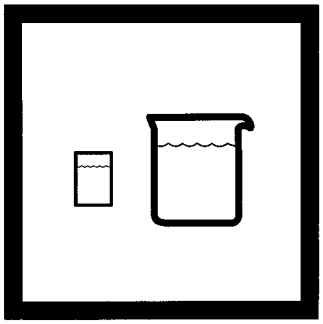
2. Rotate the wavelength dial until the small display shows:
530 nm

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

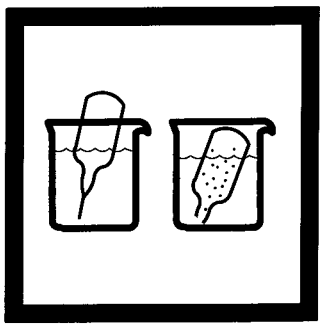


3. Press: **READ/ENTER**

The display will show:
mg/l O_3 DPD AV

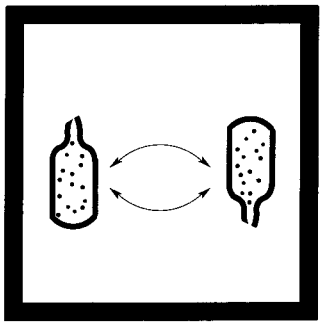


4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50–mL beaker.



5. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

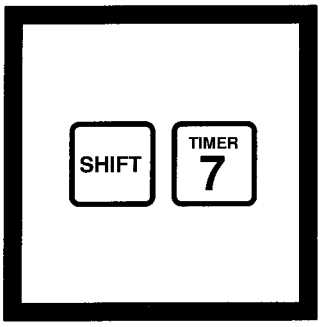
Note: *Keep the tip immersed while the ampul fills completely.*



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

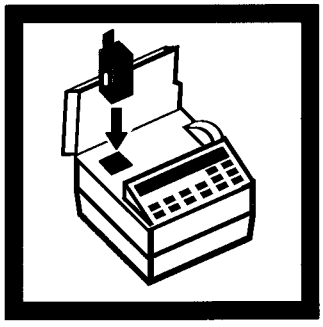
Note: *A pink color will form if ozone is present.*

Note: *Accuracy is unaffected by undissolved powder.*



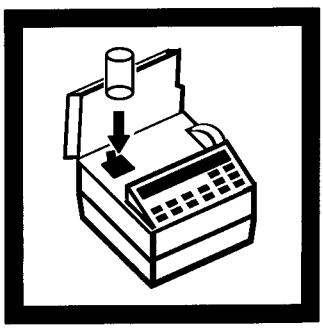
7. Press: **SHIFT TIMER**

A 3–minute reaction period will begin.

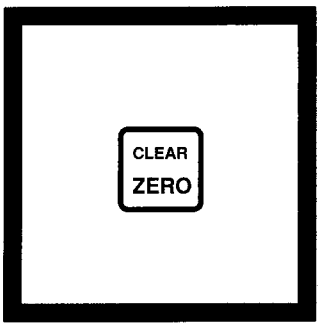


8. Place the AccuVac Vial Adapter into the cell holder of the instrument.

Note: *Place the grip tab at the rear of the cell holder.*

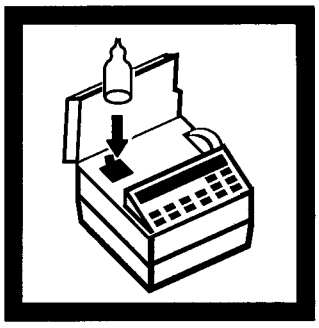


9. When the timer beeps, the display will show:
mg/l O_3 DPD AV
Place the blank into the cell holder. Close the light shield.

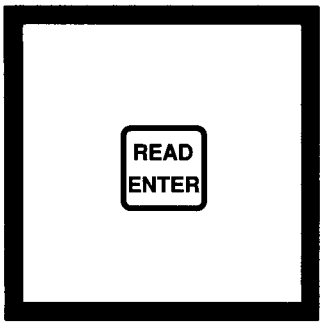


10. Press: **ZERO**

The display will show:
WAIT
then:
0.00 mg/l O_3 DPD AV



11. Within three minutes after the timer beeps, place the AccuVac ampul into the instrument cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L O_3 will be displayed.

Note: *If the sample temporarily turns yellow after sample addition, or shows OVER–RANGE, dilute a fresh sample. Repeat the test. A slight loss of ozone may occur because of the dilution. Multiply the result by the appropriate dilution factor.*

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

ACCURACY CHECK

Standard Additions Method

a) Snap the top off a Chlorine Voluette Ampule Standard Solution.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25–mL water samples. Swirl gently to mix. (For AccuVac ampuls, use 50–mL beakers.)

c) Analyze each sample as described above. Each 0.1–mL addition of standard should cause an equal increase in the ozone determination. Check the certificate enclosed with the Voluettes for the incremental chlorine value, then divide by 1.45 to obtain the value for ozone.

d) If these increases do not occur, see Standard Additions in Section I for more information.

INTERFERENCES

Samples containing more than 300 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$ may not develop the full amount of color, or it may instantly fade. Neutralize these samples to a pH of 6 to 7 with 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution. Determine the amount required on a separate 25–mL sample, then add the same amount to the sample to be tested. Correct the test result for volume additions *(see Correction for Volume Additions in Section I).*

Chlorine, bromine, iodine and oxidized forms of manganese and chromium also may react and show as ozone.

DPD Reagent Powder Pillows and AccuVac Ampuls contain a buffer system which will withstand high levels (greater than 1000 mg/L) of hardness without interference.

SUMMARY OF METHOD

Ozone reacts with DPD (N,N–diethyl–p–phenylenediamine) to form a red color which is proportional to the ozone concentration.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Units	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	14064–99

REQUIRED REAGENTS (Using AccuVac Ampuls)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030–25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968–00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac vial	1	each	43784–00
Beaker, 50 ml	1	each	500–41
Vial, zeroing	1	each	21228–00

OPTIONAL REAGENTS

Chlorine Standard Solution, Voluette ampule, 10 mL	16/pkg	14268–10
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045–32
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270–32
Water, demineralized	4 L	272–56

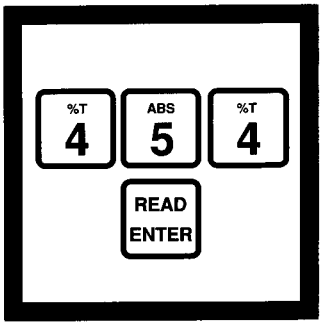
OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052–00
Cylinder, graduated, 25 mL, poly	each	1081–40
Graph Paper, linear	100/pkg	22313–00
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
Pour–Thru Cell Assembly Kit	each	45215–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

OZONE (0 to 0.25 mg/L O_3, 0 to 0.75 mg/L O_3 or 0 to 1.50 mg/L O_3)

Indigo Method (Using AccuVac Ampuls)



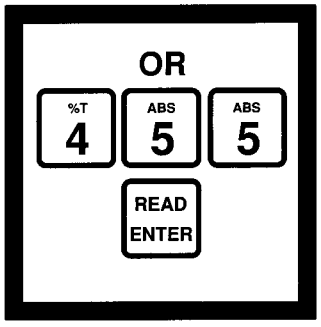
1. Enter the stored program number for ozone (O_3)–AccuVac ampuls.

Press: **4 5 4 READ/ENTER** for low range (0–0.25 mg/L)

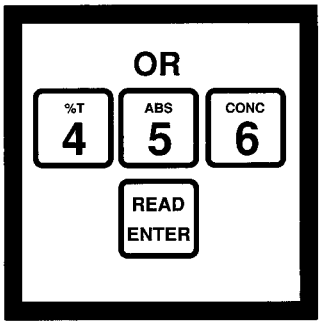
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

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

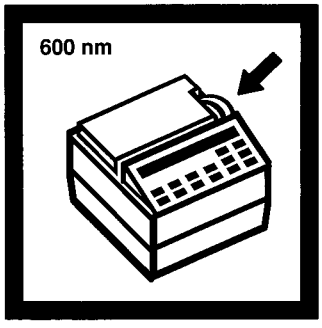


OR
Press: **4 5 5 READ/ENTER** for mid range (0–0.75 mg/L)

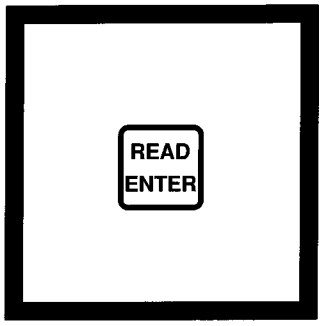


OR
Press: **4 5 6 READ/ENTER** for high range (0–1.50 mg/L)

The display will show:
DIAL nm TO 600

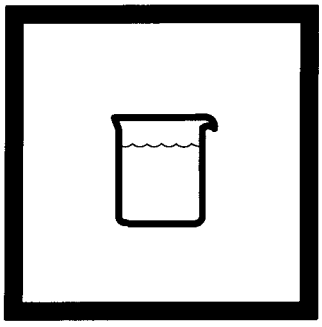


2. Rotate the wavelength dial until the small display shows:
600 nm

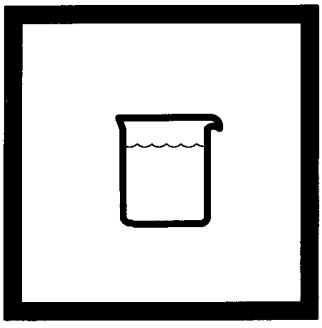


3. Press: **READ/ENTER**

The display will show:
for #454
mg/l O_3 Indigo L
for #455
mg/l O_3 Indigo M
for #456
mg/l O_3 Indigo H

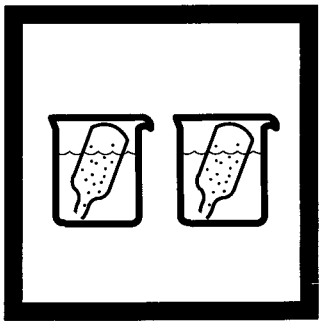


4. Gently collect at least 40 mL of sample in a 50–mL beaker.



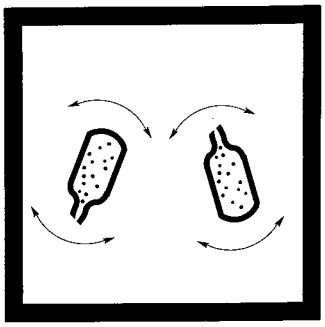
5. Collect at least 40 mL of ozone–free water (blank) in another 50–mL beaker.

Note: Ozone–free water used for the blank may be demineralized water or tap water.



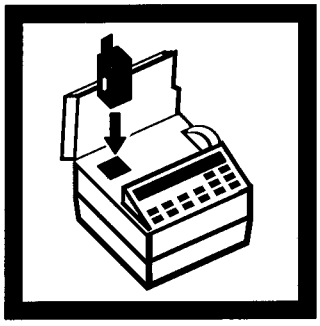
6. Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and one ampul with the blank.

Note: Keep the tip immersed while the ampul fills.



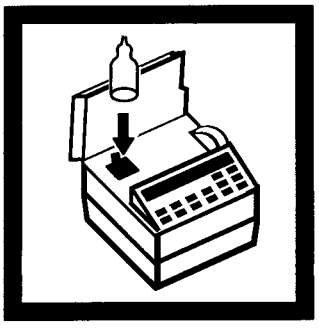
7. Quickly invert both ampuls several times to mix. Wipe off any liquid or fingerprints.

Note: Part of the blue color will be bleached if ozone is present.

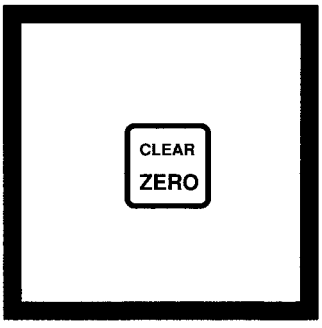


8. Place the AccuVac Vial Adapter into the cell holder.

Note: Place the grip tab at the rear of the cell holder.



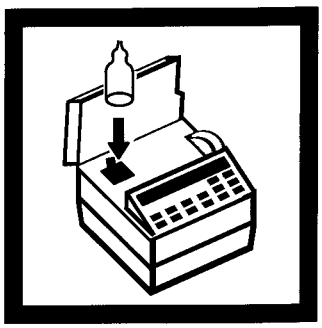
9. Place the **sample** AccuVac ampul into the cell holder. Close the light shield.



10. Press: **ZERO**

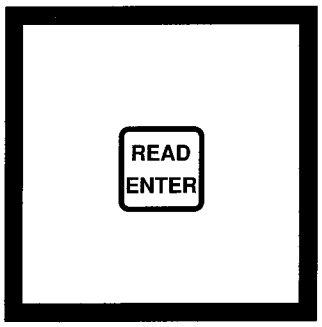
The display will show:
WAIT
then:
0.00 mg/l O_3

Note: Standardization for this procedure is intentionally reversed.



11. Place the AccuVac ampul containing the **blank** into the cell holder. Close the light shield.

Note: The DR/2000 will display a flashing decimal or a flashing number when no sample or a sample of lower absorbance than that used in Step 10 is in the cell holder. This may be accompanied by an OVER–RANGE indication and/or a beep. This is normal. The normal display will return when the blank ampul is used in this step.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L ozone (O_3) will be displayed.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

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.

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 Per Test	Units	Cat. No.
Select one or more based on range:			
Ozone AccuVac Ampuls			
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

REQUIRED APPARATUS

Adapter, AccuVac	1	each	43784–00
Beaker, 50 mL	2	each	500–41

OPTIONAL APPARATUS

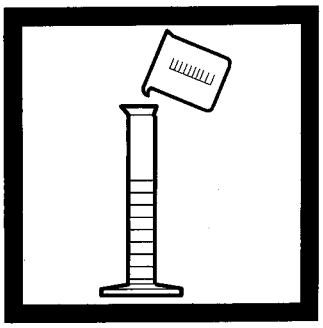
AccuVac Snapper Kit		each	24052–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

PHENOLS (0 to 0.200 mg/L)

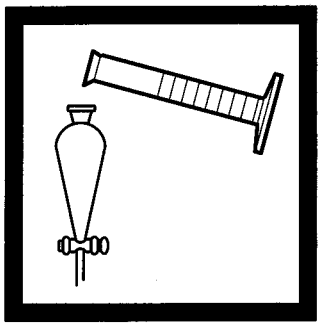
For water, wastewater and seawater

4–Aminoantipyrine Method*, USEPA accepted for reporting wastewater analysis (distillation required; *see Section 1*).**

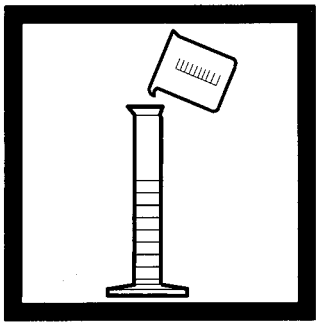


1. Measure 300 mL of demineralized water in a 500–mL graduated cylinder.

Note: *Analyze samples within four hours to avoid oxidation; see* Sampling and Storage *following these steps.*

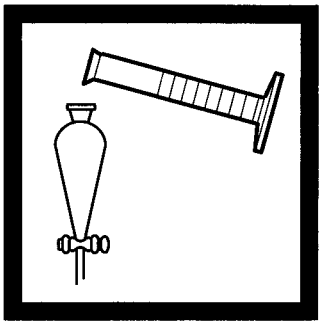


2. Pour the measured demineralized water into a 500–mL separatory funnel (the blank).

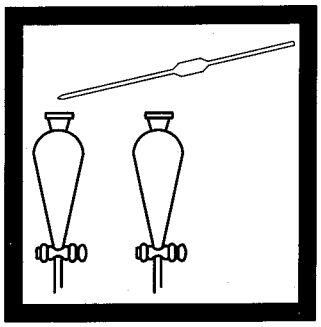


3. Measure 300 mL of sample in a 500–mL graduated cylinder.

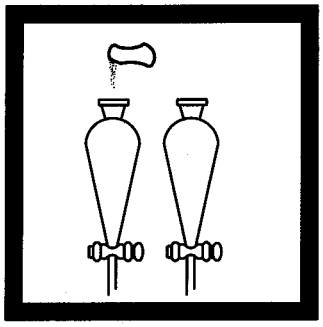
Note: *For proof of accuracy, use a phenol standard solution (preparations given in the* Accuracy Check*) in place of the sample.*



4. Pour the measured sample into another 500–mL separatory funnel (the prepared sample).

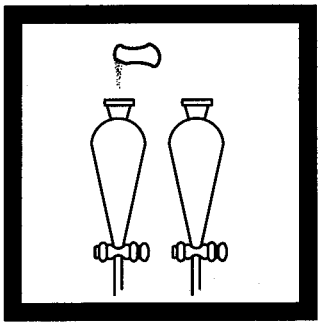


5. Add 5 mL of Hardness 1 Buffer to each separatory funnel. Stopper. Shake to mix.

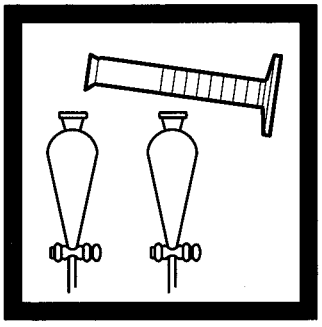


6. Add the contents of one Phenol Reagent Powder Pillow to each separatory funnel. Stopper. Shake to dissolve.

Note: *Spilled reagent affects test accuracy and is hazardous to skin and other materials.*



7. Add the contents of one Phenol 2 Reagent Powder Pillow to each separatory funnel. Stopper. Shake to dissolve.

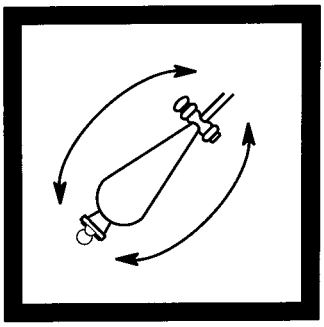


8. Add 30 mL of chloroform to each separatory funnel. Stopper each funnel.

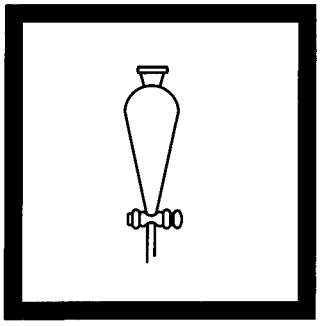
Caution: *Use chloroform only with proper ventilation.*

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

**Procedure is equivalent to USEPA method 420.1 for wastewater

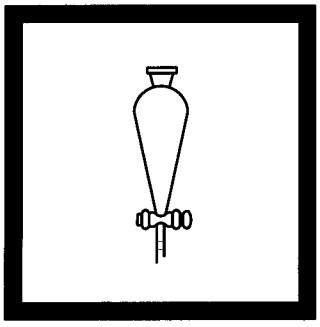


9. Invert each funnel and temporarily vent. Shake each funnel briefly and vent. Then vigorously shake each funnel for a total of 30 seconds.

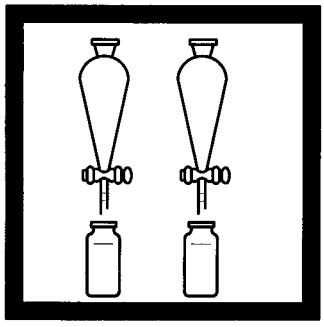


10. Remove the stoppers. Allow both funnels to stand until the chloroform settles to the bottom of the funnel.

Note: The chloroform will be yellow to amber if phenol is present.



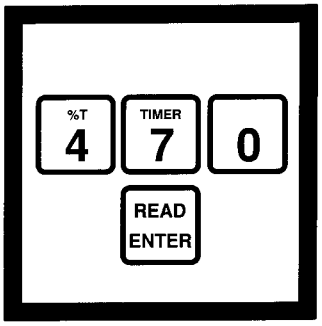
11. Insert a large (pea size) cotton plug into the delivery tube of each funnel.



12. Drain the chloroform layers into separate sample cells – one for the blank and one for the prepared sample.

Note: Filtering the chloroform layer through dry absorbent cotton removes any suspended water or particles. The volume of the chloroform extract will be about 25 mL due to slight solubility of chloroform in water.

Note: Proceed promptly through the rest of the procedure since the chloroform will evaporate, causing high readings.



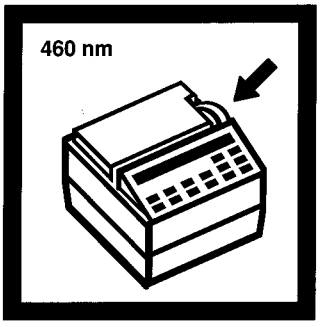
13. Enter the stored program number for phenols.

Press: **4 7 0 READ/ENTER**

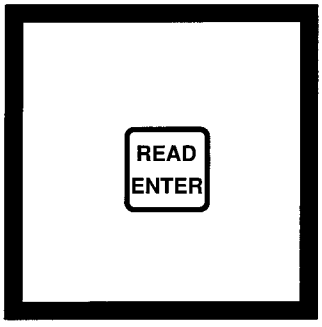
The display will show:
DIAL nm to 460

Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 15. Proceed with Step 16.

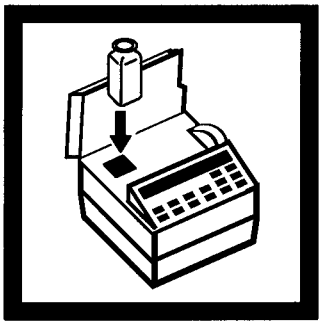


14. Rotate the wavelength dial until the small display shows:
460 nm



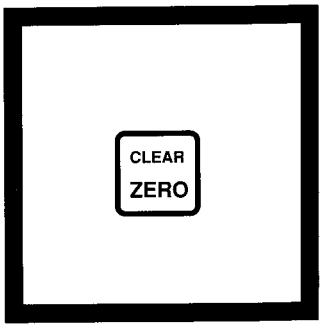
15. Press: **READ/ENTER**

The display will show:
mg/l PHENOL



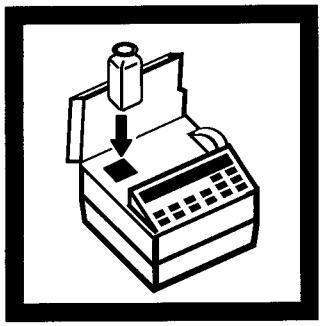
16. Place the blank into the cell holder. Close the light shield.

Note: The Pour-Thru Cell cannot be used with this procedure.

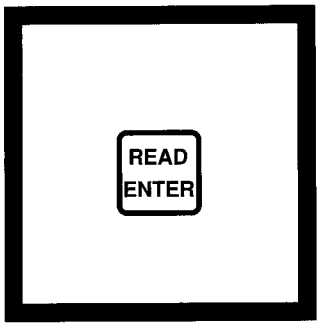


17. Press: **ZERO**

The display will show:
WAIT
then:
0.000 mg/l PHENOL



18. Place the prepared sample into the cell holder. Close the light shield.



19. Press: **READ/ENTER**

The display will show:
WAIT
then the results in mg/L as phenol will be displayed.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

SAMPLING AND STORAGE

Most reliable results are obtained when samples are analyzed within four hours after collection. The following storage instructions are necessary only when prompt analysis is impossible. Collect 500 mL of sample in clean glass containers and add the contents of two Copper Sulfate Powder Pillows. Adjust the sample pH to 4 or below with 10% Phosphoric Acid Solution. Store at 4 °C (39 °F) or lower and analyze within 24 hours.

ACCURACY CHECK

Standard Solution Method

Verify accuracy of the test by performing the analysis procedure using known phenol standard solutions in place of the test sample. For greatest accuracy, standard solutions should be analyzed periodically to verify test accuracy and when new reagent lots are first used. Prepare standards as follows:

a) Weigh out 1.00 g of phenol, ACS. Transfer to a 1–liter volumetric flask. Dilute to the mark with freshly boiled and cooled demineralized water. This is a 1–g/L stock solution.

b) Transfer 1.00 mL of the 1–g/L stock solution to a 100–mL volumetric flask. Dilute to the mark with demineralized water. This is a 10–mg/L working solution.

c) Prepare 0.06, 0.12 and 0.18 mg/L standard solutions by using a pipet to add 3, 6 and 9 mL of the 10–mg/L working solution, respectively, to three separate 500–mL volumetric flasks. Dilute each to the mark with demineralized water.

d) Perform the procedure with each of the three standard solutions and verify that the test results are correct.

PRECISION

In a single laboratory, using a standard solution of 0.08 mg/L phenol and two lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.0047 mg/L phenol.

INTERFERENCES

The sample pH must be between 3 and 11.5 for the best results. In the presence of sulfides or suspended matter, the following pretreatment will be necessary:

a) Take a water sample by filling a clean 500–mL graduated cylinder to the 350–mL mark. Pour the sample into a clean 500–mL erlenmeyer flask.

b) Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.

c) Filter 300 mL of the sample through a folded filter paper. Use this solution in Step 4.

Interference can be caused by reducing agents and oxidizing agents such as chlorine.

Sample distillation as described in the following steps

will eliminate interferences.

a) Set up the distillation apparatus for the test by assembling the general purpose apparatus as shown in the Hach Distillation Apparatus Manual. Use the 500–mL erlenmeyer flask to collect the distillate. It may be necessary to elevate the flask with a laboratory jack. Place a stirring bar into the distillation flask.

b) Measure 300 mL of sample in a 500–mL graduated cylinder. Pour it into the distillation flask.

c) Using a serological pipet, add 1 mL of Methyl Orange Indicator Solution to the distillation flask. Turn on the heater power switch. Set the stir control to 5.

d) Add Phosphoric Acid Solution, 100%, drop–wise, until the indicator changes from yellow to orange.

e) Add the contents of one Copper Sulfate Powder Pillow and allow to dissolve. (Omit this step if copper sulfate was used to preserve the sample.)

f) Turn on the water and adjust so a constant flow of water is maintained through the condenser. Set the heat control setting to 10.

g) Turn off the still after collecting 275 mL of distillate.

h) Fill a 25–mL graduated cylinder to the 25–mL mark with demineralized water. Turn the still back on. Add the water to the flask. Resume heating until another 25 mL of distillate is collected.

SUMMARY OF METHOD

The 4–aminoantipyrine method determines all ortho– and meta–substituted phenols or napthols but not parasubstituted phenols. These phenols react with 4–aminoantipyrine in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is then extracted from aqueous solution with chloroform and the color is measured at 460 nm. Sensitivity of the method varies with the type of phenolic compound. Because a water sample may contain various types of phenolic compounds, the results of the test are expressed as the equivalent concentration of phenol. Wastewater and seawater samples may require pretreatment.

REQUIRED REAGENTS

	Cat. No.
Phenols Reagent Ste (100 Tests)	22439–00
Includes: (3) 424–49, (2) 14458–17, (4) 1836–66, (8) 872–68	

Description	Quantity Required Per Test	Units	Cat. No.
Buffer Solution, Hardness 1	10 mL	500 mL	424–49
Chloroform, ACS	60 mL	4 L	14458–17
Phenol 2 Reagent Powder Pillow	2 pillows	50/pkg	1836–66
Phenol Reagent Powder Pillow	2 pillows	25/pkg	872–68
Water, demineralized	300 mL	4 L	272–56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968–00
Cotton Balls	2	100/pkg	2572–01
Cylinder, 50 mL graduated	1	each	508–41
Cylinder, 500 mL graduated	1	each	508–49
Funnel, 500 mL separatory	2	each	520–49
Pipet, volumetric, Class B, 5 mL	1	each	515–37
Ring, support, 4"	2	each	580–01
Stand, support, 5" x 8" base	1	each	563–00

OPTIONAL REAGENTS

Copper Sulfate Powder Pillows	50/pkg	14818–66
Methyl Orange Indicator Solution	500 mL	148–49
Phenol, ACS	113 g	758–14
Phosphoric Acid Solution, 10%	100 mL	14769–32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418–99

OPTIONAL APPARATUS

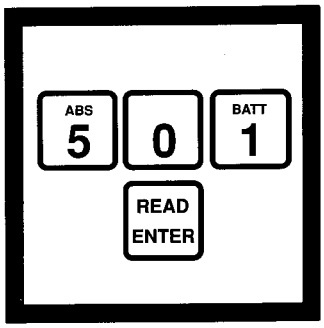
Cylinder, 25 mL, graduated	each	508–40
Distillation Apparatus General Purpose Accessories	each	22653–00
Distillation Apparatus Heater, 115 V	each	22744–00
Distillation Apparatus Heater, 230 V	each	22744–02
Filter Paper, 12.5 cm folded	100/pkg	1894–57
Flask, 500 mL erlenmeyer	each	505–49
Flask, volumetric, Class A, 100 mL	each	14574–42
Flask, volumetric, Class A, 500 mL	each	14574–49
Flask, volumetric, Class A, 1000 mL	each	14574–53
Funnel, 65 mm poly	each	1083–67
Jack, laboratory	each	22743–00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391–33
Pipet, serological, 1.0 mL	each	532–35
Pipet, volumetric, Class A, 1 mL	each	14515–35
Pipet, volumetric, Class A, 3 mL	each	14515–03
Pipet Filler, safety bulb	each	14651–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

PHOSPHONATES (0 – 2.5 to 0 – 125 mg/L)

For water, wastewater and seawater

Persulfate UV Oxidation Method*



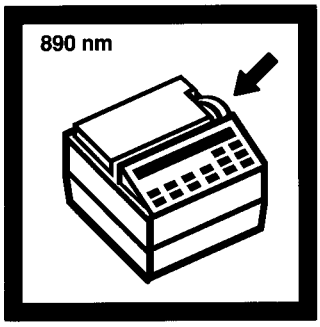
1. Enter the stored program number for phosphonates.

Press: **5 0 1 READ/ENTER**

The display will show:
DIAL nm TO 890

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

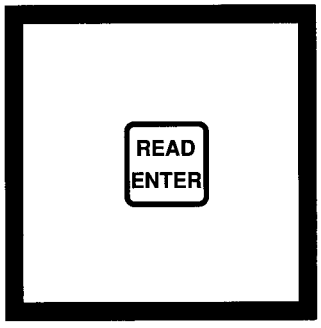
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



2. Rotate the wavelength dial until the small display shows:
890 nm

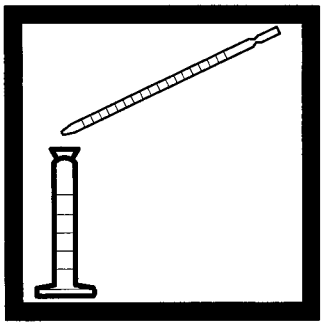
Note: *If sample cannot be analyzed immediately, see* Sampling and Storage *following these steps.*

Note: *For instruments with software versions that do not have stored program method 501, refer to* Instrument Setup.



3. Press: **READ/ENTER**

The display will show:
mg/l PHOSPHONATE

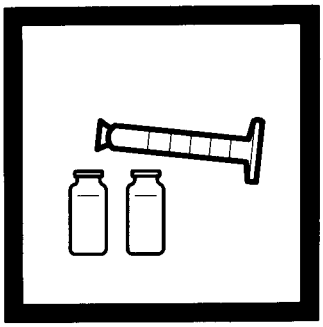


4. Choose a sample size from *Table 1* below. Pipet the chosen sample volume into a 50–mL graduated mixing cylinder. Dilute the sample to 50 mL with demineralized water. Mix well.

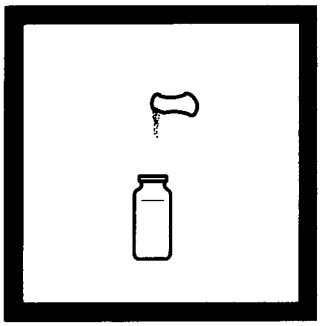
Table 1

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

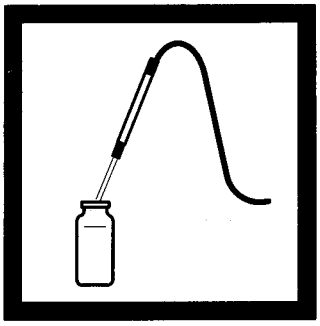
*Adapted from Blystone, P.; Larson, P., *A Rapid Method for Analysis of Phosphonate Compounds,* International Water Conference, Pittsburgh, Pa. (Oct. 26–28, 1981)



5. Split the diluted sample by pouring 25 mL into each of two sample cells.



6. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to one of the two sample cells. Swirl to mix. This sample cell contains the prepared sample.

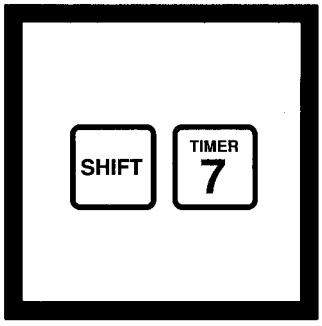


7. Insert the ultraviolet (UV) lamp into the prepared sample. the other sample cell is the blank.

Note: UV safety goggles should be worn 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 phosphate detergents to wash glassware.*

Note: *A specially designed cord adapter (Cat. No. 19485–00) is available for performing two digestions with a single power supply. A second UV Lamp (Cat. No. 20823–00) also is required.*



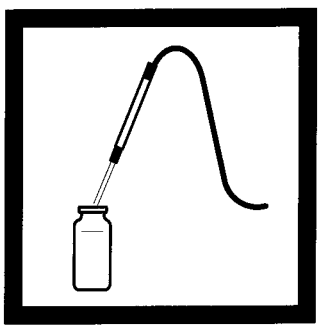
8. Turn on the UV lamp to digest the prepared sample.

Press: **SHIFT TIMER**

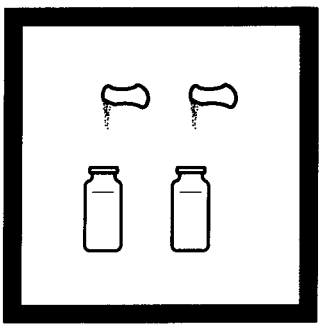
A 10–minute reaction period will begin.

Note: *Phosphonates are converted to orthophosphate in this step.*

Note: *The digestion step is normally completed in less than 10 minutes. However, contaminated sample or a weak lamp could result in incomplete conversion to phosphate. Conversion efficiency can be checked by running a longer digestion and seeing if readings increase.*

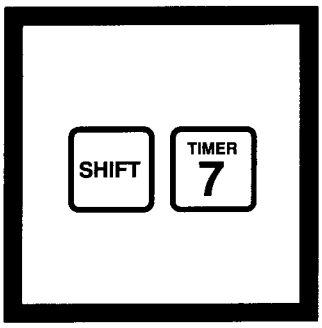


9. When the timer beeps, turn off the UV lamp. Remove it from the sample cell.



10. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to each sample cell. Swirl immediately to mix.

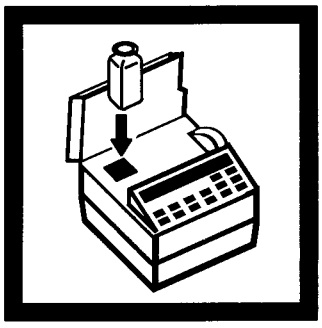
Note: *A blue color will form if phosphate (produced by the digestion) is present.*



11. Press: **SHIFT TIMER**

A 2–minute reaction period will begin.

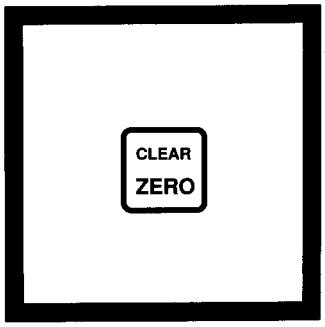
Note: *If sample is colder than 15 °C, 4 minutes are required for color development.*



12. When the timer beeps, the display will show: **mg/l PHOSPHONATE** Place the blank into the cell holder. Close the light shield.

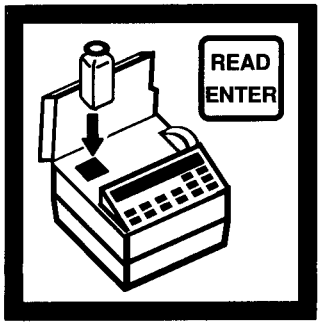
Note: *The Pour–Thru Cell can be used with this procedure.*

Note: *Perform Steps 13 and 14 within three minutes after the timer beeps.*



13. Press: **ZERO**

The display will show:
WAIT
then:
0.0 mg/l PHOSPHONATE

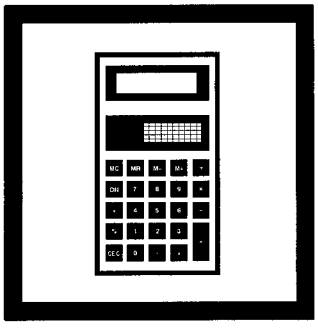


14. Place the prepared sample into the cell holder.

Press: **READ/ENTER**

The display will show:
WAIT
then a value in mg/L phosphonate as PO_4 will be displayed. Multiply this value by the appropriate multiplier from *Table 2* to obtain the concentration of the actual sample.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*



15. Results may be expressed in terms of active phosphonic acid by picking the appropriate conversion factor and using the following equation found in *Table 3*.

Table 3

Phosphonate Type	Conversion Factor
Bayhibit AM PBTC	2.84
Dequest 2000, Wayplex NTP, ATMP	1.050
Dequest 2010, Wayplex HEDPA-60, HEDP	1.085
Dequest 2041, EDTMPA	1.148
Dequest 2051, HMDTMPA	1.295
Dequest 2060, DETPMPA	1.207

Active Phosphonic Acid, mg/L = Phosphate Value from Step 14 x Conversion Factor

Table 2

Sample Volume (mL)	Multiplier
50	.1
25	.2
10	.5
5	1.
1	5.

mg/L Phosphonate = Instrument Reading x Multiplier

INSTRUMENT SETUP

For a DR/2000 with software versions before 2.0 that does not have Phosphonates, Persulfate UV Oxidation Method, Stored Program Method #501, enter the following calibration as an operator–programmed calibration. Follow the steps in the *Operation* section of the *DR/2000 Instrument Manual*. Store the method as follows:

nm = 890
Decimal = 000.0
Units = mg/l
Symbol = PHOSPHONATE
Timer 1 = 10:00
Timer 2 = 2:00

The calibration is first entered with 0.000 absorbance values for zero and #1 standard. To do this, leave the sample cell compartment empty, and begin by storing zero and #1 standard concentrations of 0 and 24.3, respectively. Accept 0.000 Abs as the value for both standards. Next, the values for the zero standard and #1 standard must be changed to the values given below.

Standard	Absorbance	Concentration
0	0.019	0
1	1.375	24.3

The method is now stored as an operator–programmed method with a method number between 950 and 999. Record the method number for future reference.

For a DR/2000 with software versions 2.0 and 2.2 that do not have the Phosphonates, Persulfate UV Oxidation Method, Stored method #501, enter the calibration as a Hach–entered program.

1. Press: [I/O]

2. Press: [SHIFT] [CONFIG METH]

3. Press: [ABS 5] [0] [BATT 1] [READ ENTER]

4. Within three seconds, press:

[SHIFT] [PROG 3] [CONFIG METH]

The display will show:

ENTER nm

If the display returns to the METHOD prompt, repeat the sequence.

5. Press: [− 8] [+ 9] [0]

Note: If an error is made, press SHIFT, CLEAR and re–enter the number. When the number is correct, press READ/ENTER.

The display will show:

DECIMAL? 00.00

6. Use the arrow keys to correctly position the decimal point. For this method, press the **RIGHT ARROW** key once. The display will read:

DECIMAL ? 000.0

7. With the decimal point correctly positioned, press **READ/ENTER.** The display will show:

UNITS?

8. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. the display will show:

mg/l

9. With the proper unit of measure displayed, press **READ/ENTER.** The display will read:

SYMBOL?

10. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key repeatedly until the display reads:

mg/l p

11. Press **SHIFT** to make the "p" upper case. The display will show:

mg/l P

12. Press **READ/ENTER** to accept the capital "P" Continue to construct the display to read:

mg/l PHOSPHONATE

13. When the last character of the symbol is accepted with the **READ/ENTER** key, the display will show:

TIMER?

There are two timers for this method. Press **SHIFT, TIMER.** The display will read:

MM:SS TIME 1?

14. To enter the first timer value of 10:00 minutes, press:

[BATT 1] [0] [0] [0]

The display will read:
10:00 TIME 1?

15. Press **READ/ENTER** to accept the value. The display will then read:
MM:SS TIME 2?

16. To enter the second timer value of 2:00 minutes, press:

The display will read:
02:00 TIME 2?

17. Press **READ/ENTER** to accept the timer value. The display will then read:
MM:SS TIME 3?

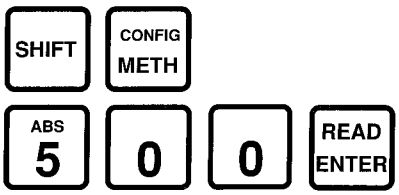
18. Press **READ/ENTER** to complete timer entry. The display will show:
 # 1 Data 0

19. Enter the following twelve numbers as shown. Complete each number with the **READ/ENTER** key.

# 1	**DATA**	**0**
# 2	**DATA**	**4886**
# 3	**DATA**	**5910**
# 4	**DATA**	**5655**
# 5	**DATA**	**5655**
# 6	**DATA**	**5654**
# 7	**DATA**	**5910**
# 8	**DATA**	**65535**
# 9	**DATA**	**65535**
# 10	**DATA**	**10922**
# 11	**DATA**	**512**
CHECKSUM		**20434**

The final number is a check value which is used to determine that the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1 and the entire sequence must be re–entered. If all numbers are correctly entered, the update procedure is complete and the display will return to the method prompt:
METHOD?

20. With a new method 501 successfully entered, block access to the now obsolete method 500. Press:



21. Within 3 seconds, press:

Access to method 500 is now blocked.

SAMPLING AND STORAGE

Collect sample in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized 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 the sample at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See *Section I* for more information on dilution factors, cleaning instructions, etc.

INTERFERENCES

When testing a 5–mL sample volume, the following may interfere when present in concentrations exceeding those listed below:

Aluminum	100 mg/L
Benzotriazole	10 mg/L
Bicarbonate	1000 mg/L
Bromide	100 mg/L
Calcium	5000 mg/L
CDTA	100 mg/L
Chloride	5000 mg/L
Chromate	100 mg/L
Copper	100 mg/L
Cyanide*	100 mg/L
Diethanoldithiocarbamate	50 mg/L
EDTA	100 mg/L
Iron	200 mg/L
Nitrate	200 mg/L
NTA	250 mg/L
Orthophosphate	15 mg/L
Silica	500 mg/L
Silicate	100 mg/L
Sulfate	2000 mg/L
Sulfite	100 mg/L
Thiourea	10 mg/L

*The UV digestion should be increased to 30 minutes.

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.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment *(see pH Interference in Section I)*. Arsenate and sulfide interfere directly. Phosphites and organophosphorous compounds other than phosphonates react quantitatively. Meta and polyphosphates do not interfere.

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.

REQUIRED REAGENTS

	Cat. No.
Phosphonates Reagent Set (100 Tests) Includes: (2) 2125–99, (1) 20847–69	22440–00

Description	Quantity Required Per Test	Units	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	2 pillows	100/pkg	2125–99
Potassium Persulfate Powder Pillow for Phosphonate	1 pillow	100/pkg	20847–69
Water, demineralized	varies	4 L	272–56

REQUIRED APPARATUS

Clippers, for opening pillows	1	each	20658–00
Cylinder, mixing, graduated, 50 mL	1	each	1896–41
Goggles, UV safety	1	each	21134–00
Pipet, volumetric, Class A, 5.00 mL	1	each	14515–37
UV Lamp with power supply, 115 Vac	1	each	20828–00
OR			
UV Lamp with power supply, 230 Vac	1	each	20828–02

OPTIONAL REAGENTS

Hydrochloric Acid, 6.0 N (1:1)	500 mL	884–49
Sulfuric Acid, ACS	500 mL	979–49

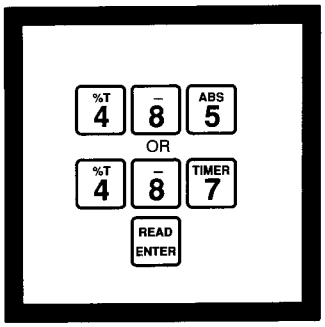
OPTIONAL APPARATUS

Cord Adapter, single-to-dual UV lamp	each	19485–00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391–33
Pipet, serological, 2 mL	each	532–36
Pipet Filler, safety bulb	each	14651–00
Pour–Thru Cell Assembly Kit	each	45215–00
UV Lamp	each	20823–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

PHOSPHORUS, REACTIVE (0 to 30.00 mg/L PO_4^{3-}) For water, wastewater and seawater

(also called orthophosphate) Amino Acid Method*



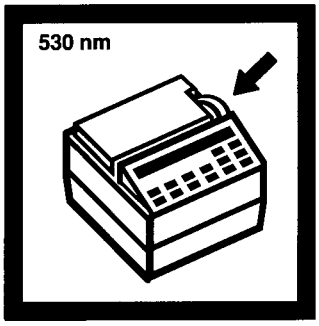
1. Enter one of the following stored program numbers for the reactive phosphorus, amino acid method.

Press: **4 8 5 READ/ENTER** for units of mg/L PO_4^{3-}
OR
4 8 7 READ/ENTER for units of mg/L P

The display will show:
DIAL nm TO 530

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

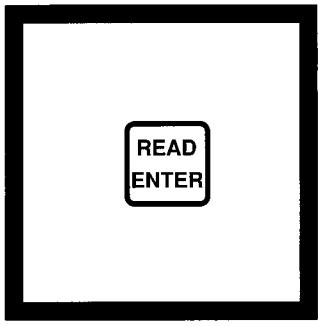
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



2. Rotate the wavelength dial until the small display shows:
530 nm

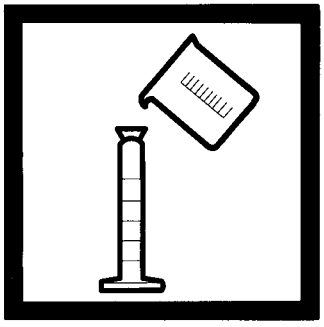
Note: *If sample cannot be analyzed immediately, see* Sampling and Storage *following these steps.*

Note: *For instruments with software versions that do not have stored program method 487, refer to* Instrument Setup *following these steps.*



3. Press: **READ/ENTER**

The display will show:
mg/l PO_4^{3-} AA
or
mg/l P AA

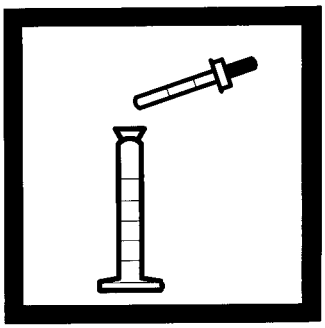


4. Fill a 25–mL graduated mixing cylinder with 25 mL of sample.

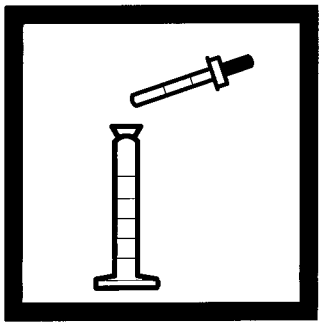
Note: *For proof of accuracy, use a 10.0 mg/L as PO_4^{3-} (3.3 mg/L as P) phosphorus standard solution (preparation given in the* Accuracy Check*) in place of the sample.*

Note: *A reagent blank should be run with each lot of reagent. Repeat the test using demineralized water as a sample. Subtract this value from each result obtained with this lot of reagent.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater,* 12th ed.



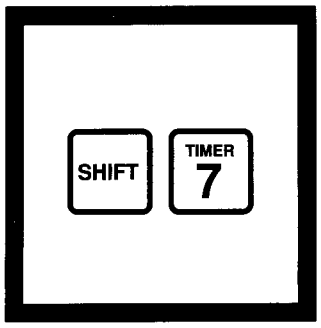
5. Add 1 mL of Molybdate Reagent using a 1–mL calibrated dropper.



6. Add 1 mL of Amino Acid Reagent Solution. Stopper and invert several times to mix (the prepared sample).

Note: A blue color will form if phosphate is present.

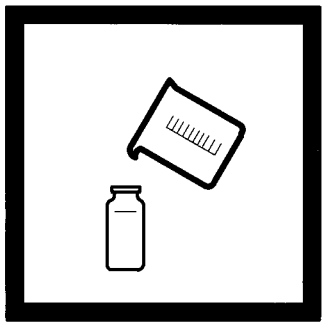
Note: Substitute the contents of one Amino Acid Reagent Powder Pillow for 1 mL of amino acid reagent solution if desired.



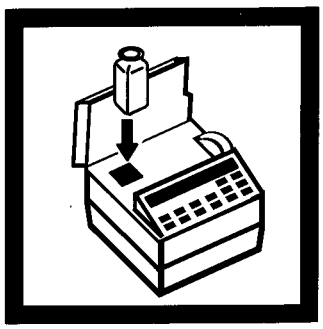
7. Press: **SHIFT TIMER**

A 10–minute reaction period will begin.

Note: Do step 8 while the timer is running.

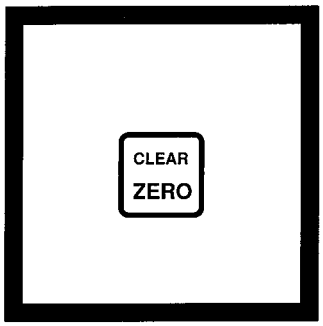


8. Pour 25 mL of sample (the blank) into a sample cell.



9. When the timer beeps, the display will show:
mg/l PO_4^{3-} AA
or
mg/l P AA
Place the blank into the cell holder. Close the light shield.

Note: The Pour–Thru Cell can be used with this procedure.

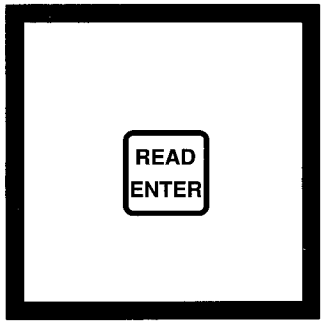


10. Press: **ZERO**

The display will show:
WAIT
then:
0.00 mg/l PO_4^{3-} AA
or
0.00 mg/l P AA

11. Pour the prepared sample into a sample cell. Place the prepared sample into the cell holder. Close the light shield.

Note: If more than five minutes elapse after the timer beeps, ZERO SAMPLE may appear. If so, remove the prepared sample, insert the blank and press: ZERO. Insert the prepared sample and press: READ/ENTER.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L PO_4^{3-} or mg/L P will be displayed.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

Note: Use the following conversion factors to express the results in terms of other phosphorous forms:
mg/L $PO_4^{3-} \div 3.07$ = mg/L P
mg/L PO_4^{3-} X 0.747 = mg/L P_2O_5
mg/L P X 2.29 = mg/L P_2O_5

INSTRUMENT SETUP

For a DR/2000 with a software version before 2.0 which does not have the Phosporus, Reactive, Amino Acid Method, Stored Method #487, enter the following calibration as an operator– programmed calibration. Follow the steps in the *Operation* section of the *DR/2000 Instrument Manual.* Store the method as follows:

nm = 530
Decimal = 00.00
Units = mg/l
Symbol = P AA
Timer 1 = 10:00

The calibration is first entered with 0.000 absorbance values for zero and #1 standard. To do this, leave the sample cell compartment empty, and begin by storing zero and #1 standard as concentrations of 0 and 9.40 mg/l, respectively. Accept 0.000 Abs as the value for both standards. Next, the values for the zero standard and #1 standard must be changed to the values given below.

Standard	Absorbance	Concentration
0	0.009	0
1	1.375	9.40

The method is now stored as an operator–programmed method with a number between 950 and 999. Record the method number for future reference.

For a DR/2000 with software versions 2.0 and 2.2 that do not have the Phosphorus, Reactive, Amino Acid Method, Stored Method #487, enter the calibration as a Hach–entered program.

1. Press: [I/O]

2. Press: [SHIFT] [CONFIG METH]

3. Press: [%T 4] [– 8] [TIMER 7] [READ ENTER]

4. Within 3 seconds, press:

[SHIFT] [PROG 3] [CONFIG METH]

The display will show:

ENTER nm

If the display returns to the METHOD prompt, repeat the sequence.

5. Press: [ABS 5] [PROG 3] [0]

Note: If an error is made, press SHIFT, CLEAR and re–enter the number. When the number is correct, press READ/ENTER. The display will show:

DECIMAL? 00.00

6. The decimal point is already correctly positioned. Press **READ/ENTER.** The display will read:

UNITS?

7. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. The display will show:

mg/l

8. With the proper unit of measure displayed, press **READ/ENTER.** The display will read:

SYMBOL?

9. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key repeatedly until the display reads:

mg/l p

10. Press **SHIFT** to make the "p" upper case. The display will show:

mg/l P

11. Press **READ/ENTER** to accept the capital "P". Continue to construct the display to read:

mg/l P AA

12. When the last character of the symbol is accept with the **READ/ENTER** key, the display will show:

TIMER?

There is one timer for this method. Press **SHIFT TIMER.** The display will read:

MM:SS TIME 1?

13. To enter the first timer value of 10:00 minutes, press:

[BATT 1] [0] [0] [0]

The display will read:

10:00 TIME 1?

14. Press **READ/ENTER** to accept the value. The display will then read:

MM:SS TIME 2?

15. Press **READ/ENTER** to complete timer entry. The display will show:

 # 1 DATA

16. Enter the following 12 numbers as shown. Complete each number with the **READ/ENTER** key.

#1 DATA	**0**
#2 DATA	**20566**
#3 DATA	**22102**
#4 DATA	**22102**
#5 DATA	**22102**
#6 DATA	**22102**
#7 DATA	**22102**
#8 DATA	**65535**
#9 DATA	**65535**
#10 DATA	**3276**
#11 DATA	**512**
CHECKSUM	**61746**

The final number is a check value which is used to determine that the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1 and the entire sequence must be re-entered. If all numbers are correctly entered, the update procedure is complete and the display will return to the method prompt:

METHOD?

With a new method 487 successfully entered, block access to the now obsolete method 486. Press:

Within 3 seconds, press:

Access to method 486 is now blocked.

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized 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 up to 24 hours by storing at or below 4 °C. For longer storage periods, add 4.0 mL of mercuric chloride to each liter of sample and mix. (Use of mercuric chloride is discouraged due to health and environmental concerns.) Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L or higher to prevent mercury interference in the test. Spike samples low in chloride with a sodium chloride solution (5 ml of 10,246 mg/L sodium chloride solution per liter of sample).

ACCURACY CHECK

Standard Addition Method

a) Snap the neck off a Phosphate Voluette Ampule Standard, 500 mg/L PO_4^{3-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectivley, to three 25–mL aliquots of a water samples. Mix well.

c) Analyze each sample as described in the procedure. Compare the results with the original test sample. Each 0.1–mL addition should increase the orthophosphate (PO_4^{3-}) 2.0 mg/L for stored program 485. When using stored program 487, the increase should be 0.65 mg/L P for each 0.1–mL addition of standard.

d) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method

Prepare a 10.0–mg/L PO_4^{3-}(3.3 mg/L P) standard solution by pipetting 10.0 mL of Phosphate Standard Solution, 50 mg/L as PO_4^{3-}, into a 50–mL volumetric flask. Dilute to volume with demineralized water.

Or, prepare a 10.0–mg/L PO_4^{3-} (3.3 mg/L P) standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate Voluette Ampul Standard, 500 mg/L PO_4^{3-}, into a 50–mL volumetric flask. Dilute to volume with demineralized water.

PRECISION

In a single laboratory, using a standard solution of 12 mg/L PO_4^{3-} and two lots of reagents with a DR/2000, a single operator obtained a standard deviation of ±0.02 mg/L PO_4^{3-}.

INTERFERENCES

Samples with large amounts of turbidity may give inconsistent results. Some of the suspended particles may dissolve because of the acid used in the test. Also, results will vary because of the variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25–mL sample. Use this in place of the sample as the blank to zero the instrument in Step 10. Use a pipet and pipet filler when meausring the sulfuric acid solution.

For best results, the temperature of the sample should be 21 ±3 °C (70 ±5 °F).

Sulfide interferes. For samples with sulfide concentrations of less than 5 mg/L, use the following procedure to eliminate the interference:

a) Measure 50 mL of sample in a graduated cylinder. Pour the measured sample into a 125–mL erlenmeyer flask.

b) Add Bromine Water dropwise with a constant swirling until a permanent yellow color develops.

c) Add Phenol Solution drop-wise until the yellow color just disappears.

d) Use this solution in Steps 4 and 8 of the procedure.

Nitrites bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid to the sample. Swirl to mix. Continue with Step 5.

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

Chloride	150,000 mg/L as Cl^-
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	40,000 mg/L as $CaCO_3$

When phosphate is determined in waters containing high salt levels, low results may occur. To eliminate this interference, dilute the sample until two successive dilutions yield approximately the same results.

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_4^{3-}. If a color other than blue is formed, dilute the sample and retest.

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

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

	Cat. No.
High Range Reactive Phosphorus Reagent Set (100 Tests) Includes: (1) 1934–32, (1) 2236–32	22441–00

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid Reagent	1 mL	100 mL MDB*	1934–32
Molybdate Reagent	1 mL	100 mL MDB*	2236–32

REQUIRED APPARATUS

Cylinder, 25 mL, graduated mixing	1	each	1896–40

OPTIONAL REAGENTS

Amino Acid Reagent Powder Pillows	100/pkg	804–99
Bromine Water, 30 g/L	29 mL*	2211–20
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884–49
Mercuric Chloride Standard Solution	100 mL	14994–42
Phenol Solution, 30 g/L	29 mL	2112–20
Phosphate Standard Solution, 50 mg/L PO_4^{3-}	500 mL	171–49
Phosphate Standard Solution, Voluette ampule, 500 mg/L PO_4^{3-}, 10 mL	16/pkg	14242–10
Sodium Chloride Standard Solution, 10,246 mg/L NaCl	100 mL	23074–42
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB*	2450–32
Sulfamic Acid, ACS	113 g	2344–14
Sulfuric Acid Standard Solution, 10 N	1 L	931–53
Water, demineralized	4 L	272–56

*Larger sizes available.

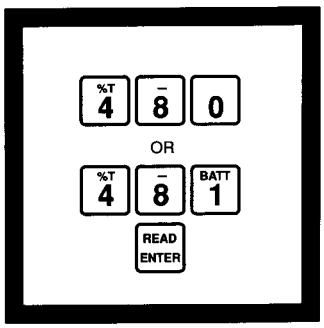
OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968–00
Clippers, for opening powder pilows	each	968–00
Cylinder, graduated, 50 mL	each	508–41
Flask, erlenmeyer, 125 mL	each	505–43
Flask, volumetric, Class, A, 50.00 mL	each	14574–41
Funnel, poly, 65 mm	each	1083–67
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391–33
pH Meter, EC10, portable	each	50050–00
Pipet Filler	each	12189–00
Pipet, serological, 2 mL	each	532–36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 TenSett Pipet	50/pkg	21856–96
Pipet, volumetric, Class A, 10.00 mL	each	14515–38
Pour–Thru Cell Assembly Kit	each	45215–00
Spoon, measuring, 0.05 g	each	492–00
Thermometer, –20 to 105 °C	each	1877–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

PHOSPHORUS, REACTIVE (0 to 45.0 mg/L PO_4^{3-})

(also called orthophosphate) Molybdovanadate Method*



1. Enter the stored program number for reactive phosphorus, molybdovanadate method.

Press: **4 8 0 READ/ENTER** for units of mg/L PO_4^{3-}

OR

Press: **4 8 1 READ/ENTER** for units of mg/L P

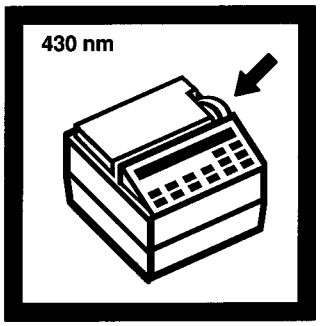
The display will show:

DIAL nm to 430

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

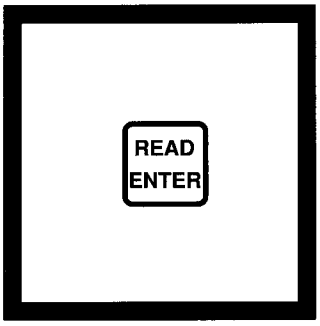
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If sample cannot be analyzed immediately, see* Sampling and Storage *following these steps.*



2. Rotate the wavelength dial until the small display shows:

430 nm



3. Press: **READ/ENTER**

The display will show:

mg/l PO_4^{3-} MoV

OR

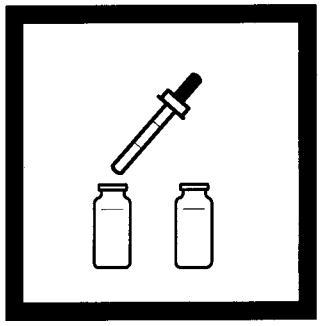
mg/l P MoV

4. Fill a sample cell (the blank) with 25 mL of demineralized water with a 25–mL graduated cylinder.

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

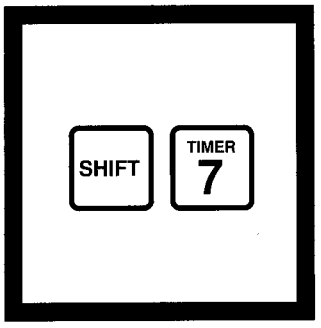
5. Fill a second sample cell (the prepared sample) with 25 mL of sample with a 25–mL graduated cylinder.

Note: For proof of accuracy, use a 10.0 mg/L phosphate (3.3 mg/L phosphorus) standard solution (preparation given in the Accuracy Check*) in place of the sample.*



6. Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Swirl to mix.

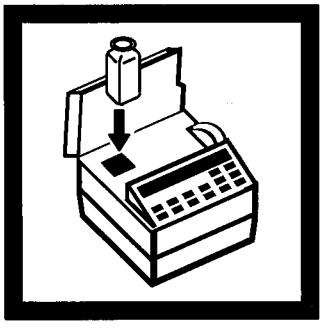
Note: A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank because of the reagent.



7. Press: **SHIFT TIMER**

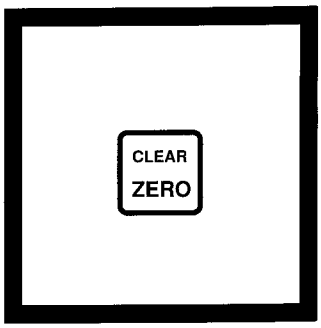
A 3–minute reaction period will begin.

Note: If the sample concentration is greater than 24 mg/L PO_4^{3-}, read at exactly 3 minutes or make a 1:1 dilution of the sample.



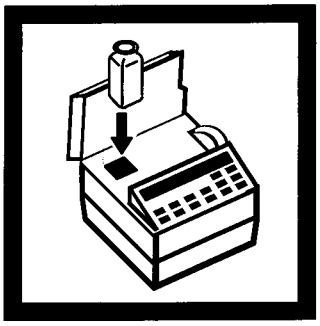
8. When the timer beeps, the display will show:
mg/l PO_4^{3-} MoV
OR
mg/l P MoV
Place the blank into the cell holder. Close the light shield.

Note: The Pour–Thru Cell can be used with this procedure.

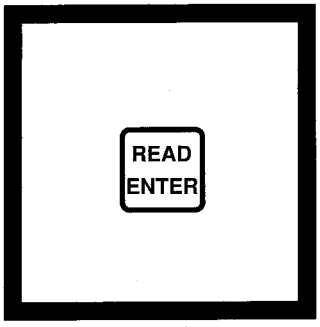


9. Press: **ZERO**

The display will show:
WAIT
then:
0.0 mg/l PO_4^{3-} MoV
OR
0.0 mg/l P MoV



10. Place the prepared sample into the cell holder. Close the light shield.



11. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L PO_4^{3-} or mg/L P will be displayed.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

Note: Phosphorus Conversions
mg/L X PO_4^{3-} = mg/L P X 3.07
mg/L P_2O_5 = mg/L P X 2.25
mg/L P_2O_5 = mg/L PO_4^{3-} X 0.75

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized water. Do not use a commercial detergent because the phosphate content will contaminate the sample.

If samples cannot be analyzed the same day, adjust the pH to 2 or less by adding about 2 mL of sulfuric acid, ACS, per liter of sample. Store the sample at 4 °C (39 °F) or below. Samples can be stored up to 24 hours. For longer storage periods, add 4.0 mL of Mercuric Chloride Solution for each liter of sample taken and mix. Use of mercuric chloride is discouraged to minimize the amount of mercury released to the environment. Sample refrigeration is still required. Sample preserved with mercuric chloride must be spiked with 0.1 g sodium chloride level to 50 mg/L or more if the sample is low in chloride. The addition of chloride prevents mercury interference in the test.

Before analysis, adjust the acidified sample to about pH 7 by adding 5.0 N Sodium Hydroxide Standard Solution. Mix thoroughly. Warm to room temperature before analyzing.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as 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 water samples. Mix well.

c) Analyze each sample as described in the procedure and compare the results with that of the original test sample. Each 0.1–mL addition of standard should cause an increase of 2.0 mg/L PO_4^{3-} or 0.67 mg/L P.

d) If these increases do not occur, see Standard Additions in Section I for more information.

Standard Solution Method

A 10.0 mg/L phosphate standard can be prepared by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L PO_4^{3-}, into a 50–mL volumetric flask. Dilute to volume with demineralized water.

Wavelength Check

This test is sensitive to the wavelength setting. To ensure accuracy, the test should be run on a 10–mg/L standard solution and blank. Repeat Step 8 to 11 at slightly different wavelengths, setting the dial from higher to lower values until the correct result is obtained. The wavelength should be 430 ±2 nm. Always set this wavelength from high to low values.

PRECISION

In a single laboratory, using standards of 20.0 mg/L PO_4^{3-}, two lots of reagent and the DR/2000, a single operator obtained a standard deviation of ±0.09 mg/L PO_4^{3-}.

INTERFERENCES

Sulfide interference may be removed by oxidation with Bromine Water as follows:

a) Measure 25 mL of sample into a sample cell.

b) Add Bromine Water drop–wise with constant swirling until permanent yellow color develops.

c) Add Phenol Solution drop–wise until the yellow color just disappears. Proceed with Step 5 using this treated sample.

Positive interferences are caused by silica and arsenate only if the sample is heated. Negative interferences are caused by arsenate, fluoride, thorium, bismuth, sulfide, thiosulfate, thiocyanate or excess molybdate. Blue color is caused by ferrous iron but this does not affect results if ferrous iron concentration is less than 100 mg/L. Ions that do not interfere in concentrations up to 1000 mg/L are pyrophosphate, molybdate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{3+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, $C10_4^{-}$, CN^{-}, IO_3^{-}, SiO_4^{4-}.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment *(see pH Interference in Section I)*.

SUMMARY OF METHOD

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

Description	Quantity Required Per Test	Units	Cat. No.
Molybdovanadate Reagent	2.0 mL	100 mL* MDB	20760–32
Water, demineralized	25 mL	4 L	272–56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508–40

OPTIONAL REAGENTS

Bromine Water	29 mL*	2211–20
Hydrochloric Acid Solution, 1:1	500 mL	884–49
Mercuric Chloride Solution, 10 g/L	100 mL	14994–42
Phenol Solution, 30 g/L	29 mL	2112–20
Phosphate Standard Solution, 50 mg/L as PO_4^{3-}	500 mL	171–49
Phosphate Standard Solution, Voluette ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242–10
Sodium Chloride, ACS	454 g	182–01
Sodium Hydroxide Standard Solution, 5.0 N	100 mL** MDB	2450–32
Sulfuric Acid, ACS	500 mL*	979–49

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968–00
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 Indicator Paper, 1 to 11 pH	5 rolls/pkg	391–33
pH Meter, EC10, portable	each	50050–00
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, volumetric, Class A, 10.00 mL	each	14515–38
Pipet Filler	each	12189–00
Pour–Thru Cell Assembly Kit	each	45215–00
Spoon, measuring, 0.1 g	each	511–00

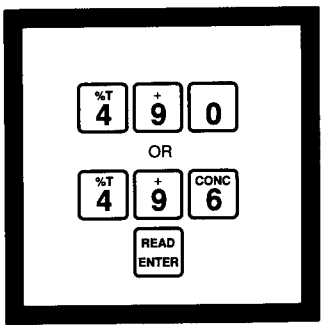
For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

*Contact Hach for larger sizes

PHOSPHORUS, REACTIVE (0 to 2.50 mg/L PO_4^{3-}) For water, wastewater and seawater

(also called Orthophosphate) PhosVer 3 (Ascorbic Acid) Method* (Powder Pillows or AccuVac Ampuls), USEPA accepted for reporting**

USING POWDER PILLOWS



1. Enter a stored program number for reactive phosphorus powder pillows.

Press: **4 9 0 READ/ENTER** for units of mg/L PO_4^{3-}

OR

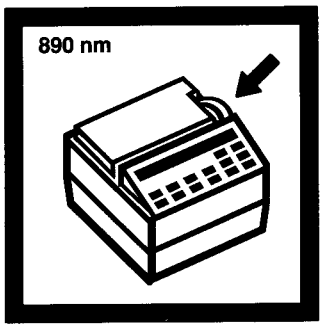
Press: **4 9 6 READ/ENTER** for units of mg/L P

The display will show:
DIAL nm TO 890

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

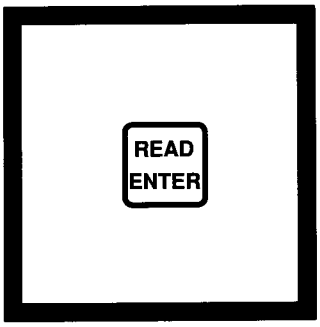
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *Measurement range for P is 0 to 0.83 mg/L*



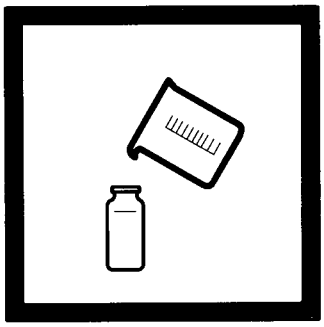
2. Rotate the wavelength dial until the small display shows:
890 nm

Note: *For instruments with software versions that do not have stored program method 496, refer to* Instrument Setup *following these steps.*



3. Press: **READ/ENTER**

The display will show:
mg/l PO_4^{3-} PV
OR
mg/l P PV



4. Fill a sample cell with 25 mL of sample.

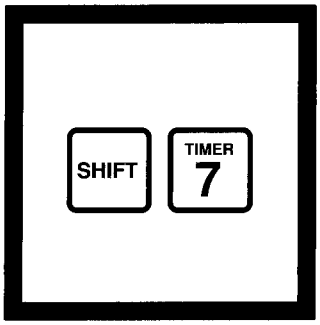
Note: *For proof of accuracy, use a 1.0 mg/L Phosphate (0.33 mg/L P) Standard Solution listed under* Optional Reagents *in place of the sample.*

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

** Procedure is equivalent to USEPA method 365.2 and Standard Method 4500–P–E for wastewater.

5. Add the contents of one PhosVer 3 phosphate Powder Pillow to the sample cell (the prepared sample). Swirl immediately to mix.

Note: A blue color will form if phosphate is present.

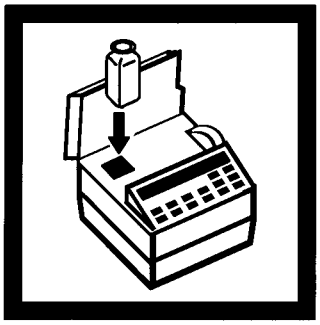


6. Press: **SHIFT TIMER**

A 2–minute reaction period will begin.

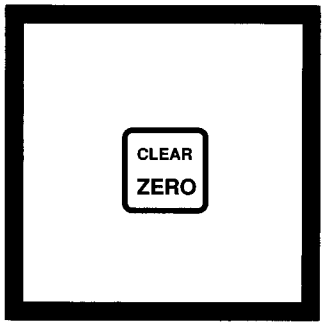
Note: An 8–10 minute reaction period should be used if determining total phosphate following the acid–persulfate digestion.

Note: If the sample temperature is less than 15 °C (59 °F), allow 4 minutes of reaction time.



7. Fill another sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

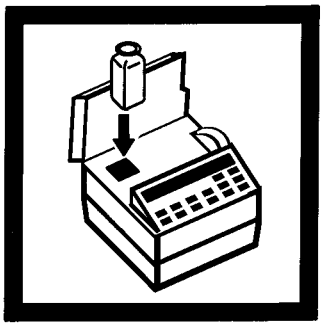
Note: The Pour–Thru Cell can be used with this procedure.



8. When the timer beeps, the display will show:
mg/l P PV

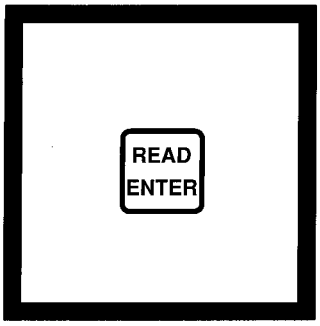
Press: **ZERO**

The display will show:
WAIT
then:
0.00 mg/l PO_4^{3-} PV
OR
0.00 mg/l P PV



9. Place the prepared sample into the cell holder. Close the light shield.

Note: Run a reagent blank for this test. Use demineralized water in place of the sample is Step 4. Subtract this result from all test results run with this lot of PhosVer 3.



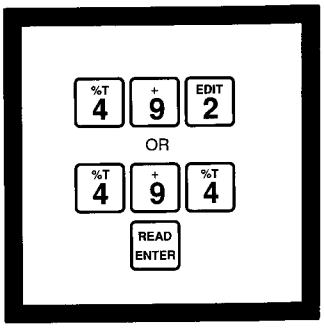
10. Press: **READ/ENTER**

The display will show:
WAIT
then the results in mg/L PO_4^{3-} or mg/L P will be displayed.

Note: mg/L PO_4^{3-} results can be expressed as mg/L phosphorus by dividing by 3 or as mg/L phosphorus pentoxide (P_2O_5) by multiplying by 0.75.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

USING ACCUVAC AMPULS



1. Enter the stored program number for reactive phosphorus AccuVac ampuls.

Press: **4 9 2 READ/ENTER** for units of mg/L PO_4^{3-}

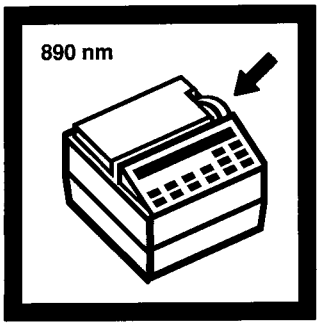
OR

Press: **4 9 4 READ/ENTER** for units of mg/L P

The display will show:
DIAL nm TO 890

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

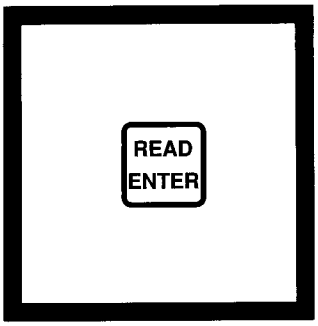
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



2. Rotate the wavelength dial until the small display shows:

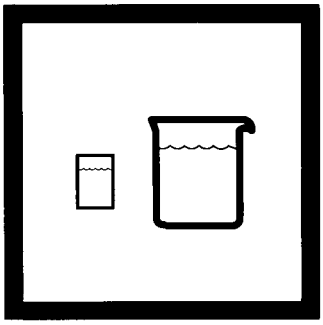
890 nm

Note: *For instruments with software versions that do not have stored program method 494, refer to* Instrument Setup.

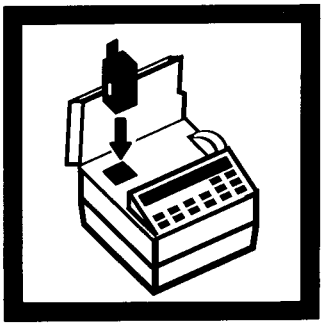


3. Press: **READ/ENTER**

The display will show:
mg/l PO_4^{3-} PV AV
OR
mg/l P PV AV

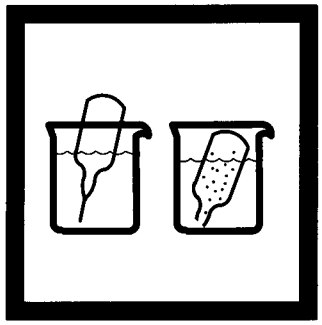


4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50–mL beaker.



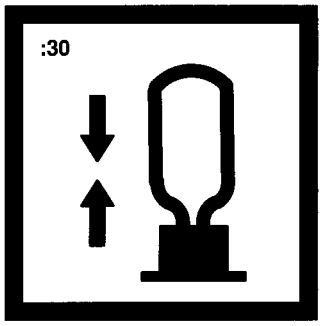
5. Place the AccuVac Vial Adapter into the cell holder.

Note: Place the grip tab at the rear of the cell holder.



6. Fill a PhosVer 3 Phosphate AccuVac ampul with sample.

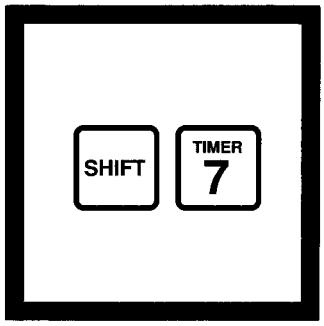
Note: Keep the tip immersed while the ampul fills completely.



7. Place an ampul cap securely over the tip of the ampul. Shake the ampul for approximately 30 seconds. Wipe off any liquid and fingerprints.

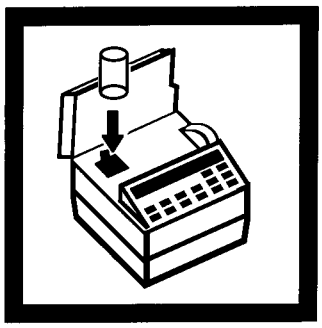
Note: A blue color will form if phosphate is present.

Note: Accuracy is unaffected by undissolved powder.

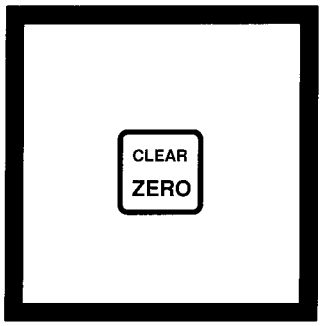


8. Press: **SHIFT TIMER**

A 2–minute reaction period will begin.

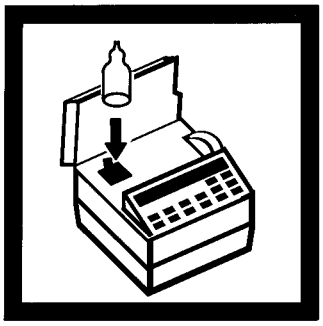


9. When the timer beeps, the display will show:
mg/l PO_4^{3-} PV AV
OR
mg/l P PV AV
Place the zeroing vial into the cell holder.



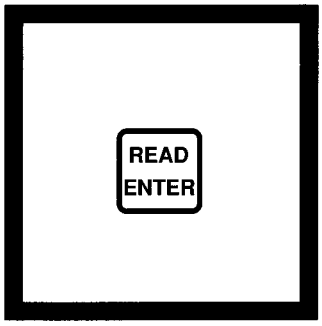
10. Press: **ZERO**

The display will show:
WAIT
then:
0.00 mg/l PO_4^{3-} PV AV
OR
0.00 mg/l P PV AV



11. Place the AccuVac ampul into the cell holder.

Note: Run a reagent blank for the test. Use demineralized water in place of the sample in Step 4. Subtract this result from all results with this lot of ampuls.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L PO_4^{3-} or mg/L P will be displayed.

Note: mg/L PO_4^{3-} results can be expressed as mg/L phosphorus by dividing by 3 or as mg/L phosphorus pentoxide (P_2O_5) by multiplying by 0.75.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

INSTRUMENT SETUP

For a DR/2000 with a software version before 2.0 which does not have Phosphorus, Reactive, Ascorbic Acid Method, Stored Method 496 and Stored Method 494, enter the following calibrations as operator–programmed calibrations. Follow the steps in the *Operation* section of the *DR/2000 Instrument Manual*. Store the method as follows:

Stored Method 496	**Stored Method 494**
nm = 890	nm = 890
Decimal = 00.00	Decimal = 00.00
Units = mg/l	Units = mg/l
Symbol = P PV	Symbol = p PV AV
Timer 1 = 02:00	Timer 1 = 02:00

The calibrations are first entered with 0.000 absorbance values for zero and #1 standards. To do this, leave the sample cell compartment empty, and begin by storing zero and #1 standard as concentrations of 0 and 0.79 mg/L, respectively, for Stored Method 496 and 0 and 0.77 mg/L for Stored Method 494. Accept 0.000 Abs as the value for all standards. Next, the values for the zero standard and #1 standard must be changed to the values given below:

Stored Method 496

Standard	Absorbance	Concentration
0	0.019	0
1	1.375	0.79

Stored Method 494

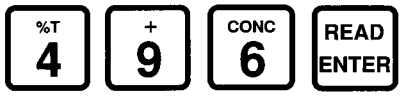
Standard	Absorbance	Concentration
0	0.003	0
1	1.250	0.77

The methods are now stored as operator–programmed methods with numbers between 950 and 999. Record the method numbers for future reference.

For a DR/2000 with a software version of 2.0 or 2.2 that does not have the Phosphorus, Reactive, Ascorbic Acid Method, Stored Method, Stored Methods #496 and #494, enter the calibrations as Hach–entered programs.

1. Press: 

2. Press: 

3. Press: 

OR

Press: 

4. Within 3 seconds, press:

The display will show:

ENTER nm

If the display returns to the METHOD prompt, repeat the sequence.

5. Press: 

Note: If an error is made, press SHIFT, CLEAR and re–enter the number. When the number is correct, press READ/ENTER.

The display will show:

DECIMAL? 00.00

6. The decimal point is already correctly positioned. Press: **READ/ENTER.** The display will read:

UNITS?

7. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. The display will show:

mg/l

8. With the proper unit of measure displayed, press **READ/ENTER.** The display will read:

SYMBOL?

9. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key repeatedly until the display reads:

mg/l p

10. Press **SHIFT** to make the "p" upper case. The display will show:

mg/l P

11. Press **READ/ENTER** to accept the capital "P". Continue to construct the display to read:

mg/l P PV

OR

mg/l P PV AV

The space is the "character" displayed after one press of the **DOWN ARROW** key.

12. When the last character of the symbol is accepted with the **READ/ENTER** key, the display will show:

TIMER?

There is one time selection for this method. Press **SHIFT, TIMER.** The display will read:

MM:SS TIME 1?

13. To enter the first timer value of 02:00 minutes, press:

The display will read:

02:00 TIME 1?

14. Press **READ/ENTER** to accept the value. The display will then read:

MM:SS TIME 2?

15. Press **READ/ENTER** to complete timer entry. The display will show:

 # 1 DATA 0

16. Enter the following 12 numbers as shown. Complete each number with the **READ/ENTER** key.

Stored Method 496	
# 1 Data	**0**
# 2 Data	**1544**
# 3 Data	**1799**
# 4 Data	**1800**
# 5 Data	**1799**
# 6 Data	**2055**
# 7 Data	**1799**
# 8 Data	**65535**
# 9 Data	**65535**
# 10 Data	**3276**
# 11 Data	**512**
CHECKSUM	**50954**

Stored Method 494	
# 1 Data	**0**
# 2 Data	**1800**
# 3 Data	**2056**
# 4 Data	**2055**
# 5 Data	**2056**
# 6 Data	**2055**
# 7 Data	**2303**
# 8 Data	**65535**
# 9 Data	**65535**
# 10 Data	**3276**
# 11 Data	**512**
CHECKSUM	**49425**

The final number in each group is a check value which is used to determine that the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1 and the entire sequence must be re–entered. If all numbers are correctly entered, the update procedure is complete and the display will return to the method prompt:

METHOD?

With the new method 496 and 494 successfully entered, block access to the now obsolete methods 491 and 493.

Press:

Within 3 seconds, press:

Press:

Within 3 seconds, press:

Access to methods 491 and 493 are now blocked.

SAMPLING AND STORAGE

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with demineralized water. Do not use commercial detergents containing phosphate for cleaning glassware used in phosphate analysis. Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, preserve samples up to 24 hours by storing at or below 4 °C. For longer storage periods, add 4.0 mL of Mercuric Chloride Solution to each liter of sample taken and mix. Use of mercuric chloride is discouraged whenever possible for health and environmental considerations. Sample refrigeration is still required. Samples preserved with mercuric chloride must have a sodium chloride level of 50 mg/L or more to prevent mercury interference. Samples low in chloride should be spiked with 0.1 g sodium chloride per liter of sample.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 50 mg/L PO_4.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25–mL water sample. Mix each thoroughly. (For AccuVac ampuls use 50–mL beakers.)

c) Analyze each sample as described above. The phosphate concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section I.*

INTERFERENCES

Large amounts of turbidity may cause inconsistent results in the phosphate tests because the acid present in the powder pillow 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 Powder Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.

The PhosVer 3 Reagent Powder Pillows shoud be stored in a cool, dry place.

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

Aluminum	200 mg/L
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Zinc	80 mg/L

Arsenate and hydrogen sulfide interfere.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment *(see pH Interference in Section I).*

PRECISION

In a single laboratory, using a standard solution of 1.00 mg/L PO_4^{3-} and two lots of reagent with a DR/2000, a single operator obtained a standard deviation of ±0.01 mg/L PO_4^{3-}.

In a single laboratory, using a standard solution of 1.00 mg/L PO_4^{3-} and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ±0.02 mg/L PO_4^{3-}.

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.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Units	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1 Pillow	100/pkg	2125–99

REQUIRED REAGENTS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent AccuVac Ampuls	1 ampul	25/pkg	25080–25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening Powder pillows	1	each	968–00

REQUIRED APPARATUS (Using Powder Pillows)

Adapter, AccuVac Vial	1	each	43784–00
Beaker, 50 mL	1	each	500–41
Cap, ampul, blue	1	6/pkg	1731–06
Vial, zeroing	1	each	21228–00

OPTIONAL REAGENTS

Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884–49
Mercuric Chloride Solution, 10 g/L	100 mL	14994–42
Phosphate Pretreatment Powder Pillows	50/pkg	14501–66
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569–42
Phosphate Standard Solution, Voluette ampul, 50 mg/L as PO_4, 10 mL	16/pkg	171–10
Sodium Chloride, ACS	454 g	182–01
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450–32
Water, demineralized	4 L	272–56

OPTIONAL APPARATUS

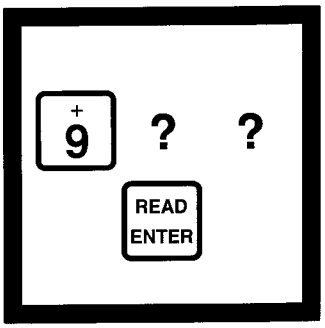
AccuVac Snapper Kit	each	24052–00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391–33
pH Meter, EC10, portable	each	50050–00
Pipet, 2 mL serological	each	532–36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01	50/pkg	21856–96
Pipet Filler, safety bulb	each	14651–00
Spoon, measuring, 0.1 g	each	511–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

*Larger sizes available.

POTASSIUM (0 to 7.0 mg/L)

Tetraphenylborate Method



1. Enter the user stored program number for potassium (K) previously determined during the calibration below.

Press: **9 ? ? READ/ENTER**

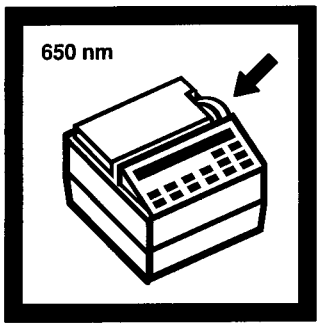
The display will show:
DIAL nm TO 650

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

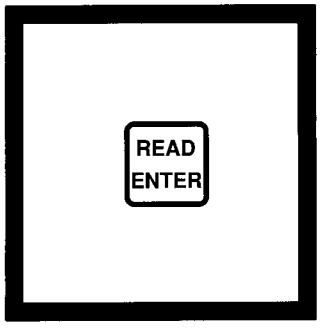
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

Note: *Because of potential variation between lots of Potassium 3 Reagent, perform a new calibration for each lot of reagent to obtain best accuracy. Prepare and store the calibration as directed under a* Calibration *below.*

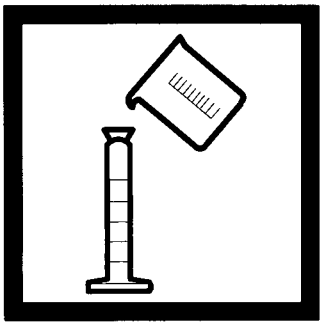


2. Rotate the wavelength dial until the small display shows:
650 nm



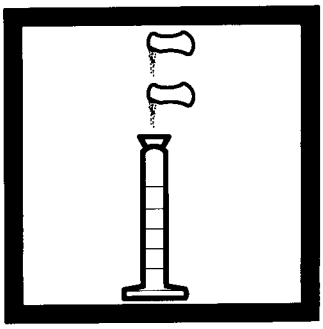
3. Press: **READ/ENTER**

The display will show:
mg/l K

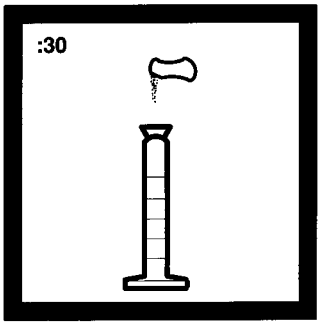


4. Fill a graduated mixing cylinder with 25 mL of sample.

Note: *Filter highly colored or turbid samples. Use filtered sample here and in Step 9.*

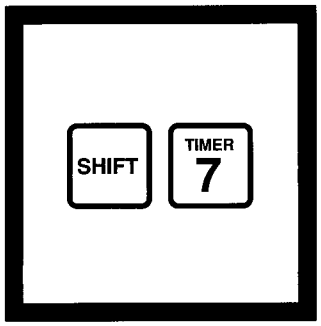


5. Add the contents of one Potassium 1 Reagent Powder Pillow. Add the contents of one Potassium 2 Reagent Solution Pillow. Stopper. Invert several times to mix.



6. Add the contents of one Potassium 3 Reagent Powder Pillow after the solution clears. Stopper. Shake for 30 seconds.

Note: A white turbidity will form if potassium is present.

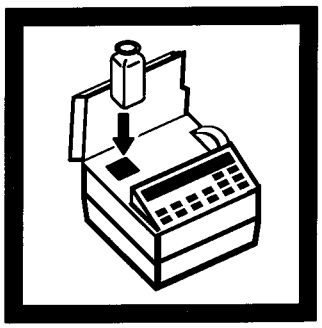


7. Press: **SHIFT TIMER**

A 3–minute reaction period will begin.

8. Pour the solution from the cylinder into a sample cell (the prepared sample).

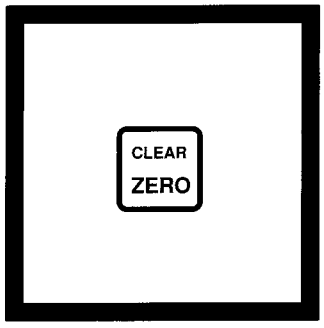
Note: The Pour–Thru Cell cannot be used with this procedure.



9. When the timer beeps, the display will show:

mg/l K

Fill the second sample cell (the blank) with 25 mL of sample. Place it into the cell holder.



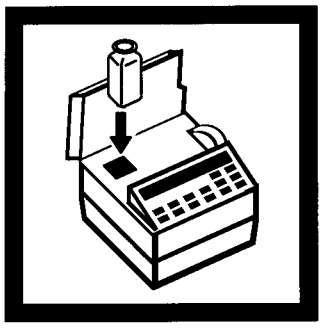
10. Press: **ZERO**

The display will show:

WAIT

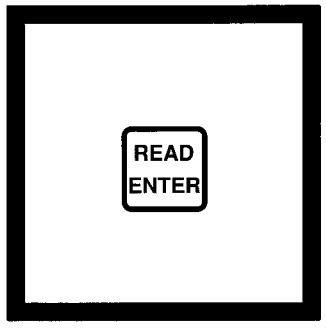
then:

0.0 mg/l K



11. Within seven minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.

Note: If more than five minutes elapse after the timer beeps, ZERO SAMPLE may appear. If so, remove the prepared sample, Insert the blank and press: ZERO. Insert the prepared sample.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L potassium will be displayed.

Note: Clean the cells with soap and a brush.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

CALIBRATION

A new calibration may be preformed for each lot of Potassium 3 Reagent Powder Pillows as follows:

a) Prepare standards of 0, 1, 2, 4, 6 and 8 mg/L potassium by diluting 0, 0.1, 0.2, 0.4, 0.6 and 0.8 mL of the contents of the Potassium Voluette Ampule Standard, 250 mg/L, to 25.0 mL with demineralized water in graduated mixing cylinders. Use a TenSette Pipet to measure the standard. Mix well. Or, pipet 0, 0.5, 1.0, 2.0, 3.0 and 4.0 mL from Potassium Voluette Ampule Standards, 500 mg/L, into 250–mL volumetric flasks. Dilute to volume. Mix well. Transfer 25 mL to each test cylinder.

b) Store the calibration in the instrument memory using the procedure in the Operation section of the instrument manual. Follow the procedure described, choosing a wavelength of 650 nm, the decimal position as 000.0, units as mg/L K, and a Timer 1 interval of 03:00. Note the program number assigned to the procedure.

c) Add the reagents to the demineralized water (0 standard–reagent blank) and to the 1 mg/L standard as described in Step 4 to 8 above, using the demineralized water blank to perform the zero calibration. Enter the potassium concentration of the first standard (1.0 mg/L) and measure the absorbance as directed by the instrument. React and measure the remaining standards.

d) Use this stored program number in the procedure above. Prepare a new calibration for each new lot of reagent, using the same stored program number.

SAMPLING AND STORAGE

Collect samples in acid–washed plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored at least six months at room temperature. Before analysis, adjust the pH to 4 to 5 with 5.0 N sodium hydroxide. Do not measure pH in the sample container with a pH electrode, as this will introduce potassium from the filling solution. Use pH paper or pour off sample and test pH in a separate beaker. Correct the test result for volume additions *(see Correction for Volume Additions in Section I).*

ACCURACY CHECK

Standard Addition Method

a) Snap the neck off a Potassium Voluette Ampule Standard Solution, 250 mg/L.

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 potassium concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method

Check test accuracy by using a 5.0 mg/L Potassium Standard Solution listed under Optional Reagents. Or, prepare this solution by diluting 5.00 mL of Potassium Standard Solution, 1000 mg/L to one liter with demineralized water.

PRECISION

In a single laboratory, using a standard solution of 4 mg/L K and one representative lot of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.13 mg/L K.

INTERFERENCES

The following ions do not interfere below the concentration shown:

Ammonium Nitrogen	15 mg/L as N
Calcium	7000 mg/L as $CaCO_3$
Chloride	15,000 mg/L
Magnesium	6000 mg/L as $CaCO_3$

SUMMARY OF METHOD

Potassium in the sample combines with sodium tetraphenylborate to form potassium tetraphenylborate, an insoluble white solid. The amount of turbidity produced is proportional to the potassium concentration.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Potassium 1 Reagent Powder Pillows	1 pillow	25/pkg	14321–98
Potassium 2 Reagent Solution Pillows	1 pillow	25/pkg	14322–98
Potassium 3 Reagent Powder Pillows	1 pillow	50/pkg	14323–96

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968–00
Cylinder, mixing, graduated, 25 mL	1	each	1896–40

OPTIONAL REAGENTS

Nitric Acid, ACS	500 mL	152–49
Nitric Acid, 1:1	500 mL	2540–42
Potassium Standard Solution, 5 mg/L	500 mL	20583–49
Potassium Standard Solution, 1000 mg/L	100 mL	22404–42
Potassium Standard Solution, Voluette ampule, 250 mg/L, 10 mL	16/pkg	14790–10
Potassium Standard Solution, Voluette ampule, 500 mg/L, 10 mL	16/pkg	21093–10
Sodium Hydroxide Solution, 5.0 N	59 mL SCDB	2450–26
Water, demineralized	4 L	272–56

OPTIONAL APPARATUS

Potassium at these levels can be determined directly using the Potassium Ion Selective Electrode (BNC connector), Cat. No. 44530–71

Ampule Breaker Kit	each	21968–00
Filter Paper, folded, 12.5 cm	100/pkg	1894–57
Flask, volumetric, 250 mL	each	547–46
Flask, volumetric, 1000 mL	each	547–53
Funnel, poly, 65 mm	each	1083–67
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391–33
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 TenSette Pipet 19700–01	50/pkg	21856–96
Pipet, volumetric, 0.50 mL, Class A	each	14515–34
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

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

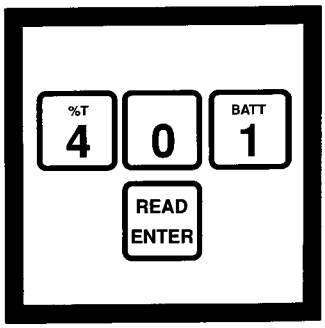
QUATERNARY AMMONIUM COMPOUNDS

Method 8337

(0 to 5 mg/L as CTAB)

For cooling tower and pool/spa water

Direct Binary Complex Method



1. Enter the stored program number for quaternary ammonium compounds (QAC).

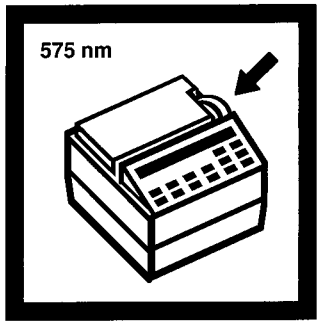
Press: **401 READ/ENTER**

The display will show:
DIAL nm TO 575

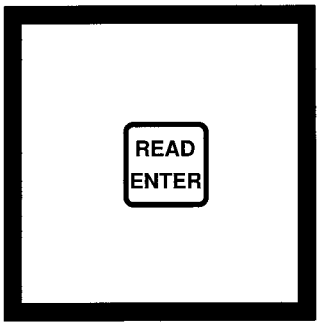
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *See* Instrument Setup *if using a DR/2000 without this stored program.*

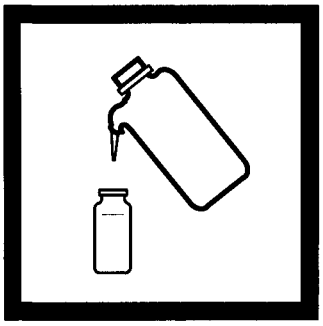


2. Rotate the wavelength dial until the small display shows:
575 nm

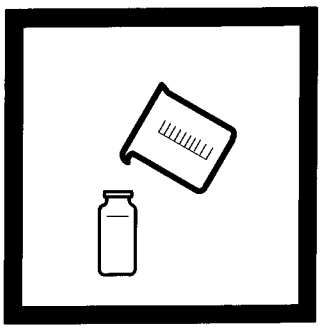


3. Press: **READ/ENTER**

The display will show:
mg/l QAC as CTAB

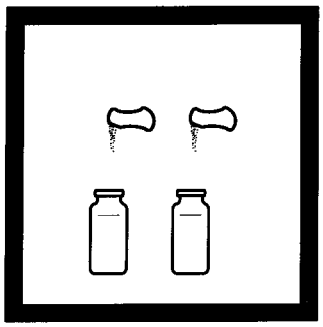


4. Fill a sample cell (the blank) with 25 mL of demineralized water.



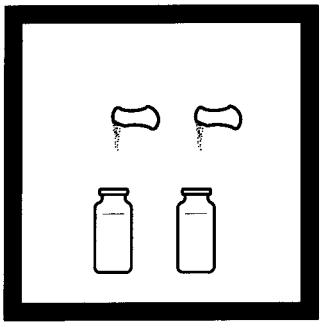
5. Fill another sample cell (the prepared sample) with 25 mL of sample.

Note: *For proof of accuracy, use a 5.0 mg/L CTAB standard solution (preparation given in the* Accuracy Check*) in place of the sample.*



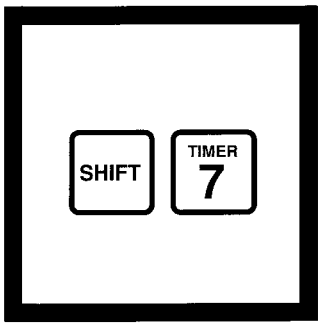
6. Add the contents of one Q.A.C. Reagent 1 Powder Pillow to each sample cell. Swirl (do not shake) the sample cells to dissolve the reagents.

Note: *Shaking the sample cell causes air bubble turbidity that dissipates slowly, interfering with the test results.*



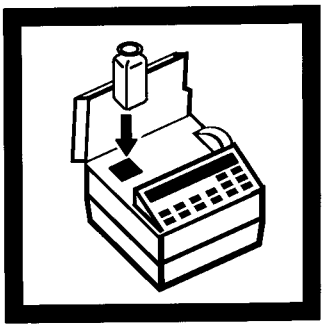
7. Add the contents of one Q.A.C. Reagent 2 Powder Pillow to each sample cell. Swirl (do not shake) the sample cells to dissolve the reagents.

Note: *A purple color will form if a quaternary ammonium compound is present.*



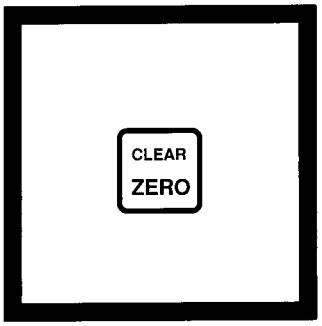
8. Press: **SHIFT TIMER**

A 2–minute reaction period will begin.



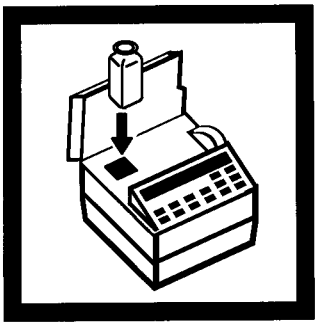
9. When the timer beeps, the display will show:
mg/l QAC as CTAB
Place the blank into the cell holder. Close the light shield.

Note: The Pour–Thru Cell can be used with this procedure.

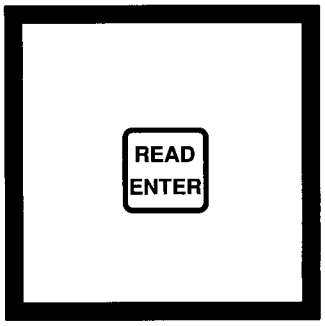


10. Press: **ZERO**

The display will show:
0.0 mg/l QAC as CTAB



11. Place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L quaternary ammonium compounds as cetyl–trimethylammonium bromide will be displayed.

Note: In the constant on mode, pressing READ/ENTER is not required. When the display stabilizes, read the result.

INSTRUMENT SETUP

For a DR/2000 with software versions 1.261 and 1.27 enter the Quaternary Ammonium Compounds, Direct Binary Complex Method, Stored Method #401, calibration as an operator–programmed calibration. Follow the steps in the *Operation* section of the *DR/2000 Instrument Manual.* Store the method as follows:

nm = 575
Decimal = 000.0
Units = mg/l
Symbol = QAC as CTAB
Timer 1 = 02:00

The calibration is first entered with 0.000 absorbance values for zero, #1 standard and #2 standard. To do this, do not place anything in the sample cell compartment. Begin by storing zero, #1 standard and #2 standard as concentrations of 0, 0.5 and 5.0, respectively, with nothing in the sample cell compartment. Accept 0.000 Abs. as the value for all standards. Next, the absorbance values for the #1 standard and #2 standard must be changed to the values given below.

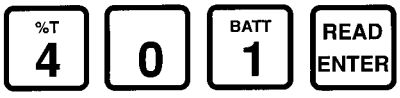
Standard	Concentration	Absorbance
0	0	0
1	0.5	0.065
2	5.0	1.132

The method is now stored as an operator–programmed method with a method number between 950 and 999. Record the method number for future reference.

For DR/2000s with software version 2.0 and 2.2 that do not have the Quaternary Ammonium Compounds Method, Stored Method #401, add the calibration as a Hach–stored program.

1. Press: 

2. Press: 

3. Press: 

4. Within 3 seconds press: **SHIFT PROGRAM METHOD.**
The display will show:
ENTER nm
If the display returns to the METHOD prompt, repeat the sequence.

5. Press: 

Note: If you make an error press SHIFT CLEAR and re–enter the number. When the number is correct, press READ/ENTER.

The display will show:
DECIMAL? 00.00

6. Use the arrow keys to correctly position the decimal point. For this method, press the **DOWN ARROW** key once. The display will show:
DECIMAL? 000.0

7. When the decimal point is correctly positioned, press **READ/ENTER.** The display will show:
UNITS?

8. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key once. The display will show:
mg/l

9. With the proper unit of measure displayed, press **READ/ENTER.** The display will show:
SYMBOL?

10. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key down repeatedly until you see:
mg/l q

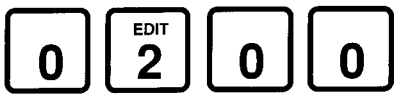
11. Press **SHIFT** to make the "q" upper case. The display will show:
mg/l Q

12. Press the **READ/ENTER** key to accept the capital "Q".

13. Continue to construct the display:
mg/l QAC as CTAB
The space is the character displayed after one press of the **DOWN ARROW** key.

14. When the last character of the symbol is accepted with the **READ/ENTER** key, the display will show:
TIMER?

15. This method uses one timed period, so press **SHIFT TIMER.** The display will show:
MM:SS TIME 1 ?

16. To enter the timer value of 2 minutes, press:


The display will show:
02:00 TIME 1 ?

17. Press **READ/ENTER** to accept the timer value. The display will show:
MM:SS TIME 2 ?

18. Press **READ/ENTER** to complete the timer entry. The display will show:
 # 1 Data

19. Enter the following 12 numbers as shown. Complete each number entry by pressing **READ/ENTER.**

# 1 Data	**0**
# 2 Data	**1798**
# 3 Data	**1285**
# 4 Data	**1286**
# 5 Data	**1285**
# 6 Data	**1285**
# 7 Data	**65535**
# 8 Data	**65535**
# 9 Data	**65535**
# 10 Data	**32760**
# 11 Data	**512**
CHECKSUM	**25328**

The final number is a check value which is used to determine if the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1 and the entire sequence must be re–entered. If all numbers are correctly entered, the display will return to the method prompt and is ready for use.

SAMPLING AND STORAGE

Collect samples in glass bottles that have been rinsed several times with sample before final sample filling. Do not use plastic containers as plastic adsorbs QACs.

ACCURACY CHECK

Standard Solution Method

To assure the accuracy of the test, use a 5 mg/L CTAB Standard Solution prepared as follows:

a) Pipet 5 mL from the Q.A.C. Standard Solution, 100 mg/L as CTAB, into a 100–mL volumetric flask.

b) Dilute the solution to 100 mL with demineralized water. Mix thoroughly.

c) Analyze 25 mL of the 5 mg/L CTAB standard solution according to the preceding procedure. The result should be 5.0 ±0.1 mg/L.

Standard Additions Method

a) Use a TenSette Pipet to add 0.5, 1.0 and 1.5 mL of Q.A.C. Standard Solution, 100 mg/L as CTAB, to three 50–mL samples. Mix thoroughly.

b) Analyze 25 mL each sample according to the above procedure. The QAC concentration should increase by 1.0 mg/L CTAB for each 0.5 mL addition of standard.

INTERFERENCES

Interference studies were conducted by preparing a CTAB standard solution of approximately 3 mg/L as well as a solution of the potential interference. The constituent was said to interfere when the resulting concentration changed by 10%.

Constituent	Level Above Which Constituent Interferes (mg/L)
Positive Interferences:	
Calcium (as $CaCO_3$)	1,350
Chlorine, HOCl and OCl^-	7
Igepal nonionic surfactant	3
Iodine, I_3^-	3
Iron, Fe^{3+}	80
Liquimine 14–P, filming amine	1,825
Magnesium, Mg^{2+}	1,350
Sodium polyphosphate	1,325
Tribenzylamine	7
Triton X–100 nonionic surfactant	4
Urea	8
Negative Interferences:	
Cyanuric acid	70
Niaproof anionic surfactant	11
Polyacrylic acid	16
Sodium lauryl sulfate	8

No Interferences:	Highest Concentration Tested (mg/L)
Potassium alum, $AlKS_2O_8$	500
Silica, H_2SiO_3	400
Sodium thiosulfate, $Na_2S_2O_3$	30

Highly buffered samples or extreme sample pH, may exceed the buffering capacity of the reagents and require sample pretreatment. Adjust the sample pH to between 3 and 5 by using a pH meter or pH paper and adding, dropwise, an appropriate amount of acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution. If significant volumes of acid or base are used, a volume correction should be made by dividing the total volume (sample + acid + base) by the original sample volume and then multiplying the test result by this factor.

After several samples have been analyzed, the sample cells may exhibit a build–up of a pink or purple color. A rinse with 1.0 N Sodium Hydroxide Solution followed by an Alconox detergent wash and demineralized water rinse will eliminate the build–up when it occurs.

PRECISION

In a single laboratory, using standard solutions of 2.0 mg/l CTAB and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.07 mg/L CTAB.

SUMMARY OF METHOD

The test method makes use of a colorimetric chemistry in which a quaternary ammonium compound reacts with an indicator to produce a color change from pale pink to vivid purple. The test is conducted in a stabilized, acid–buffered solution containing a masking agent to eliminate potential interferences. This test is applicable to the monitoring of QACs in swimming pools, cooling towers.

QUATERNARY AMMONIUM COMPOUNDS, continued

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Q.A.C. Reagent 1	2 pillows	50/pkg	24010–66
Q.A.C. Reagent 2	2 pillows	25/pkg	24012–68

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508–40
Clippers, for opening powder pillows	1	each	968–00

OPTIONAL REAGENTS

Q.A.C. Standard Solution, 100 mg/L as CTAB	100 mL	24153–42
Sodium Hydroxide Standard Solution, 1.0 N	1000 mL	1045–53
Sulfuric Acid Standard Solution, 1.0 N	59 mL SCDB	270–26
Water, demineralized	4 L	272–56

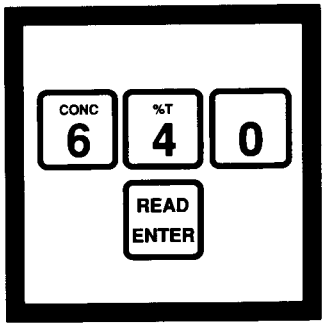
OPTIONAL APPARATUS

Filter Paper, folded, 12.5 cm	100/pkg	1894–57
Flask, volumetric, 100 mL, Class A	each	14574–42
Funnel, poly, 65 mm	each	1083–67
Pipet, TenSette 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 TenSette Pipet	50/pkg	21856–96
Pipet, volumetric, 5 mL, Class A	each	14515–37
Pipet Filler	each	12189–00
Pour–Thru Cell Assembly Kit	each	45215–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SELENIUM (0 to 1.00 mg/L)

Diaminobenzidine Method*



1. Enter the stored program number for selenium (Se).

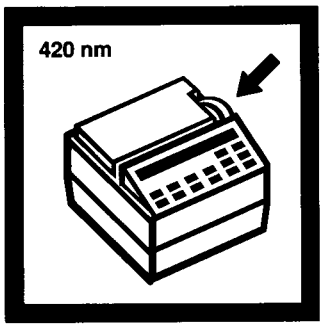
Press: **6 4 0 READ/ENTER**

The display will show:
DIAL nm TO 420

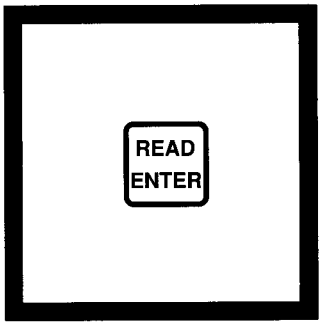
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: If sample cannot be analyzed immediately, see Sampling and Storage *following these steps. Adjust the pH of stored samples before analysis.*

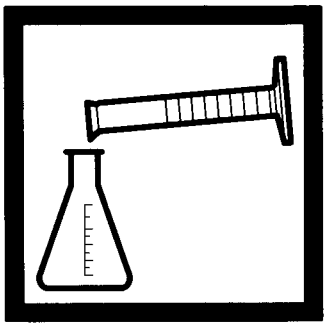


2. Rotate the wavelength dial until the small display shows:
420 nm



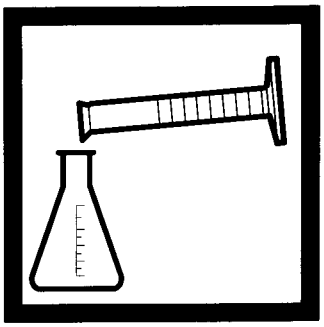
3. Press: **READ/ENTER**

The display will show:
mg/l Se



4. Measure 100 mL of demineralized water into a 500–mL erlenmeyer flask (the blank).

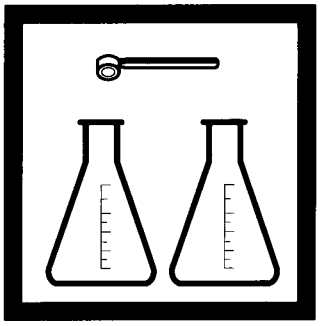
*Adapted from *Standard Methods for the Examination of Water and Wastewater*



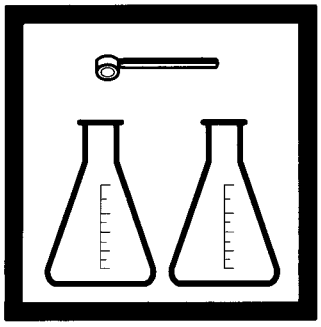
5. Measure 100 mL of sample into a second 500–mL erlenmeyer flask (the prepared sample).

Note: *To determine total selenium, perform a distillation. See* Distillation *at end of this procedure. Use this distillate in Step 5.*

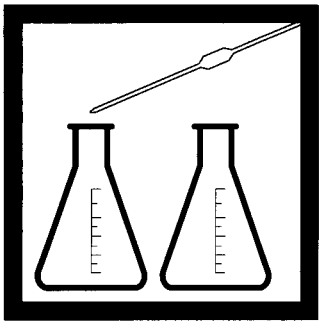
Note: *For proof of accuracy, use a 0.5 mg/L selenium standard solution (preparation given in the* Accuracy Check*) in place of the sample.*



6. Add a 0.2–g scoop of TitraVer Hardness Reagent to each flask. Swirl to mix.

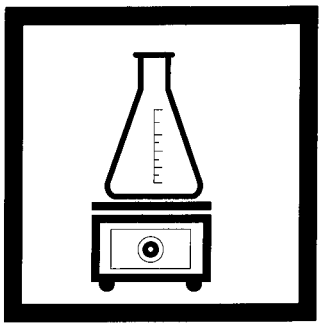


7. Add 0.05–g scoop of diaminobenzidine tetrahydrochloride to each flask. Swirl to mix.

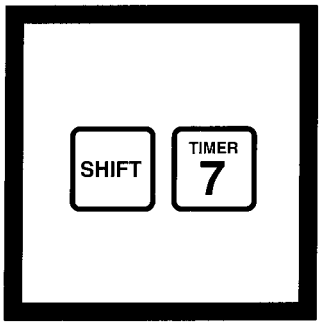


8. Add 5.0 mL of Buffer Solution, sulfate type, pH 2.0, to each flask. Swirl to mix.

Note: *If the sample has been distilled as described under* Distillation*, omit the Buffer Solution. Adjust the pH of the sample distillate to 2.7 (±0.2 pH) using 5 N Sodium Hydroxide Standard Solution. Adjust the demineralized water blank to the same pH value using 5.25 N Sulfuric Acid Standard Solution.*



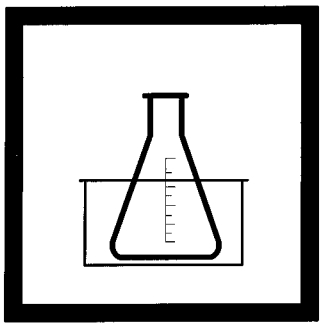
9. Heat each flask on a hot plate or over a flame, bringing the contents to a gentle boil.



10. Press: **SHIFT TIMER**

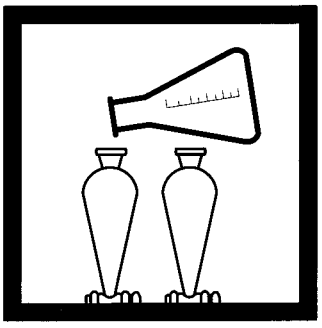
A 5–minute reaction period will begin.

Note: *A yellow color will develop if selenium is present.*



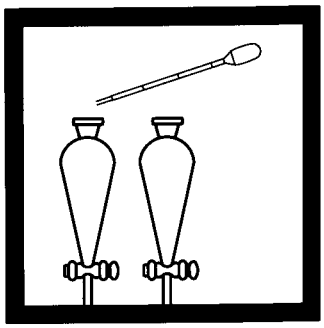
11. When the timer beeps, remove both flasks. Cool to room temperature using a water bath.

Note: *Do not boil more than one minute after the timer beeps.*

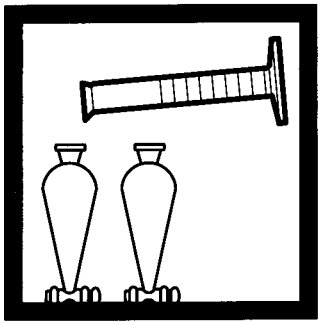


12. Transfer the contents of each flask to a separate 250–mL separatory funnel. Label the funnels "blank" and "prepared sample".

Note: *Transfer the blank to a separatory funnel labeled "blank."*

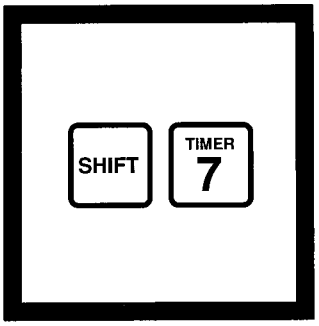


13. Add 2.0 mL of 12 N Potassium Hydroxide Standard Solution to each funnel using a calibrated 1.0–mL plastic dropper. Stopper. Shake each funnel to mix.



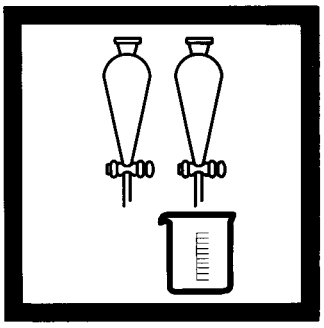
14. Add 30 mL of toluene to each funnel. Stopper. Shake each funnel vigorously for 30 seconds.

Note: Use toluene only with adequate ventilation.



15. Press: **SHIFT TIMER**

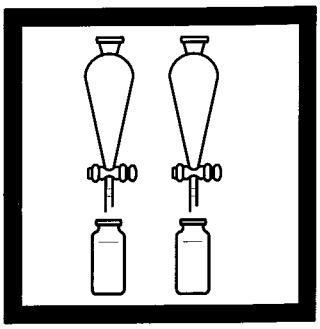
A 3–minute reaction period will begin.



16. When the timer beeps, the display will show:

mg/l Se

Drain the lower water layer from each funnel and discard.

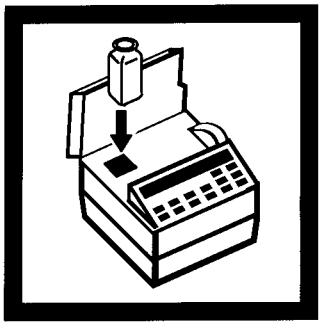


17. Insert a cotton plug into the delivery tube of each separatory funnel. Slowly drain the toluene into respective blank and sample cells. Stopper the sample cells.

Note: Do not wait more than 30 minutes after the timer beeps before completing Steps 18 through 20. One sample cell should be labeled, "blank."

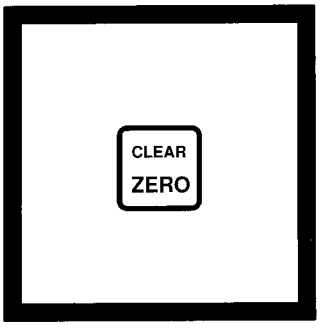
Note: Filtering the toluene through dry absorbent cotton will remove any water or suspended particles.

Note: The developed color is stable but should be read as soon as possible.



18. Place the blank into the cell holder. Close the light shield.

Note: The Pour–Thru Cell cannot be used with this procedure.



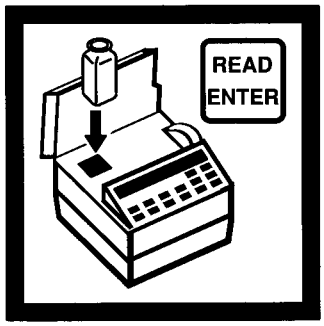
19. Press: **ZERO**

The display will show:

WAIT

then:

0.00 mg/l Se



20. Place the prepared sample in the cell holder. Close the light shield.

Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L selenium will be displayed.

Note: Acetone is a suitable solvent for removing toluene from glassware.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

DISTILLATION

a) Measure 500 mL of sample. Pour into a 1000–mL beaker.

b) Add 1 mL of Methyl Orange Indicator Solution. Stir with a glass rod.

c) Add 0.1 N Hydrochloric Acid Standard Solution drop-wise until the solution becomes pink. Then add an additional 2 mL.

d) Pipet 5.0 mL Calcium Chloride Solution into the beaker. Mix well.

e) Add 1–g/L Potassium Permanganate Standard Solution drop-wise until the solution is purple.

f) Place the beaker on a hot plate. Evaporate the solution to approximately 250 mL. Periodically add 1–g/L Potassium Permanganate Solution to keep the solution purple.

Note: *Any precipitate formed at this step is manganese dioxide and may be ignored.*

g) Cool the solution. Set up the distillation apparatus for the test by assembling the general purpose accessories as shown in the Hach Distillation Manual. Pour the treated sample solution into the distillation flask. Add a stirring bar.

h) Pipet 5.0 ml of 0.1 N Sodium Hydroxide Standard Solution. Set the heater switch to ON. Set the stir control to 5.

i) Turn on the water and adjust so a constant flow is maintained through the condenser. Set the heat control to 10.

j) Set the power switch to off when only a few milliliters are left in the distillation flask. The distillate in the erlenmeyer flask may be discarded.

k) Add 50 ml of 19.2 N Sulfuric Acid Standard Solution to the flask after the distillation flask has cooled. Add the contents of one Potassium Bromide Powder Pillow.

Note: *Perform this step under a hood.*

l) Fill a 250–mL beaker to the 75–mL mark with demineralized water. Place it under the drip tube. Elevate the beaker with a laboratory jack so the tube extends below the level of the water.

m) Add 1.0 mL of 30% Hydrogen Peroxide Solution to the flask. Turn the stir control to 5 and the heat control to 10.

n) Heat the distillation flask until the yellow bromine color is gone from the complete distillation apparatus, including the J–tube and condenser. Remove the beaker from under the drip tube.

o) Turn off the heater switch. When the J–tube and condenser have cooled, rinse them with demineralized water. Add the washings to the 250–mL beaker. Total volume in the beaker should be approximately 100 mL.

p) Add Phenol Solution drop-wise to the distilled sample to discharge the bromine color. (A white precipitate of tribromophenol will form.)

q) Allow the precipitate to settle. Using a dropper, test the solution for completeness of precipitation by adding 2 drops of Phenol Solution. If the solution is cloudy or white precipitate forms, residual bromine is still present. Continue to add Phenol Solution until a clear solution is obtained when the precipitation test is repeated.

r) Transfer the solution back to the 500–mL cylinder and dilute to volume with demineralized water. Mix well. The distillate is now ready for analysis.

SAMPLING AND STORAGE

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored for up to six months at room temperature. Correct the test result for volume additions *(see Correction for Volume Additions in Section I).*

ACCURACY CHECK

Standard Additions Method

a) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of a Selenium Standard Solution, 100 mg/L, to three 100–mL samples. Mix each thoroughly.

b) Analyze each sample as described above. The selenium concentration should increase 0.1 mg/L for each 0.1 ml of standard added.

c) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method

Prepare a 0.5–mg/L Se standard solution by pipetting 1.00 mL of a Selenium Standard Solution, 100 mg/L, into a 200–mL volumetric flask. Dilute to volume with demineralized water. Transfer 100 mL of the standard into a 500–mL erlenmeyer flask. Perform the test as described above.

PRECISION

In a single laboratory, using a standard solution of 0.6 mg/L Se and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.01 mg/L.

INTERFERENCES

There are no positive inorganic interferences with this method. Strong oxidizing agents such as iodine, bromine or chlorine can react with the indicator to give low results.

Manganese and up to 2.5 mg/L ferric iron will not interfere.

Interferences will be eliminated by following the distillation procedure.

SUMMARY OF METHOD

An EDTA masking agent is added to the sample to remove interferences, such as iron, prior to the test. The addition of a sulfate buffer adjusts the sample to the optimum pH of 1 to 2. Under these conditions, diaminobenzidine reacts with all selenium present as selenite (Se^{4+}) to give a yellow–colored piazselenol complex which is extracted and the color intensity measured colorimetrically. (Selenium present as Se^{2-} and Se^{6+} is not detected unless the sample is first distilled.)

REQUIRED REAGENTS

	Cat. No.
Selenium Reagent Set (45 tests)	22442–00
Includes: (1) 452–49, (1) 7062–22, (2) 230–32, (1)204–26, (1) 14470–49	

Description	Quantity Required Per Test	Units	Cat. No.
Buffer Solution, sulfate type, pH 2.0	10 mL	500 mL	452–49
Diaminobenzidine, tetrahydrochloride	0.1 g	5 g	7062–22
Potassium Hydroxide Standard Solution, 12 N	4 mL	100 mL	230–32
TitraVer Hardness Reagent	0.4 g	100 g	204–26
Toluene, ACS	60 mL	500 mL	14470–49
Water, demineralized	100 mL	4 L	272–56

REQUIRED APPARATUS

Balls, cotton, absorbent	1	100/pkg	2572–01
Cylinder, graduated, 100 mL	1	each	508–42
Cylinder, graduated, 50 mL	1	each	508–41
Dropper, 0.5 and 1.0 mL marks, glass	1	5/pkg	14197–05
Flask, erlenmeyer, 500 mL	2	each	505–49
Funnel, separatory, 250 mL	2	each	520–46
Pipet, volumetric, Class A, 5 mL	1	each	14515–37
Pipet Filler, safety bulb	1	each	14651–00
Ring, support, 83 mm (3 inches)	2	each	580–00
Spoon, measuring, 0.05 g	1	each	492–00
Spoon, measuring, 0.2 g	1	each	638–00
Squeezer, 0.25 to 1.00 mL, plastic dropper	1	10/pkg	21247–10
Stand, support, 127 X 203 mm (5 X 8")	2	each	563–00
Select one based on available voltage:			
Hot Plate, 3.5" diameter, 120 Vac	1	each	12057–01
Hot Plate, 3.5" diameter, 240 Vac	1	each	12057–02

OPTIONAL REAGENTS

Acetone, ACS	500 mL	14429–49
Calcium Chloride Solution	1000 mL	428–53
Hydrochloric Acid Standard Solution, 0.1 N	1000 mL	14812–53
Hydrogen Peroxide, 30%	500 mL	144–49
Methyl Orange Indicator Solution, 0.50 g/L	500 mL	148–49
Nitric Acid Solution, ACS	500 mL	152–49
Phenol Solution, 30 g/L	29 mL	2112–20
Potassium Bromide Powder Pillows	100/pkg	14819–99
Potassium Permanganate Standard Solution	100 mL	14164–42
Selenium Standard Solution, 100 mg/L	100 mL	12184–42
Sodium Hydroxide Standard Solution, 0.1 N	1000 mL	191–53
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	2450–32
Sulfuric Acid Standard Solution, 5.25 N	100 mL	2449–32
Sulfuric Acid Standard Solution, 19.2 N	100 mL	2038–42

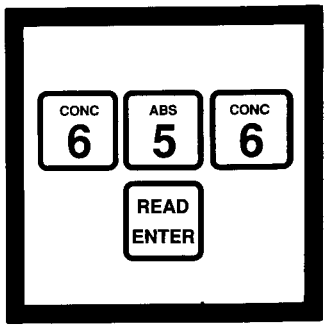
OPTIONAL APPARATUS

Beaker, 250 mL	each	500–46
Beaker, 1000 mL	each	500–53
Bottle, wash, 500 mL	each	620–11
Clippers, for opening pillows	each	968–00
Cylinder, graduated, 500 mL	each	508–49
Distillation apparatus accessories for general purpose	each	22653–00
Distillation apparatus heater, 115 V	each	22744–00
Distillation apparatus heater, 230 V	each	22744–02
Dropper, 1 mL mark	6/pkg	23185–06
Jack, laboratory	each	22743–00
pH Meter, EC10, portable	each	50050–00
Pipet, serological, 10 mL	each	532–38
Pipet, TenSette 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 TenSette Pipet	50/pkg	21856–96
Pipet, volumetric, 5.00 mL, Class A	each	14515–37
Pipet, volumetric, 1.00 mL, Class A	each	14515–35
Rod, stirring, glass	3/pkg	1770–01
Stoppers, for cells, hollow, No. 1	6/pkg	14480–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SILICA, HR (0 to 100.0 mg/L)

Silicomolybdate Method



1. Put the instrument in the constant–on mode and enter the stored program for high range silica (SiO_2).

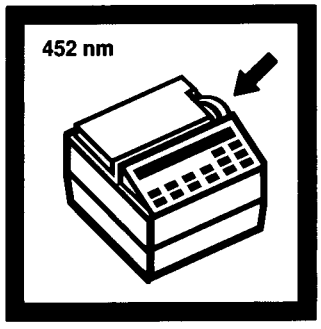
Press: **6 5 6 READ/ENTER**

The display will show:
DIAL nm to 452

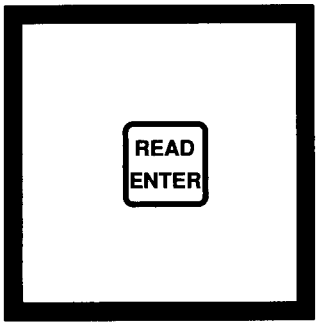
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: If samples cannot be analyzed immediately, see Sampling and Storage *following these steps.*

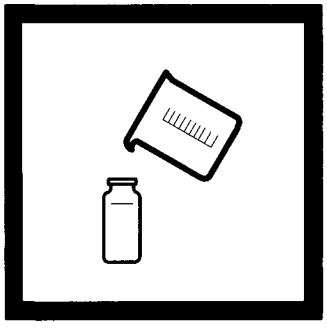


2. Rotate the wavelength dial until the small display shows:
452 nm



3. Press: **READ/ENTER**

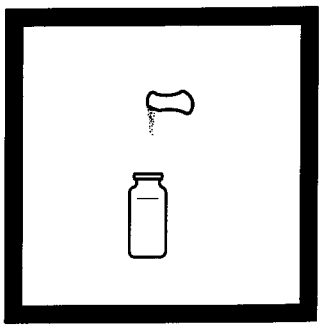
The display will show:
mg/l SiO_2 H



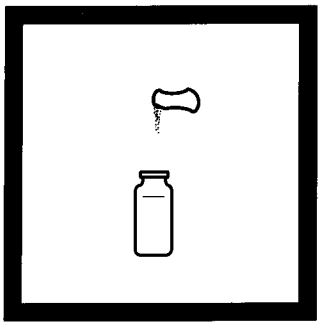
4. Fill a sample cell with 25 mL of sample.

Note: For proof of accuracy, use a 50 mg/L Silica Standard Solution (listed under Optional Reagents*) in place of the sample.*

Note: Sample temperature should be 15 to 25 °C (59 to 77 °F)

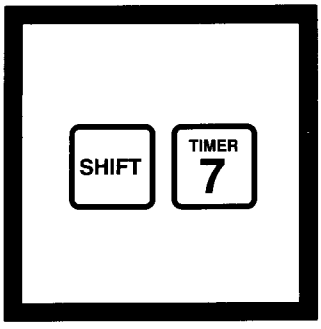


5. Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica. Swirl to mix.



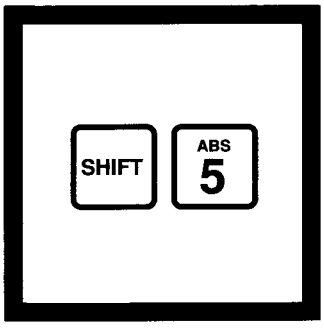
6. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Swirl to mix.

Note: Silica or phosphate will cause a yellow color to develop.



7. Press: **SHIFT TIMER**

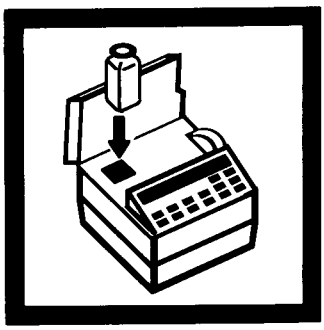
A 10–minute reaction period will begin.



8. Press: **SHIFT ABS**
The display will show:
Abs

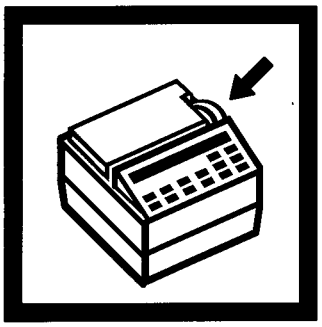
Press: **ZERO**

The display will show:
WAIT
then:
0.000 ABS



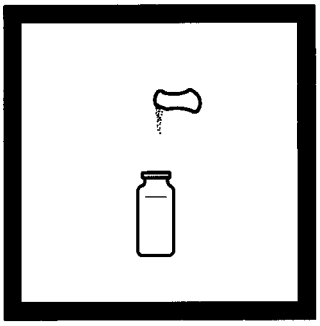
9. Place the capped square mixing bottle with the holmium trichloride solution into the cell holder and close the light shield.

Note: *Prepare holmium trichloride solution by adding 1 Holmium Trichloride Powder Pillow to 25 mL of demineralized water in a square mixing bottle. Cap and mix to dissolve. This capped solution may be kept indefinitely.*



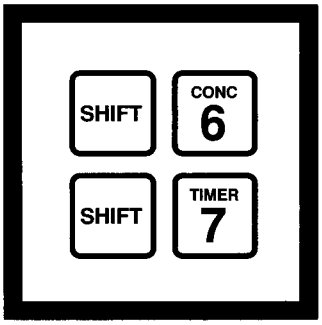
10. Starting at 460 nm, slowly turn the wavelength control dial to decrease the wavelength. Watch the display for the peak absorbance reading. This should occur between 450–454 nm. Without moving the wavelength dial, remove the holmium trichloride solution from the cell holder.

Note: *It is not necessary to reset the wavelength for more than one sample analysis or if the instrument is turned off. Resetting is necessary if the wavelength is changed. If the absorbance peak is outside the 452 ±1 nm range, the small display will flash "452 nm".*



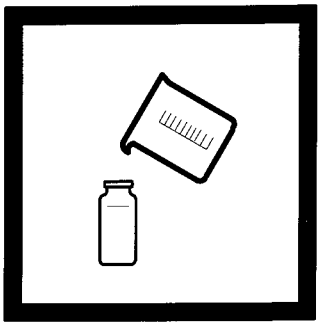
11. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

Note: *The yellow color due to phosphate will disappear.*



12. Press: **SHIFT CONC** and **SHIFT TIMER**

A 2–minute reaction period will begin.

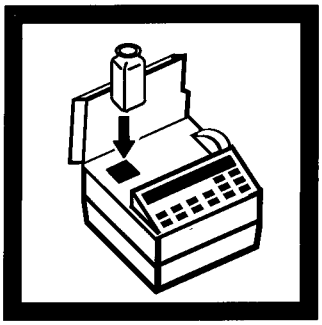


13. When the timer beeps, the display will show:

0.0 mg/l SiO_2 H

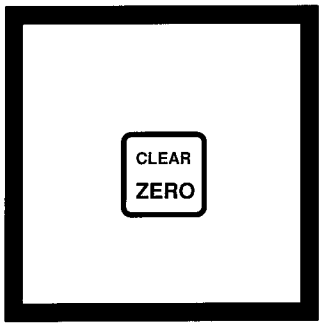
Fill a second sample cell with 25 mL of sample (the blank).

Note: *The display value may vary depending upon the wavelength setting determined in Step 10. The exact zero will be set in Step 15.*



14. Place the blank in the cell holder. Close the light shield.

Note: *The Pour–Thru Cell can be used with this procedure.*



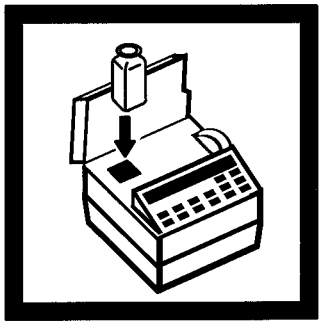
15. Press: **ZERO**

The display will show:

WAIT

then:

0.0 mg/l SiO_2 H



16. Within three minutes after the second timer beeps, place the prepared sample in the cell holder. Close the light shield. The results in mg/L silica will be displayed.

INSTRUMENT SETUP

For a DR/2000 with software versions before 2.0, the High Range Silica test calibration must be entered manually as the next available user–entered program. Follow the steps in the section on entering a calibration in the *DR/2000 Instrument Manual*. Store the method as follows:

nm = 452
Decimal = 000.0
Units = mg/l
Symbol = SiO_2 H
Timer 1 = 10.00 minutes
Timer 2 = 2.00 minutes

The calibration is first entered with 0.000 absorbance values for zero and #1 standards. To do this, do not place anything in the sample cell compartment. Begin by storing zero and #1 standard as concentrations of zero and 118.5, respectively, with nothing in the sample compartment. Accept 0.000 Abs as the value for all standards. Next, the absorbance value for #1 standard must be changed to the value given below.

Standard	Concentration	Absorbance
0	0	0
1	118.5	1.250

The method is now stored as an operator–programmed method with a method number between 950 and 999. Record the method number for future reference. Use this method number in place of # 656 in Step 1.

For a DR/2000 with software version 2.0 and 2.2 that do not have the High Range Silica method stored as method #656, enter the calibration as follows as a Hach–stored program.

1. Press: I/O

2. Press: SHIFT CONFIG METH

3. Press: CONC 6 ABS 5 CONC 6 READ ENTER

4. Within three seconds, press **SHIFT PROGRAM METHOD.** The display will show:

ENTER nm

If the display returns to the METHOD prompt, repeat the sequence.

5. Press: %T 4 ABS 5 EDIT 2

Note: If you make an error press SHIFT CLEAR and re–enter the number. When the number is correct, press READ/ENTER.

The display will show:

DECIMAL? 00.00

6. Use the arrow keys to correctly position the decimal point. For this method, press the **RIGHT ARROW** key once. The display will show:

DECIMAL? 000.0

7. When the decimal point is correctly positioned, press **READ/ENTER.** The display will show:

UNITS?

8. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. The display will show:

mg/l

9. With the proper unit of measure displayed, press **READ/ENTER.** The display will show:

SYMBOL?

10. Use the arrow keys to construct the correct symbol display. For this method, press the **DOWN ARROW** key repeatedly until you see:

mg/l s

11. Press **SHIFT** to make the "s" upper case. The display will show:

mg/l S

12. Press the **READ/ENTER** key to accept the capital "S." Continue to construct the display:

mg/l SiO_2 H

To enter the subscript 2, press 2 on the numeric keypad. Press **READ/ENTER** to accept subscript 2. The space is the character displayed after one press of the **DOWN ARROW** key.

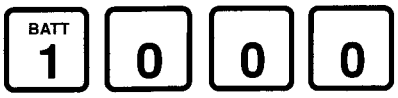
13. When the last character of the symbol is accepted with the **READ/ENTER** key, press **READ/ENTER** a second time to end display entry mode. The display will show:

TIMER?

14. This method has two timed periods, so press **SHIFT TIMER.** The display will show:

MM:SS TIME 1 ?

15. To enter the timer value of 10 minutes, press:



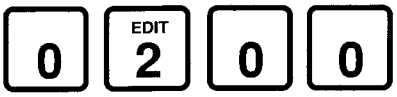
The display will read:

10:00 TIME 1?

16. Press **READ/ENTER** to accept the timer value. The display will show:

MM:SS TIME 2 ?

17. To enter the second timer value of 2:00 minutes, press:



The display will then read:

02:00 TIME 2?

18. Press **READ/ENTER** to accept the timer value. The display will read:

MM:SS TIME 3?

19. Press **READ/ENTER** to complete the timer entry. The display will show:

 # 1 Data

20. Enter the following 12 numbers as shown. Complete each number entry by pressing **READ/ENTER.**

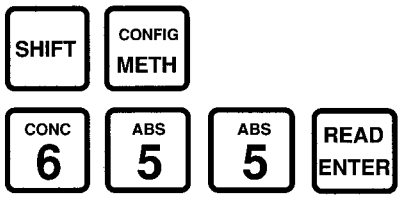
#1 Data	**0**
#2 Data	**30327**
#3 Data	**30327**
#4 Data	**30327**
#5 Data	**30582**
#6 Data	**30582**
#7 Data	**65535**
#8 Data	**65535**
#9 Data	**65535**
#10 Data	**2730**
#11 Data	**512**
CHECKSUM	**41224**

The final number is a check value which is used to determine if the data sequence was correctly entered. If an error is made during number entry, the display will return to the prompt for data number 1 and the entire sequence must be re–entered. If all numbers are correctly entered, the display will return to the method prompt:

METHOD #?

With the new method 656 successfully entered, block access to the now obsolete method number 655.

21. Press:



Within three seconds, press:

Access to method 655 is now blocked.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to seven 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 Pillow, 250 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of the standard to three 25–mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method

To check the accuracy of the method, use the Silica Standard Solutions, 10 and 25 mg/L as SiO_2, listed under *Optional Reagents*. Analyze according to the above procedure using demineralized water as the blank.

PRECISION

In a single laboratory, using a standard solution of 50.0 mg/L SiO_2 and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.45 mg/L silica.

INTERFERENCES

Color and turbidity interferences are eliminated by zeroing the instrument with the original water sample.

Sulfides and large amounts of iron interfere.

At levels of 50 mg/L phosphate (PO_4^{3-}), interference is not a problem. At 60 mg/L PO_4^{3-}, an interference of minus 2% is observed. At 75 mg/L, the interference is minus 11%.

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.
Due to wavelength sensitivity of the test, holmium trichloride is used to reproducibly set the wavelength required.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set (100 tests)	22443–00
Includes: (2) 1042–66, (1)14548–99, (2) 1041–66	

Description	Quantity Required Per Test	Units	Cat. No.
Acid Reagent Powder Pillows for High Range Silica	1	50/pkg	1042–66
Citric Acid Powder Pillows	1	100/pkg	14548–99
Holmium Trichloride Powder Pillows	1	10/pkg	23432–67
Molybdate Reagent Powder Pillows for High Range Silica	1	50/pkg	1041–66
Water, demineralized	25 mL	4 L	272–56

REQUIRED APPARATUS

Bottle, square mixing, 25–mL mark	1	each	17042–00
Cap, bottle	1	each	21667–06
Clippers, for opening powder pillows	1	each	968–00

OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L	500 mL	1403–49
Silica Standard Solution, 50 mg/L	237 mL	1117–31
Silica Standard Solution Pillows, 250 mg/L as SiO_2	16/pkg	14244–10
Sodium Bicarbonate, ACS	454 g	776–01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270–32

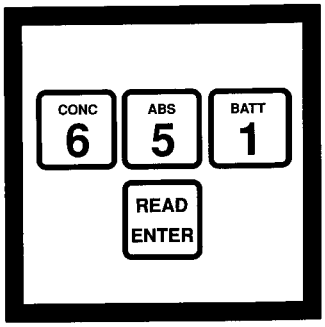
OPTIONAL APPARATUS

Clippers, shears, 7 1/4"	each	23694–00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 Pipet	50/pkg	21856–96
Pour–Thru Cell Assembly	each	45215–00
Standard Methods for the Examination of Water and Wastewater	each	22708–00
Thermometer, – 20 to 105 °C	each	1877–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SILICA, LR (0 to 1.600 mg/L)

Heteropoly Blue Method*



1. Enter the stored program number for low range silica (SiO_2).

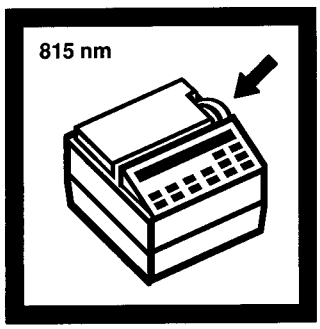
Press: **6 5 1 READ/ENTER**

The display will show:
DIAL nm TO 815

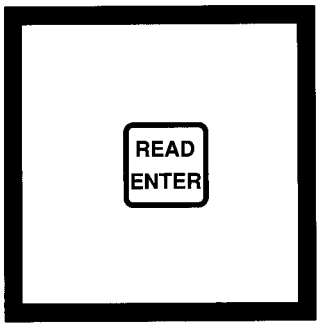
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: If samples cannot be analyzed immediately, see Sampling and Storage *following these steps.*

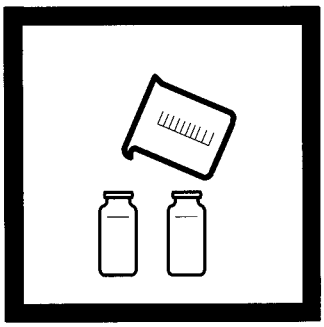


2. Rotate the wavelength dial until the small display shows:
815 nm



3. Press: **READ/ENTER**

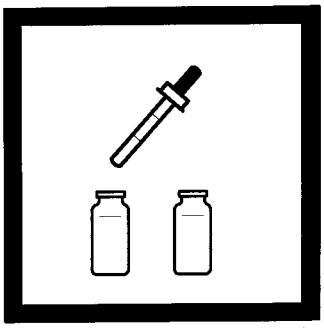
The display will show:
mg/l SiO_2 L



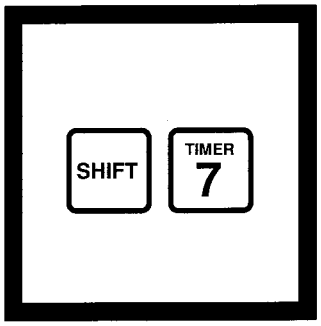
4. Fill two sample cells to the 25–mL line with sample.

Note: For proof of accuracy, use a 0.5-mg/L Silica Standard Solution in place of the sample (see Optional Reagents).

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



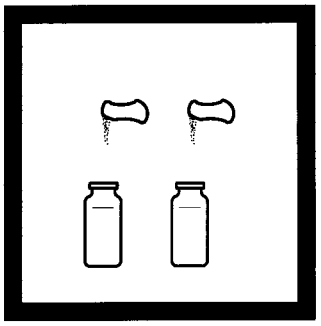
5. Add 0.5 mL of Molybdenum 3 Reagent to each sample cell. Swirl to mix.



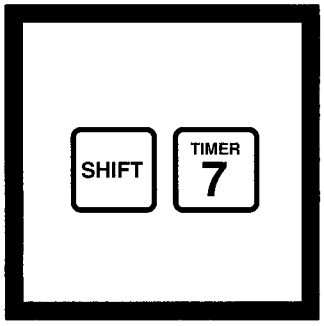
6. Press: **SHIFT TIMER**

A 4–minute reaction period will begin.

Note: Reaction time depends on sample temperature. The 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.



7. When the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.



8. Press: **SHIFT TIMER**

A 1–minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.

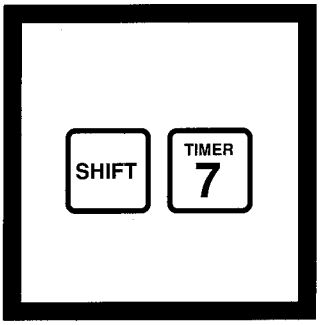
Note: Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 2 minutes. If the sample temperature is 30 °C (86 °F), wait 30 seconds

9. When the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cell. Swirl to mix. This is the prepared sample.

Note: If very low levels of silica are being determined, use the Pour–Thru Cell. Also, use 0.5 mL of Amino Acid F Solution instead of Amino Acid F Powder Pillows; see Reagent Preparation following these steps.

Note: The sample cell without the Amino Acid F Reagent is the blank.

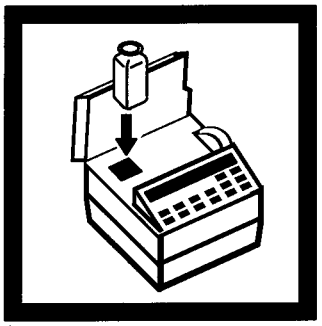
Note: A blue color will develop if silica is present.



10. Press: **SHIFT TIMER**

A 1–minute reaction period will begin.

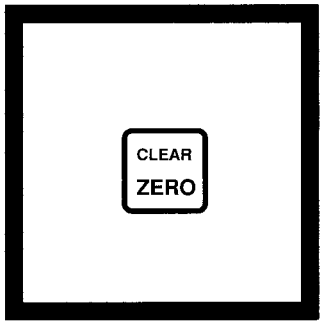
Note: The Pour–Thru Cell is recommended for this procedure.



11. When the timer beeps, the display will show:

mg/l SiO_2 L

Place the blank (solution without Amino Acid F Reagent) into the cell holder. Close the light shield.



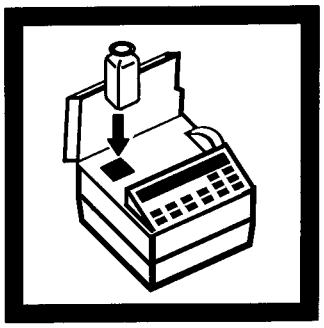
12. Press: **ZERO**

The display will show:

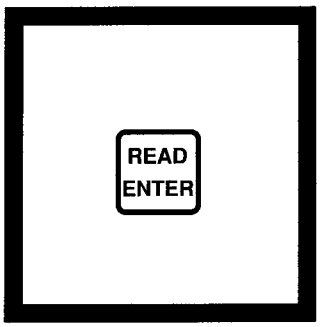
WAIT

then:

0.000 mg/l SiO_2 L



13. Place the prepared sample into the cell holder. Close the light shield.



14. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L silica will be displayed.

Note: In the constant on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

INSTRUMENT SETUP

For a DR/2000 with software version 3.0 only – enter the calibration as follows as a Hach Update. (Stored program 650 has been replaced with number 651).

1. Press: I/O

2. Press: SHIFT, CONFIG METH

The display will show:
MOMENTARY or CONSTANT ON

3. Press the **UP ARROW** twice to display HACH UPDATE. Press **READ/ENTER**. The display will show:
ENTER #:

4. Press: CONC 6, ABS 5, BATT 1, READ ENTER

The display will show;
P651 ENTER nm

5. Press: – 8, BATT 1, ABS 5

Note: If you make an error press SHIFT CLEAR and re–enter the number. When the number is correct, press READ/ENTER.

The display will show:
P651 DECIMAL? 00.00

6. Use the arrow keys to correctly position the decimal point. For this method, press the **UP ARROW** key once. The display will show:
P651 DECIMAL? 0.000

7. When the decimal point is correctly positioned, press **READ/ENTER.** The display will show:
P651 UNITS?

8. Use the arrow keys to select the appropriate unit of measure. For this method, press the **DOWN ARROW** key twice. The display will show:
P651 mg/l

9. With the proper unit of measure displayed, press **READ/ENTER.** The display will show:
P 651 SYMBOL?

10. Construct the following symbol, one character at a time, by scrolling for the proper character, using the **SHIFT** key to capitalize, subscript or superscript if necessary, and accepting with the **READ/ENTER** key. Use three spaces between the subscript 2 and L; i.e.,
P651 mg/l SiO_{2} _ _ _ L

When the last character of the symbol is accepted with the **READ/ENTER** key, press **READ/ENTER** again

to accept the entire symbol. The display will show:
P651 TIMER

11. This method has three timed periods, so press **SHIFT TIMER.** The display will show:
P651 MM:SS TIME 1?

12. To enter the timer value of 4 minutes, press:

The display will read:
P651 04:00 TIME 1?

13. Press **READ/ENTER** to accept the timer value. The display will show:
P651 MM:SS TIME 2?

14. To enter the second timer value of 1:00 minutes, press:

The display will then read:
P651 01:00 TIME 2?

15. Press **READ/ENTER** to accept the timer value. The display will show:
P651 MM:SS TIME 3?

16. To enter the third timer value of 1:00 minutes, press:

The display will then read:
P651 01:00 TIME 3?

17. Press **READ/ENTER** to accept the timer value. The display will show:
P651 MM:SS TIME 4?

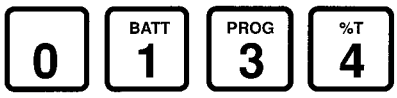
18. Press **READ/ENTER** twice to accept the timer values. The display will show:
 # 0 Standard

19. Press **READ/ENTER** to display the zero data pair. The display will show:
0.000 Abs 0.000 mg/l

20. Press **READ/ENTER.** The display will show:
 # 1 STANDARD

21. Press **READ/ENTER.** The display will prompt for entry of the first concentration data point:
 # 1 0.000 mg/l

22. Enter the first concentration by pressing:



The display will show:
 # 1 0.134 mg/l

23. Press **READ/ENTER.** The display will prompt for entry of the first absorbance value:
 # 1 0.000 Abs

24. Enter the first absorbance point by pressing:

The display will show:
 # 1 0.125 Abs

25. Press **READ/ENTER.** The display will show the first data pair:
 # 1 0.125 Abs 0.134 mg/l

26. If the data pair is correctly displayed, press **READ/ENTER** to accept the #1 Standard values. The display will show:
 # 2 STANDARD

27. In the same manner described above, enter the second data pair with values:
 # 2 1.760 mg/l
 # 2 1.516 Abs

When the data pair is correctly displayed, press **READ/ENTER** to accept the values. The display will show:
 # 3 STANDARD

28. Press **SHIFT, READ/ENTER** to complete data point entry. The display will show:
#:

29. Enter validation number 5822. The display will show:
#: 5822

30. Press **READ/ENTER.** The display will show:
COMPLETED

then:
P651 mg/l SiO_2 _ _ _ L

Note: If the display shows

INCORRECT #

and then prompts for the validation number, an error has been made. Make sure the validation number is correct. If it is, it is necessary to press METH, accept ABORT? with the READ/ENTER key and re-enter the entire method beginning with Step 2.

If the method entry is successful, the old method number 950 should be blocked from use. Proceed to step 31.

31. Press **SHIFT, CONFIG** and use the arrow keys to scroll to HACH UPDATE.

32. With the HACH UPDATE in the display, press **READ/ENTER** and use the arrow keys to display:

ERASE #:

33. Press: CONC 6 | ABS 5 | 0 | READ ENTER

The display will show:

P650 #:

34. Press: ABS 5 | ABS 5 | TIMER 7 | PROG 3

The display will show:

P650 #: 5573

35. Press **READ/ENTER.** The display will show

COMPLETED

followed by the ERASE #: prompt.

Method 650 has now been blocked.

36. Press **METH.** The instrument is ready for use with method 651.

ALTERNATIVE PROCEDURES

The DR/2000 may be calibrated to use the same reagents used in Hach's Silica Analyzers (651C, 1234D, and 31201 Trace Pump). The Alternative Procedures Table lists the reagents and sample volumes to be used, plus appropriate reaction periods for room-temperature samples.

Prepare an equivalent calibration as follows:

a) Store the calibration in the instrument memory using the procedure in the *Operation* section of the *DR/2000 Instrument Manual.* Store units as mg/l SiO_2, the decimal position as 0.000, the wavelength as 810 nm, and the appropriate timer intervals from the *Alternative Procedures Table.*

b) Prepare standards of 0.2, 0.4, 0.6, 0.8 and 1.0 mg/L SiO_2 by diluting 1, 2, 3, 4, and 5 mL of Silica Standard Solution, 50 mg/L SiO_2, to 250 mL with silica-free demineralized water.

c) Choose the appropriate column from the *Alternative Procedures Table* to match your analyzer. Prepare the samples by following the sequence of steps listed in the table. First calibrate the DR/2000 at a silica concentration of zero. Prepare this sample using 50 mL of silica-free demineralized water with the specified reagent volumes and corresponding reaction times. Now, using 25 mL of this prepared sample, measure and enter the sample absorbance as prompted by the instrument.

ALTERNATIVE PROCEDURE TABLE

Step	Series 5000 Analyzer	31201 (Trace Pump)	651C	Analyzer 1234D
1. Your stored program number	9 ? ?	9 ? ?	9 ? ?	9 ? ?
2. Sample amount needed	45 mL	50 mL	50 mL	50 mL
3. Measurement wavelength	810 nm	810 nm	810 nm	810 nm
4. Add Molybdate 3 (1995*)	1.0 mL	1.0 mL	0.5 mL	1.0 mL
5. Wait	5 min	5 min	5 min	5 min
6. Add Citric Acid (22542*) (23470**)	1.0 mL	1.0 mL	—	—
or Sodium Citrate (14908*)	—	1.0 mL	—	—
7. Wait	1 min.	1 min	—	1 min
8. Add Amino Acid (1934*) or	—	—	1.0 mL	1.0 mL
Amino Acid F (22541) (23531**)	1.0 mL	1.0 mL	—	—
9. Wait	8 min	15 sec.	8 min	8 min
10 Zero DR/2000				
11 Measure Absorbance				

*Hach Catalog Number **Hach Catalog number for Series 5000 reagent

Next prepare 50 mL of the first standard (0.2 mg/L) using the same procedure. Enter this concentration into the instrument. Again, using 25 mL of this standard, measure and enter the absorbance as prompted by the instrument. Analyze, measure and enter the remaining standards. For best results at low concentrations, use the Pour–Thru Cell.

d) To analyze your samples using this calibration, enter the stored program number you selected at the **METHOD #** prompt. The calibration does not need to be re–entered for each new lot of reagent.

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 7 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 Low Range Silica Standard Solution Pillow, 50 mg/L SiO_2.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 25–mL samples. Mix thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions in Section I for more information.

Standard Solution Method

Silica Standard Solutions of 0.500 mg/L and 1.000 mg/L SiO_2 are listed under *Optional Reagents*. Use these in place of the sample and analyze according to the above procedure.

PRECISION

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a DR/2000, a single operator obtained a standard deviation of ±0.0067 mg/L silica.

INTERFERENCES

Color and turbidity interferences are eliminated by zeroing the instrument with the original sample.

Sulfide and large amounts of iron interfere.

Phosphate does not interfere at levels less than 50 mg/L PO_4. At 60 mg/L, an interference of – 2% occurs. At 75 mg/L the interference is –11%.

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.

REAGENT PREPARATION

To prepare Amino Acid F Reagent Solution, dissolve Amino Acid F Reagent Powder in 1.0 N Sodium Hydroxide Standard Solution at a ratio of 10 grams added to 100 mL. 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. Addition of citric acid destroys the phosphate complexes. An amino acid is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

REQUIRED REAGENTS

	Cat. No.
Low Range Silica Reagent Set (100 tests)	23468–00
Includes: (1) 22538–69, (2) 14548–99 (1) 1995–32	

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid F Reagent Powder Pillows	1 pillow	100/pkg	22538–69
Citric Acid Powder Pillows	2 pillows	100/pkg	14548–99
Molybdate 3 Reagent	1.0 mL	100 mL MDB	1995–32

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968–00

OPTIONAL REAGENTS

Amino Acid F Reagent Powder (for Trace Pump Analyzer)	410 g	22833–55
Silica Standard Solution, 0.5 mg/L SiO_2	3.78 L	21008–17
Silica Standard Solution, 1 mg/L SiO_2	500 mL	1106–49
Silica Standard Solution, 50 mg/L SiO_2	237 mL	1117–31
Silica Standard Solution Pillows, 50 mg/L as SiO_2	16/pkg	1117–10
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

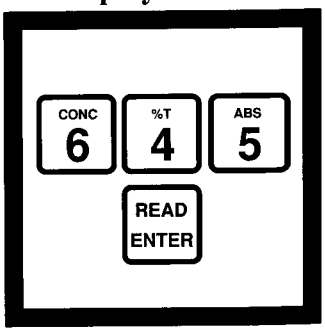
OPTIONAL APPARATUS

Bottle, 118 mL, polyethylene, oblong	6/pkg	23184–06
Dropper, 0.5– & 1.0–mL marks	6/pkg	23185–06
Flask, volumetric, 250 mL	each	14574–46
Pipet, serologic, 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, volumetric, Class A, 1.00 mL	each	14515–35
Pipet, volumetric, Class A, 2.00 mL	each	14515–36
Pipet, volumetric, Class A, 3.00 mL	each	14515–03
Pipet, volumetric, Class A, 4.00 mL	each	14515–04
Pipet, volumetric, Class A, 5.00 mL	each	14515–37
Pipet Filler, safety bulb	each	14651–00
Pour–Thru Cell Assembly	each	45215–00
Sample Cell, w/ 25–mL mark	pair	13537–00
Standard Methods for the Examination of Water and Wastewater	each	22708–00
Thermometer, – 20 to 105 °C	each	1877–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SILICA, ULTRA LOW RANGE (0 to 999.9 µg/L)

Heteropoly Blue Method*



1. Enter the stored program for ultra low range silica.

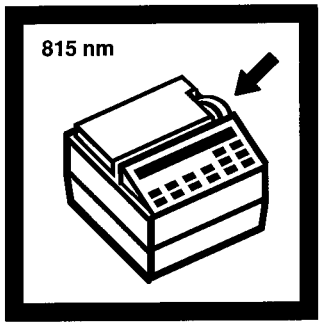
Press: **6 4 5 READ/ENTER** for software versions 3.0 or higher.

Press: **9?? READ/ENTER** for software versions 2.2 and below.

The display will show:
DIAL nm to 815

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

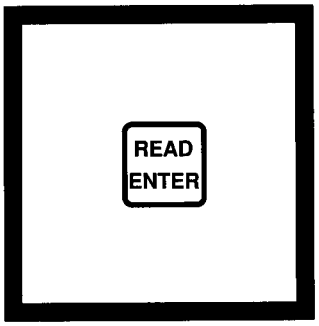
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



2. Rotate the wavelength dial until the small display shows:
815 nm

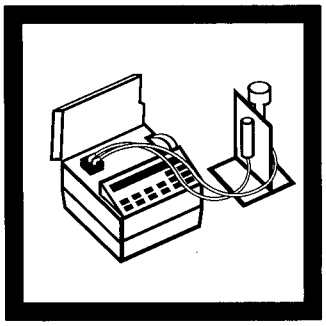
Note: *For DR/2000 users with software versions 2.2 and less, see the* Instrument Setup *section for calibration information.*

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps.*



3. Press: **READ/ENTER**

The display will show:
µg/l SiO_2 ULR

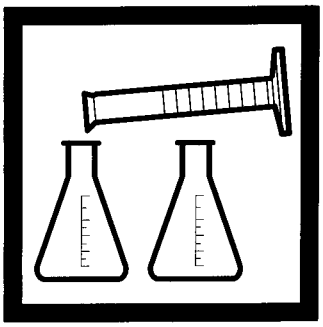


4. Install the Pour–Thru Cell and flush with 50 mL of demineralized water.

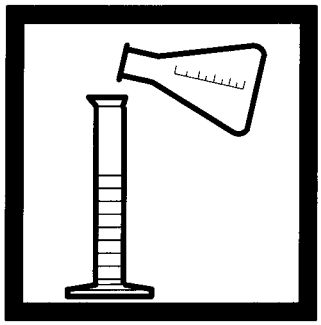
Note: *See* Analysis Labware *for more information on cleaning labware.*

Note: *The Pour–Thru Cell must be used. See* Summary of Method *section.*

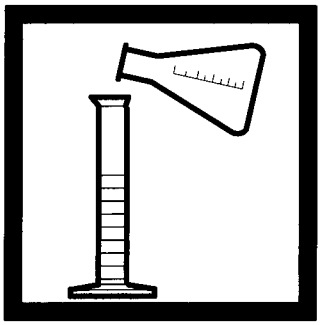
*Adapted from *Standard Methods for the Examination of Water and Wastewater*



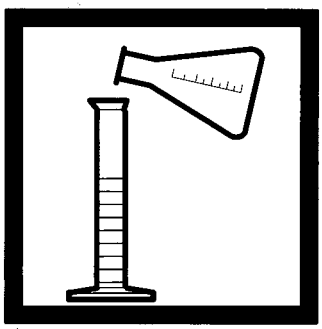
5. Fill two clean 250–mL erlenmeyer flasks to overflowing with the sample.



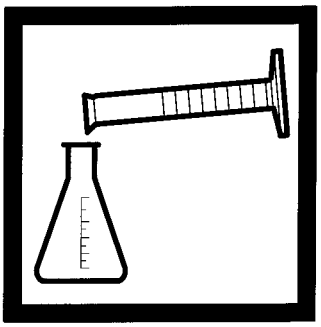
6. Fill a clean 50–mL plastic graduated mixing cylinder with sample from one of the flasks and then discard the cylinder contents.



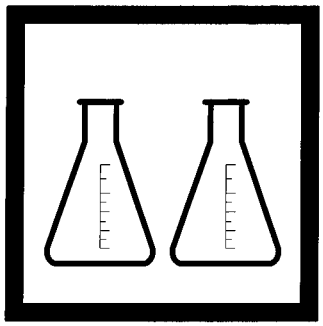
7. Repeat the rinsing of the cylinder three times from the same sample flask, discarding each rinse.



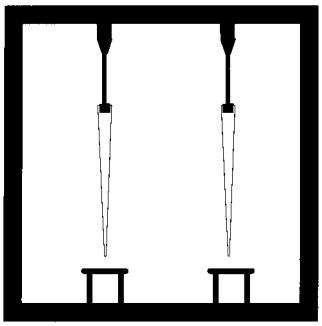
8. Fill this rinsed cylinder to the 50–mL mark with sample from the same flask, discarding any remaining sample in the flask.



9. Pour the contents of the 50–mL cylinder back into the original flask.

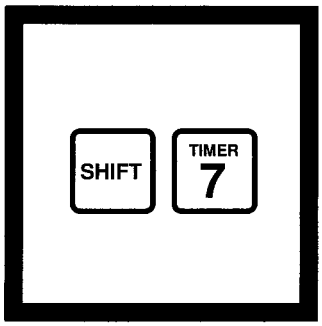


10. Repeat Steps 6–9 for the second flask containing sample, then continue with Step 11.



11. Using a TenSette Pipet, add 1.0 mL of Molybdate 3 Reagent to each flask. Swirl to mix.

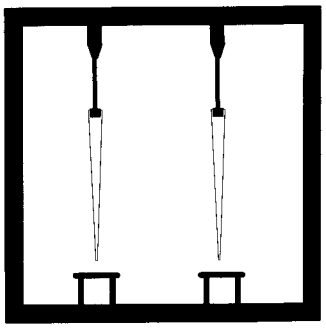
Note: *The TenSette Pipet is recommended for convenient reagent addition. An all–plastic 1.0–mL dropper is also available* (See Optional Apparatus).



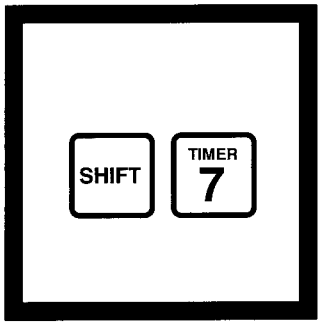
12. Press: **SHIFT TIMER**

A 4–minute reaction period will begin.

Note: *Reaction time depends on sample temperature. The 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.*



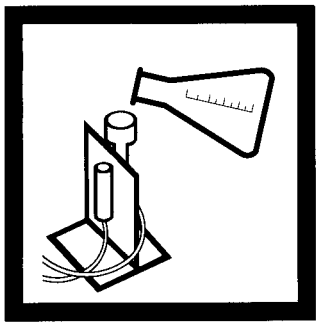
13. When the timer beeps, add 1.0 mL of Citric Acid F Reagent to each flask. Swirl to mix.



14. Press: **SHIFT TIMER**

A 1–minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.

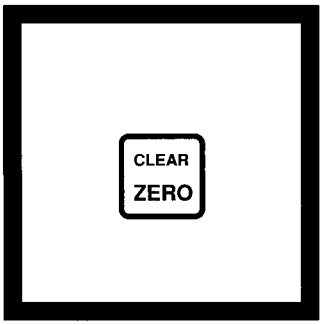
Note: *Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 2 minutes. If the sample temperature is 30 °C (86 °F), wait 30 seconds.*



15. When the timer beeps, the display will show:

µg/l SiO_2 ULR

Pour the contents of the first flask through the Pour–Thru Cell.



16. After the flow has stopped,

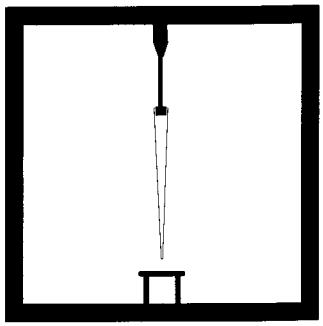
Press: **ZERO**

The display should show:

WAIT

then:

0 µg/l SiO_2 ULR

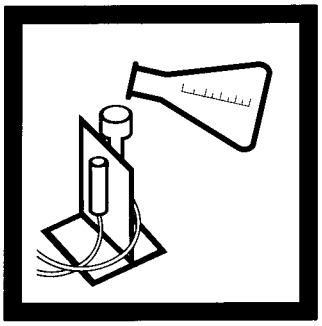


17. Add 1.0 mL of Amino Acid F Reagent Solution or pour the contents of one ampule of Amino Acid F Reagent into the second flask. Swirl to mix.

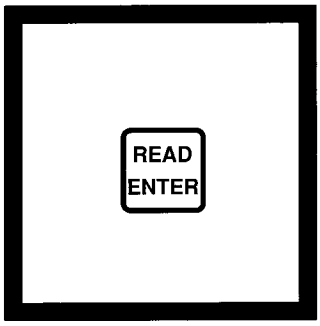
Note: *For greatest accuracy use a TenSette pipet to dispense 1.0 mL from the ampul.*

Note: *A faint blue color will develop if silica is present.*

Note: *See* Reagent Preparation *section for more information.*



18. Wait 15 seconds for color formation, then pour the contents of the second flask through the Pour–Thru Cell.



19. After the flow has stopped,

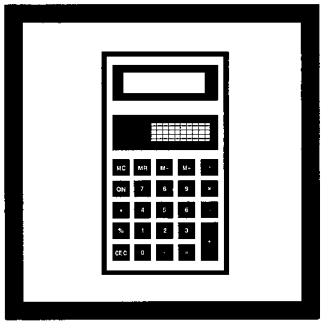
Press: **READ/ENTER**

The display will show:

WAIT

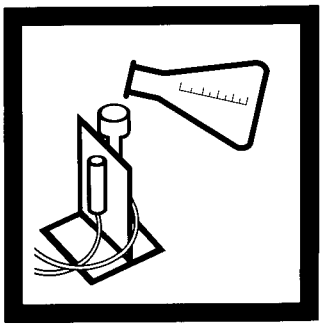
then the result in µg/L of SiO_2 will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*



20. Subtract the reagent blank value found on the Molybdate 3 analyzer reagent container from the value obtained in Step 19.

Note: *Reagent blank values printed on analyzer reagent containers vary because the reagent dilutions vary according to the instrument. For this method, use the 1234D analyzer reagent blank value for a 3.78 L volume of Molybdate 3 Reagent (Cat No. 1995–17). For a Series 5000, 2.9–L volume of Molybdate 3 Reagent (Cat. No. 1995–03), multiply the reagent blank on the label by 1.09. For 100–mL (Cat No. 1995–32) and 1–L (Cat. No. 1995–53) reagent volumes, use the values on the bottles.*



21. Flush the Pour–Thru Cell with at least 50 mL of demineralized water immediately after use.

Note: *Protect the Pour–Thru Cell from contamination by inverting a small beaker over the top of the glass funnel.*

INSTRUMENT SETUP

For DR/2000s with software versions 2.2 or less, enter the following calibration as an operator–programmed calibration. Follow these steps in the *Operation* section of the *DR/2000 Instrument Manual.* Store the method as follows:

nm = 815
Decimal = 0000.
Units = µg/l
Symbol = SiO_2 ULR
Timer 1 = 04:00
Timer 2 = 01:00

The calibration is first entered with 0.000 absorbance values for the #0 and #1 standard. To do this, do not place anything in the sample cell compartment. Begin by storing zero and #1 standard as concentrations shown in the table below with nothing in the sample compartment.

Accept 0.000 Abs as the value for all standards. Store the calibration by pressing **SHIFT READ/ENTER**. Next the value for the #1 standard must be changed to the value given below.

Standard	Concentration	Absorbance
0	0	0
1	1000	0.813

The method is now stored as an operator–programmed method with a method number between 950 and 999. Record the method number for future reference.

REAGENT PREPARATION

Amino Acid F Reagent Solution is available in either 100 mL bottles or a package of 20 unit dose ampules. The bottled reagent has a limited shelf life after opening due to air oxidation. The ampuled reagent is sealed under argon and is more stable (greater than 1 year). Instability is evidenced by reduced sensitivity at high silica concentrations. Check the bottled reagent on a routine basis by performing the test on a 1000 µg/L silica standard (Cat. No. 1106). If the concentration result is less than 950 µg/L, use a fresh bottle of Amino Acid F Reagent Solution.

Prepare larger or smaller volumes of Amino Acid F Reagent Solution by dissolving Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL. These reagents are available as the Amino Acid F Reagent Package listed under Optional Reagents. This prepared solution has limited stability; test routinely with the 1000 µg/L silica standard as above.

Users running a large number of analyses may wish to obtain one of the optional variable volume dispensers for each reagent. The variable volume dispensers are listed under *Optional Apparatus* at the end of this method. This dispenser is made of fluoropolymer plastic. Do not use a dispenser with a glass bottle or parts.

ANALYSIS LABWARE

All containers used in this test must be cleaned thoroughly to remove any traces of silica. If possible, use plastic containers for all analysis and storage because glass can contaminate the sample with silica. Small bottles or flasks with screw–type closures work well. Clean containers by normal means (do not use phosphate detergents), then rinse with high quality demineralized water with low–level silica concentration. Soak for 10 minutes with a 1:50 dilution of Molybdate 3 Reagent in low–level silica water. Rinse well with low–level silica water or the sample before use. Keep containers tightly closed when not in use. Dedicate these containers for silica analysis only. Fill the Pour–Thru Cell with this same mixture of Molybdate 3 and water, and let stand for several minutes before use. Rinse with low–level silica water.

CLEANING THE POUR–THRU CELL

The Pour–Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of ammonium hydroxide, followed by several demineralized water rinses. Cover the glass funnel when it is not in use.

SAMPLING AND STORAGE

The sampling procedure in Steps 5–9 has proven effective in harsh and dirty testing environments. It is used in this procedure to improve accuracy.

Use only plastic containers with tight–fitting closures. Glass containers can contaminate the sample with silica. Soak sampling containers with solution of one part Molybdate 3 Reagent to 50 parts low–level silica demineralized water. Fill completely and let stand for several hours. Rinse thoroughly with low–level silica water, drain and close. Repeat this cleaning periodically.

Allow the sample stream to flow for 1–2 minutes before collection. Do not adjust the flow during the sampling period— this may introduce particulates. Rinse the container well with sample before collecting the portion for analysis. Analyze as soon as possible.

ACCURACY CHECK

a) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of either a 1–mg/L or 10–mg/L Silica Standard Solution to three 50–mL samples, respectively.

b) Analyze each sample as described above. The silica concentration should increase 2.0 µg/L for each 0.1 mL of 1–mg/L standard. The silica concentration should increase 20.0 µg/L for each 0.1 mL of 10–mg/L standard.

PRECISION

In a single laboratory, using blanks and standard additions of 5 µg/L silica, a single operator obtained a standard deviation of less than 1 µg/L silica.

INTERFERENCES

Color and turbidity interferences are eliminated by zeroing the instrument with the original sample.

Phosphate does not interfere appreciably at levels less than 50 mg/L PO_4^{3-}. Sulfides and large amounts of iron interfere.

SUMMARY OF METHOD

A number of modifications are necessary to adapt the Low Range Silica method for analyzing trace levels in the Ultra Low Range method. It is absolutely necessary to use the one–inch Pour–Thru Cell and liquid reagents. The Pour–Thru Cell increases the reproducibility of the optics and reduces the instability of the readings that result with moveable sample cells. Liquid reagents contribute to more reproducible readings and lower blanks by eliminating slight turbidity that may remain when using powdered reagents. In addition, the liquid reagents are used with Hach process analyzers for continuous silica measurement.

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. An amino acid is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

SILICA, ULTRA LOW RANGE, continued

REQUIRED REAGENTS

Description	Cat. No.
ULR Silica Reagent Set (using Amino Acid F solution; 100 tests)	25535–00
Includes: (2) 1995–32, (2) 22542–32, (1) 23864–42	
ULR Silica Reagent Set (using Amino Acid F ampules; 40 tests)	25814–00
Includes: (1) 1995–32, (1) 22542–32, (2) 23864–20	

	Quantity Required Per Test	Units	
Amino Acid F Reagent Solution	1.0 mL	100 mL	23864–42
or			
Amino Acid F Reagent Ampuls	1 each	20/pkg	23864-20
Citric Acid F Reagent	2 mL	100 mL	22542–32
Molybdate 3 Reagent	2.0 mL	100 mL	1995–32

REQUIRED APPARATUS

Cylinder, graduated, 50 mL, poly	1	each	1081–41
Flask, erlenmeyer, 250 mL, PMP, w/ cap	2	each	20898–46
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700–01
Pipet Tips, for 19700–01 Pipet	1	50/pkg	21856–96
Pour–Thru Cell Assembly Kit	1	each	45215–00

OPTIONAL REAGENTS

Amino Acid F Reagent Package:		23531–03
Amino Acid F Reagent Powder	308 g	
Amino Acid F Reagent Solvent	2.7 L	
Ammonium Hydroxide, ACS	500 mL	106–49
Citric Acid F Solution	100 mL MDB	22542–32
Molybdate 3 Reagent	2.9 L	1995–03
Molybdate 3 Reagent	3.78 L	1995–17
Molybdate 3 Reagent	100 mL MDB	1995–32
Silica Standard Solution, 1 mg/L SiO_2	500 mL	1106–49
Silica Standard Solution, 10 mg/L SiO_2	500 mL	1043–49
Sodium Bicarbonate, ACS	454 g	776–01
Sulfuric Acid Standard Solution, 1.00 N	1000 mL	1270–53

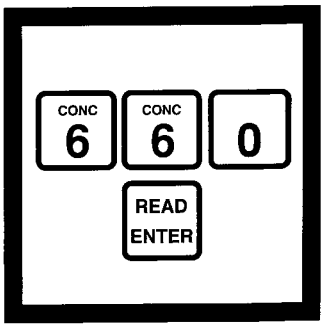
OPTIONAL APPARATUS

Ampul Breaker	each	24846-00
Beaker, polypropylene, 100 mL	each	1080–42
Bottle, 1000 mL, for use w/ variable volume dispenser	6/pkg	7137–54
Dispenser, variable volume, 1.0–5.0 mL	each	25631–37
Flask, erlenmeyer, 250 mL, PMP w/ cap	4/pkg	20898–76
Handbook, *Standard Methods for the Examination of Water and Wastewater,* 18th Ed.	each	22708-00
Measuring Dropper, squeezer type, 1 mL	10/pkg	21247–10
Pipet Tips, for 19700–01 TenSette Pipet	1000/pkg	21856–28
Thermometer, – 20 to 105 °C	each	1877–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SILVER (0 to 0.60 mg/L)

Colorimetric Method



1. Enter the stored program for silver (Ag).

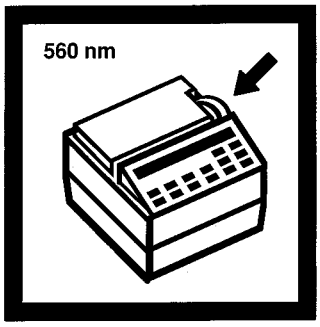
Press: **6 6 0 READ/ENTER**

The display will show:
DIAL nm to 560

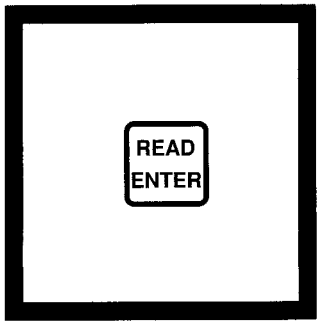
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps. Adjust the pH of stored samples before analysis.*



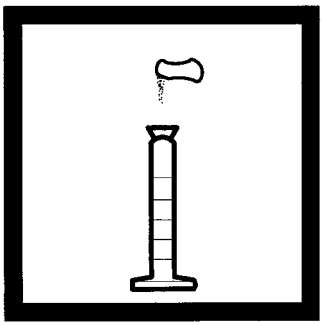
2. Rotate the wavelength dial until the small display shows:
560 nm



3. Press: **READ/ENTER**

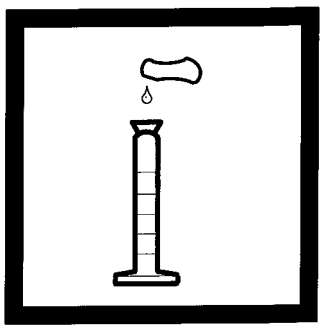
The display will show:
mg/l Ag

Note: *If cyanide is present, digest the sample; see* Digestion *below.*



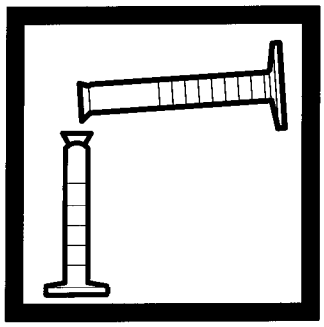
4. Add the contents of one Silver 1 Powder Pillow to a dry 50–mL graduated mixing cylinder.

Note: *If the Silver 1 Powder becomes wet at this point, the powder will not dissolve completely, which will inhibit color development.*



5. Add the contents of one Silver 2 Reagent Solution Pillow to the cylinder. Swirl to completely wet the powder.

Note: *If clumps of dry powder are present when the sample is poured in, the powder will not dissolve completely. This will inhibit color formation.*

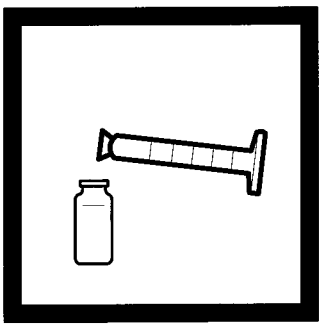


6. Using a 50–mL graduated cylinder, add 50 mL of sample to the cylinder. Stopper. Invert repeatedly for one minute.

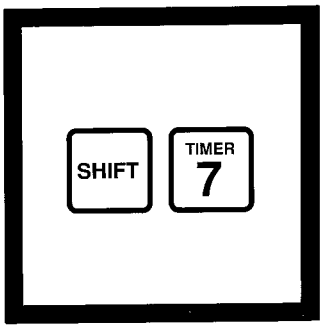
Note: *For proof of accuracy, use a 0.50 mg/L silver standard solution in place of the sample (see* Accuracy Check*).*

7. Pour 25 mL of the mixture into a sample cell (the blank). Add the contents of one Thiosulfate Powder Pillow to the sample cell. Swirl for 30 seconds to mix.

Note: *It is important to generate a blank for each sample.*

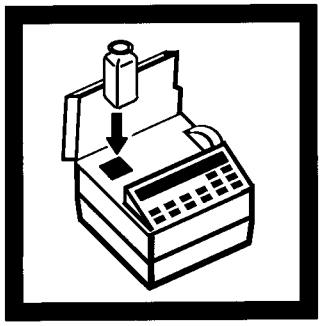


8. Pour the portion remaining in the cylinder into a second sample cell (the prepared sample).



9. Press: **SHIFT TIMER**

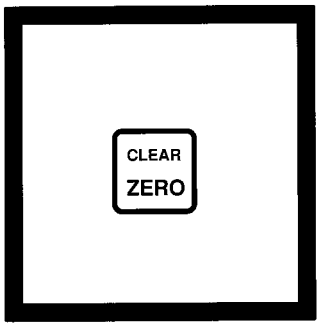
A 2–minute reaction period will begin.



10. When the timer beeps, the display will show:

mg/l Ag

Place the blank in the cell holder. Close the light shield



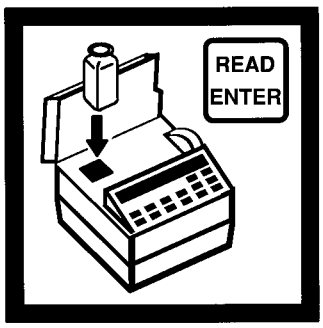
11. Press: **ZERO**

The display will show:

WAIT

then:

0.00 mg/l Ag



12. Place the prepared sample in the cell holder. Close the light shield.

Press: **READ/ENTER**

The display will show:

WAIT

then the results in mg/L silver will be displayed

Note: *Rinse the cells carefully between samples to avoid development of a film on the cell walls.*

Note: *In the constant on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

SAMPLING AND STORAGE

Collect samples in acid–cleaned plastic or glass bottles. Using pH paper, adjust the pH to 2 or less with nitric acid (about 2 mL/liter). Store preserved samples at room temperature for up to 6 months. Before analysis, adjust the pH to 9–10 with 5.0 N sodium hydroxide. Do not use a pH meter because of silver contamination from the electrode. Correct for volume additions by dividing the total volume (sample + acid + sample) by the volume of the sample and multiply the result times the final test reading.

ACCURACY CHECK

Standard Additions Method

a) Add 5.0 mL of 1000–mg/L Silver Standard Solution to a 100–mL volumetric flask. Dilute to volume with demineralized water. Mix well. This is a 50–mg/L silver standard solution.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of this standard solution to three 50–mL samples (or sample portions diluted to 50 mL). Mix well.

c) Analyze as described above. Each 0.1 mL addition of standard should increase the silver concentration by 0.1 mg/L.

d) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method

Prepare a 0.50-mg/L silver standard solution as follows:

a) Use a TenSette Pipet or 0.5–mL volumetric pipet to add 0.50 mL of 1000–mg/L Silver Standard Solution to a 1000–mL volumetric flask.

b) Dilute to the mark with demineralized water. Prepare this solution daily. Perform the silver test as described.

PRECISION

In a single laboratory, using standard solutions of 0.4 mg/L silver and two representative lots of reagent and a DR/2000, a single operator obtained a standard deviation of ±0.015 mg/L silver.

INTERFERENCES

Interfering studies were conducted by preparing a known silver solution (about 0.4 mg/L) and the potential interfering ion. The ion was said to interfere when the resulting concentration changed by ±10%.

Negative Interference:

Aluminum	30 mg/L
Ammonia	750 mg/L
Cadmium	15 mg/L
Chloride	19 mg/L
Chromium^{6+}	90 mg/L
Copper	7 mg/L
Iron	30 mg/L
Lead	13 mg/L
Manganese	19 mg/L
Nickel	19 mg/L
Zinc	70 mg/L

Positive Interference:

Calcium	600 mg/L
Magnesium	2000 mg/L
Mercury	2 mg/L

DIGESTION

This digestion is for samples containing organic matter, thiosulfate or cyanide. Possible sources for these compounds are wastewater, silver electroplating baths and silver strike solutions. Digestion should be done with a Digesdahl Digestion Apparatus.

Caution: *Poisonous hydrogen cyanide gas is generated during this digestion.*

Warning: ***Always wear safety glasses and use a safety shield, or operate the Digesdahl within a closed fume hood. Follow the additional safety precautions in the*** **Digesdahl Apparatus Manual.**

a) Add an appropriate size sample to the 100–mL volumetric flask of the Digesdahl. Add several boiling chips to prevent bumping.

Note: *Appropriate sample size is determined experimentally. The final sample concentration (after dilution to 100 mL) should be between 0–0.5 mg/L. Larger dilutions may be necessary for electroplating baths and silver strike solutions. Do not exceed the maximum sample volume of 25 mL. Several 25–mL aliquots may be digested in succession to concentrate a very dilute sample.*

b) Turn on the water aspirator and make sure there is suction in the fractionating head.

c) Add 3 mL of concentrated sulfuric acid to the sample in the volumetric flask. Immediately place the head on the volumetric flask. Never use less than 3 mL of acid.

d) Place the volumetric flask in the heater. Turn the temperature dial to 440 °C (825 °F).

e) After the sample begins to char or the sulfuric acid reflux line becomes visible, wait 3–5 minutes.

f) Visually confirm the presence of acid in the flask before adding hydrogen peroxide.

g) Add 10 mL of 50% hydrogen peroxide to the sample via the capillary funnel in the fractionating head.

h) After the hydrogen peroxide has boiled off, heat the sample until heavy white sulfuric acid fumes are present. Continue heating and reduce the sample volume to near dryness. Do not let the sample go completely dry at any time.

Note: *If the sample goes to dryness, turn the Digesdahl off and cool completely. Add water to flask before handling. Repeat digestion from the beginning.*

Note: *If only thiosulfate is present in the sample, proceed to Step l.*

i) Add another 3 mL of sulfuric acid via the capillary funnel.

j) Add another 5 mL of hydrogen peroxide. Check the solution for digestion completion. If digestion is not complete, continue adding hydrogen peroxide in 5 to 10 mL portions. Several portions may be necessary.

Note: *Digestion is complete when the digestate is colorless or the color of the digestate does not change upon addition of hydrogen peroxide. Also, a completely digested sample will not foam.*

k) After digestion is complete and all the hydrogen peroxide is boiled off, reduce the volume of the digestate to near dryness. Do not allow the sample to become completely dry. Remove the flask from the heater. Cool to room temperature.

l) Slowly add about 25 mL of demineralized water to the cooled flask.

m) Add 2 drops of 1–g/L Phenolphthalein Indicator Solution. Add 2 drops of 1–g/L Thymolphthalein Indicator Solution.

n) Using sodium hydroxide, adjust the pH of the solution to between 9–10. The solution will be pink in this pH range.

Note: A purple color indicates a pH greater than 10. If this occurs, add a drop of sulfuric acid and 2 drops of each indicator; repeat pH adjustment. Initially, use 50 % sodium hydroxide, then 1.0 N sodium hydroxide as the end point is approached.

o) Filter turbid digestates; see *Filtering Samples* (*Section I*). Quantitatively transfer the filtrate (or unfiltered sample) to a clean 100–mL volumetric flask. Dilute to the mark with demineralized water. The sample is ready for analysis.

SUMMARY OF METHOD

Silver ions in basic solution react with cadion 2B to form a green to brown to red–purple complex. The sodium thiosulfate acts as a decolorizing agent for the blank. The Silver 1 and Silver 2 reagents contain the buffer, indicator and masking agents. Organic extractions are not necessary and this method does not have as many interferences as the traditional dithizone method. It may also be used for electroplating and silver strike solutions.

REQUIRED REAGENTS

	Cat. No.
Silver Reagent Set (50 tests)	22966–00
Includes: (2) 22935–68, (2) 22936–68, (1) 22937–66	

Description	Quantity Required Per Test	Units	Cat. No.
Silver 1 Powder Pillow	1 pillow	25/pkg	22935–68
Silver 2 Powder Pillow	1 pillow	25/pkg	22936–68
Sodium Thiosulfate Powder Pillow	1 pillow	50/pkg	22937–66

REQUIRED APPARATUS

Boiling Chips, silicon carbide	2–3	500 g	20557–34
Clippers, for opening powder pillows	1	each	968–00
Cylinder, graduated, 50 mL	1	each	21179–41
Cylinder, graduated, mixing, 50 mL	1	each	1896–41

OPTIONAL REAGENTS

Hydrogen Peroxide, 50%	490 mL	21196–49
Phenolphthalein Indicator Solution, 1 g/L	15 mL SCDB	1897–36
Silver Standard Solution, 1000 mg/L Ag	100 mL	14613–42
Sodium Hydroxide Solution, 1.0 N	100 mL MDB	1045–32
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450–37
Sodium Hydroxide, 50%	500 mL	2180–49
Sulfuric Acid, ACS	4 kg	979–09
Thymolphthalein Indicator Solution, 1 g/L	15 mL SCDB	21853–36
Water, demineralized	4 L	272–56

OPTIONAL APPARATUS

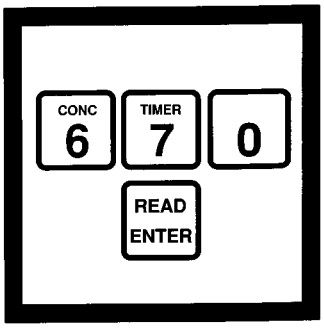
Digesdahl Digestion Apparatus, 115 Vac, 50/60 Hz	each	23130–20
Digesdahl Digestion Apparatus, 230 Vac, 50/60 Hz	each	23130–21
Flask, volumetric, Class A, 100 mL	each	14574–42
Pipet, serological, 10.0 mL	each	532–38
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700–10
Pipet Tips, for 19700–01 Pipet	50/pkg	21856–96
Pipet Tips, for 19700–10 Pipet	50/pkg	21997–96
Pipet, volumetric, Class A, 0.50 mL	each	14515–34
Pipet Filler, safety bulb	each	14651–00
Pour–Thru Cell Kit	each	45215–00
Safety Glasses	each	18421–00
Safety Shield, for Digesdahl	each	20974–00
Sample Cells, 1–inch, polystyrene, disposable	12/pkg	24102–12

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SODIUM CHROMATE (0 to 1,100 mg/L)

For water, wastewater and seawater

Direct Colorimetric Method



1. Enter the stored program for sodium chromate (Na_2CrO_4).

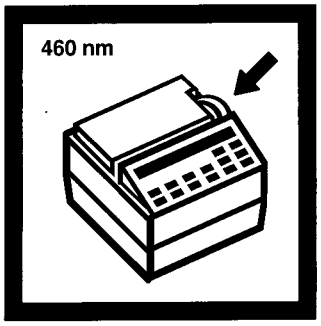
Press: **6 7 0 READ/ENTER**

The display will show:
DIAL nm to 460

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

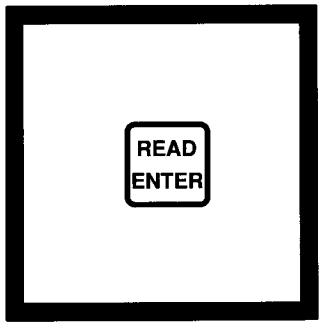
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *See* Sampling and Storage *following these steps.*



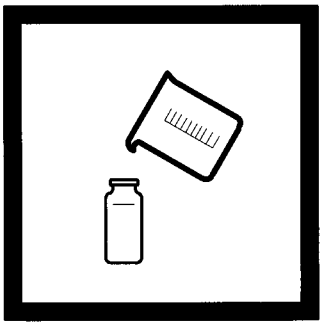
2. Rotate the wavelength dial until the small display shows:
460 nm

Note: *This test is sensitive to the wavelength setting. The 460 nm wavelength should always be approached form high to low values. For greater accuracy, a manual calibration may be stored as a user–store program; see the* Operation *section of the* DR/2000 Instrument Manual.



3. Press: **READ/ENTER**

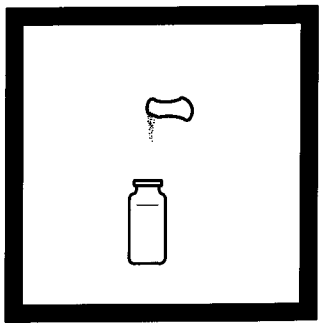
The display will show:
mg/l Na_2CrO_4



4. Fill a clean sample cell to the 25–mL mark with sample.

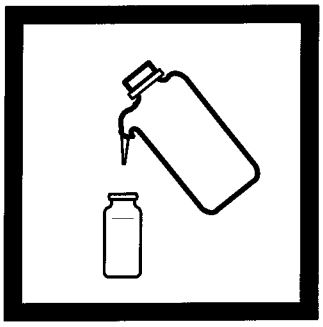
Note: *For proof of accuracy, use a 1000-mg/L Sodium Chromate Standard Solution in place of the sample* (see Optional Reagents).

Note: *Filter turbid samples using the labware listed under* Optional Apparatus.

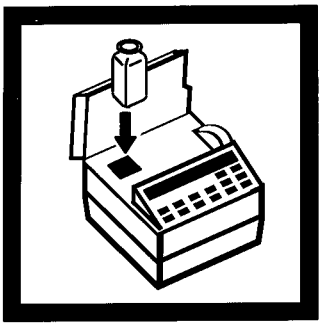


5. Add the contents of one Neutralizing Reagent Powder Pillow to the sample cell. Swirl to mix (the prepared sample).

Note: *The Neutralizing Reagent Powder Pillow is necessary only if the sample is orange or yellow–orange. If the color is in doubt, add the powder pillow.*

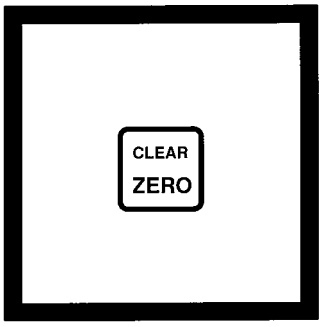


6. Fill a second cell with 25 mL of colorless water (the blank).



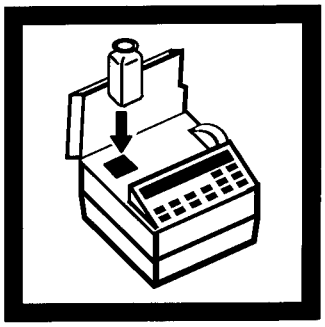
7. Place the blank in the cell holder. Close the light shield

Note: *The Pour–Thru Cell can be used for this procedure.*

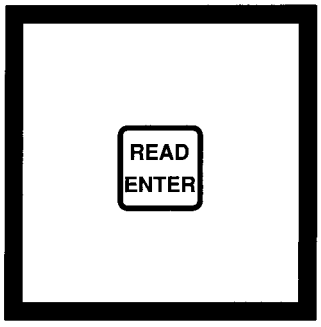


8. Press: **ZERO**

The display will show:
WAIT
then:
0. mg/l Na_2CrO_4



9. Place the prepared sample in the cell holder. Close the light shield.



10. Press: **READ/ENTER**

The display will show:
WAIT
then the results in mg/L sodium chromate (Na_2CrO_4) will be displayed

Note: *In the constant on mode, pressing READ/ENTER is not required. WAIT will note appear. When the display stabilizes, read the result.*

Note: *The results may be expressed as mg/L chromate (CrO_4^{2-}) or mg/L hexavalent chromium (Cr^{6+}) by multiplying the result by 0.72 or 0.321, respectively.*

SAMPLING AND STORAGE
Collect samples in clean glass bottles.

ACCURACY CHECK
Standard Additions Method
a) Snap the neck off a Sodium Chromate Voluette Ampule Standard Solution, 25,000 mg/L Na_2CrO_4.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25–mL samples, respectively. Swirl to mix.

c) Analyze the samples according to the above procedure. The sodium chromate concentration should increase by 100 mg/L for each 0.1 mL addition of standard.

d) If these increases do not occur, see *Standard Additions* in *Section I* for more information.

Standard Solution Method
Use a 1000 mg/L Sodium Chromate Standard Solution listed under *Optional Reagents* to check accuracy.

INTERFERENCES
Large amounts of turbidity will result in high readings.

SUMMARY OF METHOD
The test directly measures the intensity of the alkaline yellow color of the sodium chromate solution. In acid media, the solution is orange and must be treated. A neutralizing agent is added to raise the pH, giving the yellow color for the determination.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Neutralizing Reagent Powder Pillows	1 pillow	100/pkg	2127–99

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968–00

OPTIONAL REAGENTS

Sodium Chromate Standard Solution, 1000 mg/L Na_2CrO_4		500 mL	2503–49
Sodium Chromate Standard Solution, Voluette Ampule, 25,000 mg/L Na_2CrO_4		16/pkg	14255–10

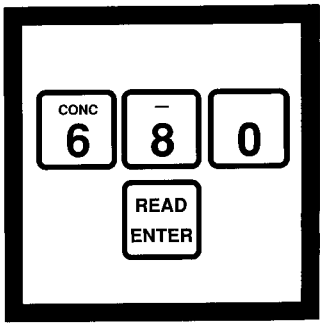
OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968–00
Funnel, poly, 65 mm		each	1083–67
Filter Paper, folded, 12.5 cm		100/pkg	1894–57
Pipet, TenSette, 0.1 to 1.0 mL		each	19700–01
Pipet Tips, for 19700–01 Pipet		50/pkg	21856–96
Pour–Thru Cell Assembly		each	45215–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SULFATE (0 to 70 mg/L)

SulfaVer 4 Method*, USEPA accepted for reporting wastewater analysis**
USING POWDER PILLOWS



1. Enter the stored program for sulfate (SO_4^{2-}).

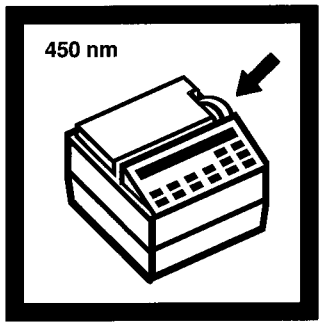
Press: **6 8 0 READ/ENTER**

The display will show:
DIAL nm to 450

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

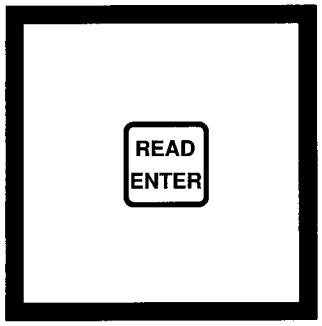
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps.*



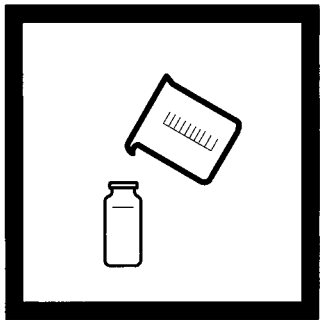
2. Rotate the wavelength dial until the small display shows:
450 nm

Note: *For greater accuracy, prepare an instrument calibration for each new lot of SulfaVer 4 Sulfate Reagent Powder Pillows; see* Calibration *following these steps.*



3. Press: **READ/ENTER**

The display will show:
mg/l SO_4^{2-}

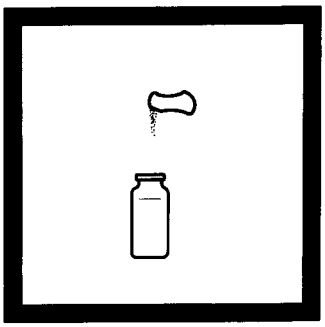


4. Fill a clean sample cell with 25 mL of sample.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and Step 6. Use labware listed under* Optional Apparatus.

Note: *For proof of accuracy, use a 50 mg/L sulfate standard solution* (see Accuracy Check) *in place of the sample.*

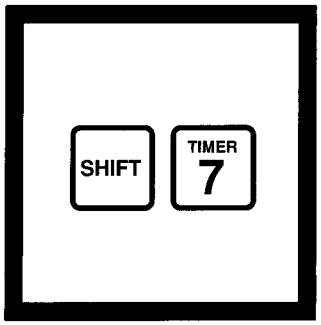
*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Procedure is equivalent to USEPA method 375.4 for wastewater.



5. Add the contents of one SulfaVer 4 Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to dissolve.

Note: *A white turbidity will develop if sulfate is present.*

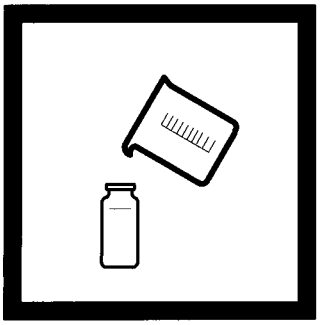
Note: *Accuracy is not affected by undissolved powder.*



6. Press: **SHIFT TIMER**

A 5–minute reaction period will begin.

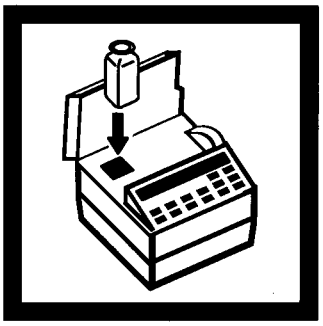
Note: *Allow the cell to stand undisturbed.*



7. When the timer beeps, the display will show:

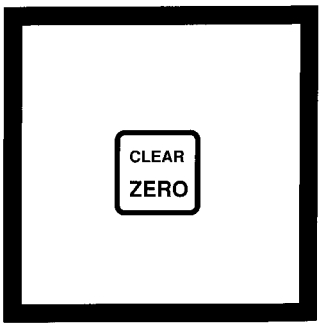
mg/l SO_4^{2-}

Fill a second sample cell with 25 mL of sample (the blank).



8. Place the blank into the cell holder. Close the light shield.

Note: *The Pour–Thru Cell cannot be used with this procedure.*



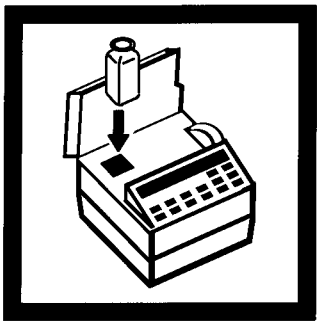
9. Press: **ZERO**

The display will show:

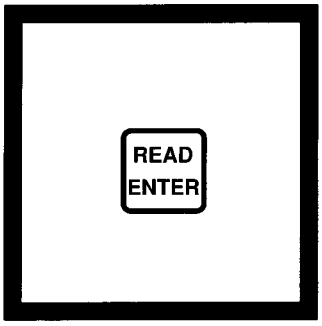
WAIT

then:

0. mg/l SO_4^{2-}



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.



11. Press: **READ/ENTER**

The display will show:

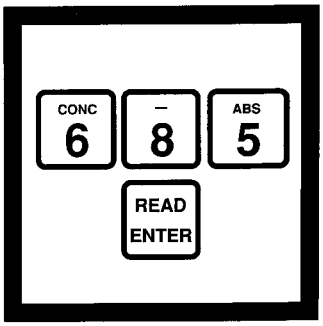
WAIT

then the results in mg/L SO_4^{2-} will be displayed.

Note: *In the constant on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

Note: *Clean the sample cells with soap and a brush.*

USING ACCUVAC AMPULS



1. Enter the stored program for sulfate (SO_4^{2-}) – AccuVac Ampuls.

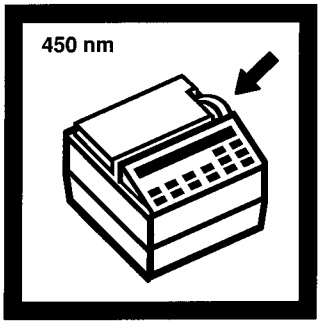
Press: **6 8 5 READ/ENTER**

The display will show:
DIAL nm to 450

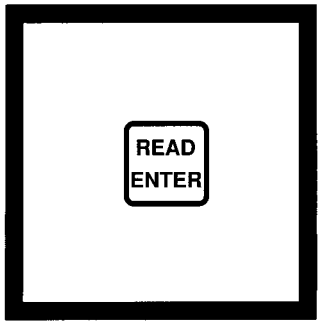
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps.*

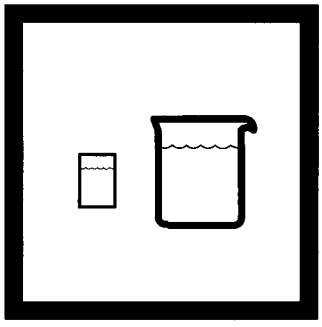


2. Rotate the wavelength dial until the small display shows:
450 nm



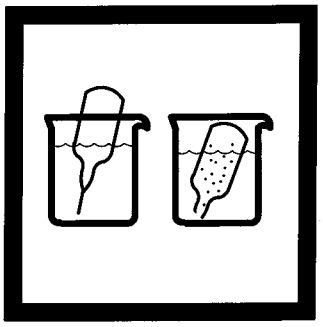
3. Press: **READ/ENTER**

The display will show:
mg/l SO_4^{2-} AV



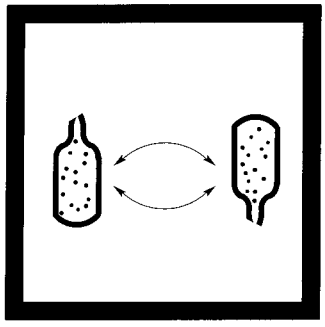
4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50–mL beaker.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and Step 5. Use labware listed under* Optional Apparatus.



5. Fill a SulfaVer 4 Sulfate AccuVac ampul with sample.

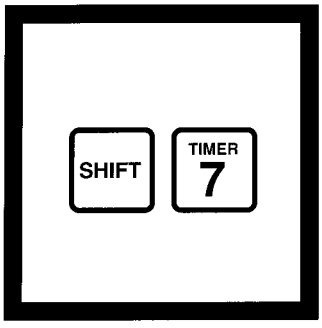
Note: *Keep tip immersed until the ampul fills completely.*



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

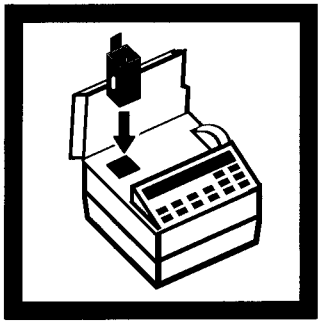
Note: *A white turbidity will develop if sulfate is present.*

Note: *Accuracy is not affected by undissolved powder.*



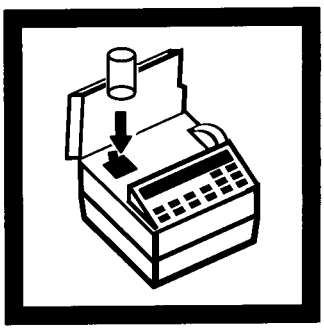
7. Press: **SHIFT TIMER**

A 5–minute reaction period will begin

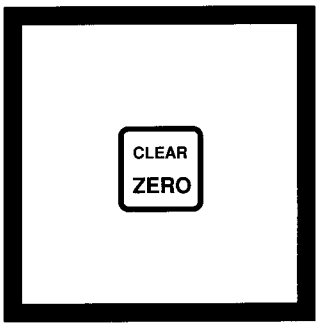


8. Place the AccuVac Vial Adapter into the cell holder.

Note: *Place the grip tab at the rear of the cell holder.*

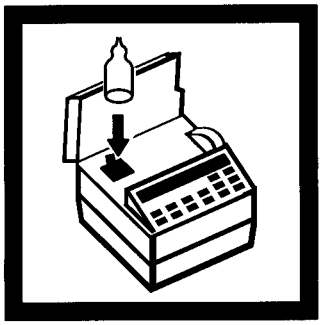


9. When the timer beeps, the display will show:
mg/l SO_4^{2-} AV
Place the blank into the cell holder. Close the light shield.

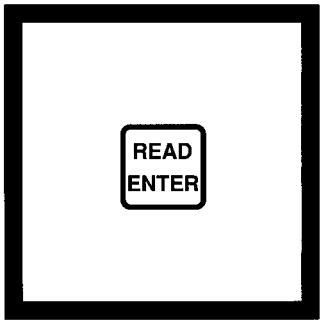


10. Press: **ZERO**

The display will show:
WAIT
then:
0. mg/l SO_4^{2-} AV



11. Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the results in mg/L SO_4^{2-} will be displayed.

Note: *In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

CALIBRATION

A new calibration should be performed for each new lot of SulfaVer 4 Sulfate Reagent Powder Pillows as follows:

a) Prepare standards of 0, 10, 20, 30, 40, 50 and 60 mg/L sulfate by diluting 0, 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 mL of the contents of a Sulfate Voluette Ampule Standard, 2500 mg/L, to 25 mL with demineralized water in graduated mixing cylinders. Use a TenSette Pipet to measure the standard. Mix well.

Or, pipet 0, 1.0, 2.0, 3.0, 4.0, 5.0, and 6.0 mL of a 1000–mg/L sulfate Standard Solution into 100-mL volumetric flasks. Dilute to volume. Mix well. Transfer 25 mL to each test cylinder.

b) Store the calibration in the instrument memory using the procedure in the *Operation* section of the *DR/2000 Instrument Manual.* Follow the procedure described, choosing a wavelength of 450 nm, the decimal position as 0000, units as mg/L SO_4^{2-} and a Timer 1 interval of 05:00. Note the program number assigned to the procedure.

c) Add the reagents to the demineralized water (0 standard–reagent blank) and to the 10 mg/L standard as described in Steps 4 to 6 of the powder pillow procedure above, using the demineralized water blank to perform the zero calibration. Enter the sulfate concentration of the first standard (10 mg/L) and measure the absorbance as directed by the instrument manual. React and measure the remaining standards.

d) Use this stored program number in the powder pillow procedure above. Prepare a new calibration for each new lot of reagent, using the same stored program number.

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Sulfate Voluette Ampule Standard Solution, 2500 mg/L.

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 25–mL samples. Mix thoroughly. For AccuVac Ampuls, use 50–mL beakers.

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 I* 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 the contents of a Voluette Ampule Standard for Sulfate into a 50–mL volumetric flask. Dilute to volume with demineralized water.

PRECISION

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of powder pillows with the DR/2000, a single operator obtained a standard deviation of ±0.9 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 DR/2000, a single operator obtained a standard deviation of ±2.2 mg/L sulfate.

INTERFERENCES

The following interfere at levels above those concentrations listed:

Calcium	20,000 mg/L as $CaCO_3$
Chloride	40,000 mg/L as $CaCO_3$
Magnesium	10,000 mg/L as $CaCO_3$
Silica	500 mg/L as $CaCO_3$

SUMMARY OF METHOD

Sulfate ions in the sample react with barium in the SulfaVer 4 and form a precipitate of 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.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Units	Cat. No.
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow	50/pkg	12065–66

REQUIRED REAGENTS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls	1 ampul	25/pkg	25090–25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968–00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	43784–00
Brush	1	each	690–00
Vial, zeroing	1	each	21228–00

OPTIONAL REAGENTS

Sulfate Standard Solution, 50 mg/L	500 mL	2578–49
Sulfate Standard Solution, 1000 mg/L	500 mL	21757–49
Sulfate Standard Solution, Voluette Ampule, 2500 mg/L, 10 mL	16/pkg	14252–10
Water, demineralized	4 L	272–56

OPTIONAL APPARATUS

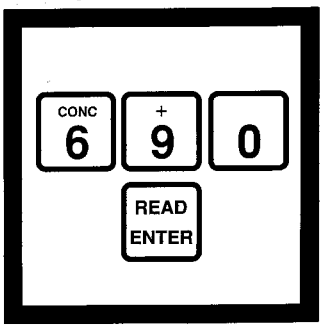
AccuVac Snapper Kit	each	24052–00
Ampule Breaker Kit	each	21968–00
Beaker, 50 mL	each	500–41
Filter Paper, folded, 12.5 cm	100/pkg	1894–57
Flask, volumetric, 50 mL, Class A	each	14574–41
Flask, volumetric, 100 mL, Class A	each	14574–42
Funnel, poly, 65 mm	each	1083–67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 Pipet	50/pkg	21856–96
Pipet, volumetric, Class A, 1.00 mL	each	14515–35
Pipet Filler, safety bulb	each	14651–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SULFIDE (0 to 0.600 mg/L S^{2-})

For water, wastewater and seawater

Methylene Blue Method*, USEPA accepted for reporting wastewater analysis**



1. Enter the stored program for sulfide (S^{2-}).

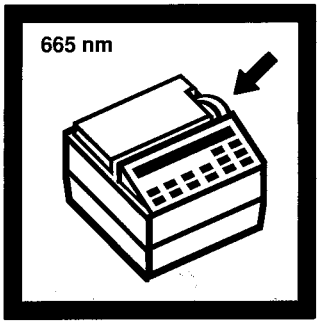
Press: **6 9 0 READ/ENTER**

The display will show:
DIAL nm to 665

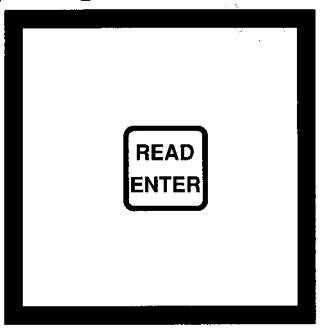
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

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

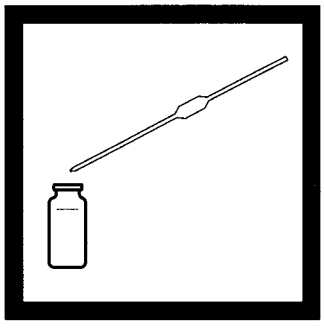


2. Rotate the wavelength dial until the small display shows:
665 nm



3. Press: **READ/ENTER**

The display will show:
mg/l S^{2-}

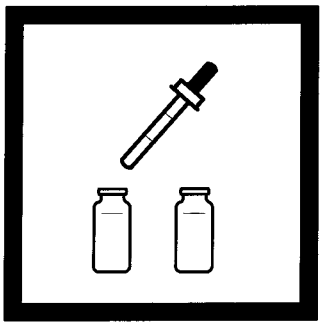


4. Fill a clean sample cell with 25 mL of sample.

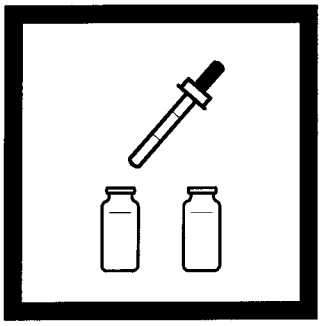
Note: *For turbid samples, see Interferences following these steps for pretreatment instructions.*

Note: *Excessive agitation will cause loss of sulfide. Use a pipet to minimize sulfide loss.*

5. Fill a second sample cell with 25 mL of demineralized water (the blank).

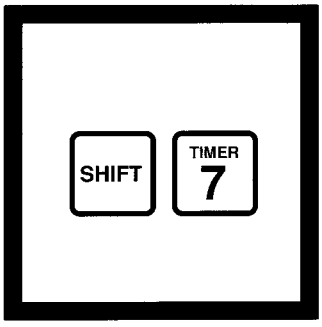


6. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.



7. 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.*

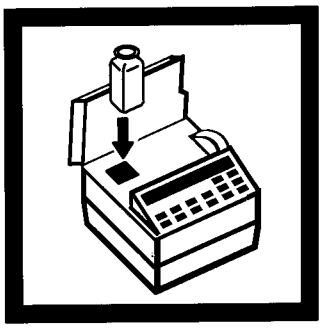


8. Press: **SHIFT TIMER**

A 5–minute reaction period will begin.

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

**Procedure is equivalent to USEPA method 376.2 and Standard Method 4500–S^{2-} D for wastewater.

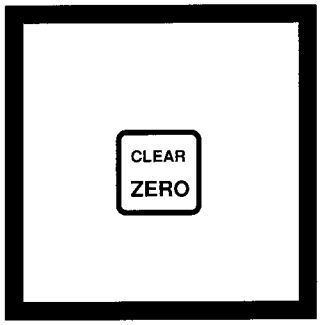


9. When the timer beeps, the display will show:

mg/l S^{2-}

Place the blank into the cell holder. Close the light shield.

Note: The Pour–Thru Cell can be used with this procedure.



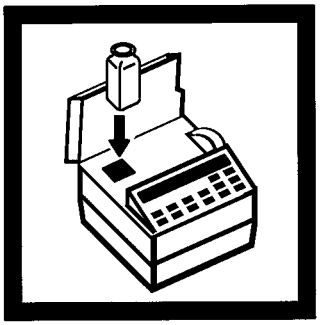
10. Press: **ZERO**

The display will show:

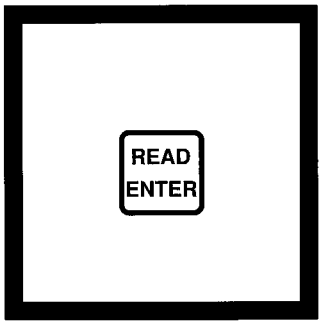
WAIT

then:

0.000 mg/l S^{2-}



11. Immediately place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L sulfide (S^{2-}) will be displayed.

Note: In the constant on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

SAMPLING

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

Standard Solution Method

Sulfide standard solutions are very unstable and should be prepared from sodium sulfate and standardized as described in *Standard Methods for the Examination of Water and Wastewater,* 17th ed., page 4–196.

PRECISION

In a single laboratory, using standard solutions of 0.250 mg/L sulfide and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.003 mg/L sulfide.

INTERFERENCES

For turbid samples, prepare a sulfide–free blank as follows. Use it in place of the demineralized water blank in the procedure.

a) Measure 25 mL of sample into a 50–mL erlenmeyer flask.

b) Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears.

c) Add Phenol Solution dropwise until the yellow color just disappears. Use this solution in Step 5 in place of demineralized water.

Strong reducing substances such as sulfite, thiosulfate and hydrosulfite interfere by reducing the blue color or preventing its development. High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.

DETERMINING 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 proper dilution.

REQUIRED REAGENTS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445–00
Includes: (2) 1816–42, (2) 1817–42	

Description	Quantity Required Per Test	Units	Cat. No.
Sulfide 1 Reagent	2 mL	100 mL MDB	1816–32
Sulfide 2 Reagent	2 mL	100 mL MDB	1817–32
Water, demineralized	25 mL	4 L	272–56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508–40
Pipet, volumetric, Class A, 25 mL	1	each	14515–40
Pipet Filler, safety bulb	1	each	14651–00

OPTIONAL REAGENTS

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

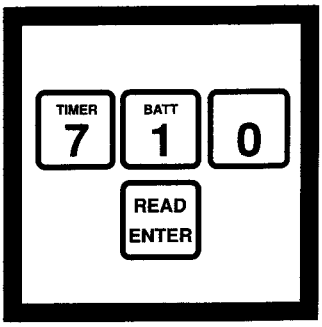
OPTIONAL APPARATUS

Dropper, for 1 oz. bottle	each	2258–00
Flask, erlenmeyer, 50 mL	each	505–41
Pour–Thru Cell Kit	each	45215–00
Standard Methods for the Examination of Water and Wastewater	each	22708–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SURFACTANTS, ANIONIC (0 to 0.275 mg/L) For water, wastewater and seawater

(Also called: Detergents) Crystal Violet Method*



1. Enter the stored program number for anionic surfactants.

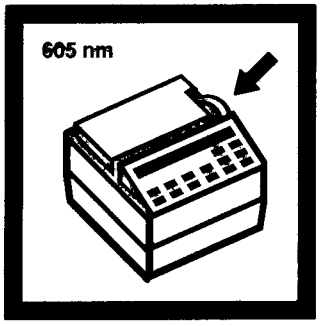
Press: **7 1 0 READ/ENTER**

The display will show:
DIAL nm to 605

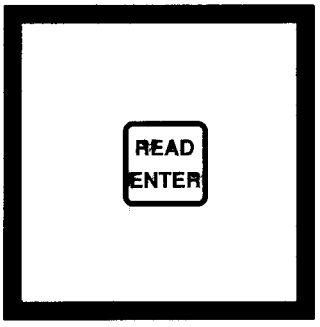
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If sample cannot be analyzed immediately see* Sampling and Storage *following these steps.*

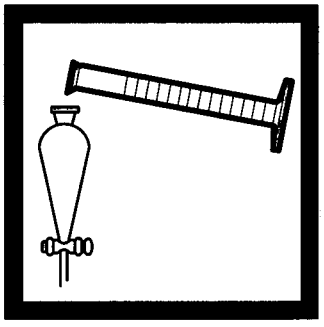


2. Rotate the wavelength dial until the small display shows:
605 nm



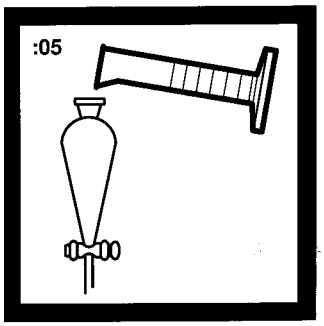
3. Press: **READ/ENTER**

The display will show:
mg/l SURF. ANION

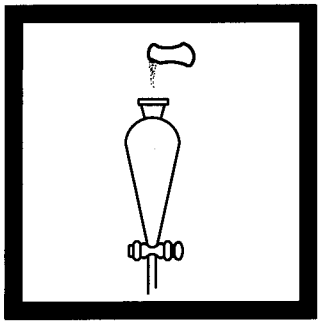


4. Fill a clean 500–mL graduated cylinder to the 300–mL mark with sample. Pour the sample into a clean 500–mL separatory funnel.

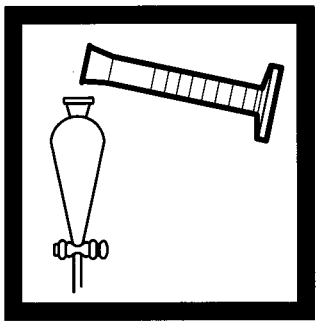
*Analytical Chemistry, 38, 791(1966)



5. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.



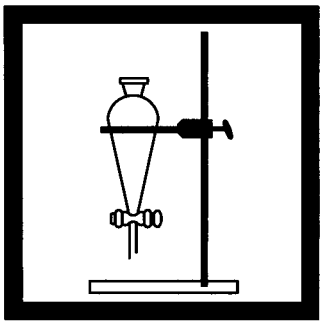
6. Add the contents of one Detergent Reagent Powder Pillow to the funnel. Stopper the funnel and shake to dissolve the powder.



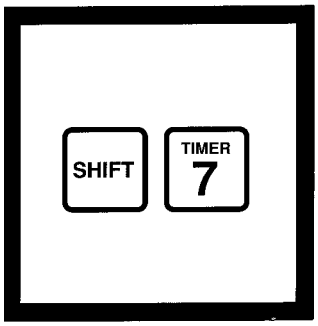
7. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.

Note: *Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.*

Note: *Use benzene only in well–ventilated area.*



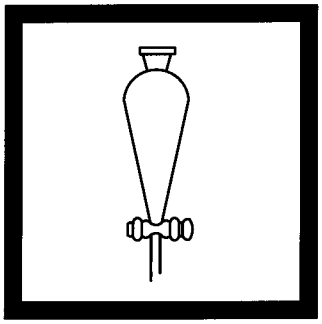
8. Allow the separatory funnel to stand for 30 minutes in a support stand.



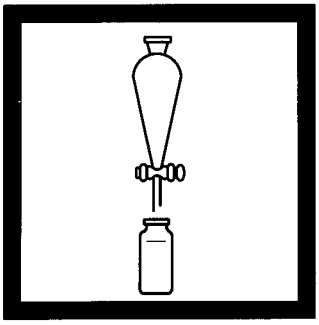
9. Press: **SHIFT TIMER**

A 30–minute reaction period will begin.

Note: *Excessive agitation may cause an emulsion to form, requiring a longer time for phase separation. For these samples, 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.*



10. After the timer beeps, the display will show:
mg/l SURF.ANION
Remove the stopper and drain the bottom layer. Discard this layer.

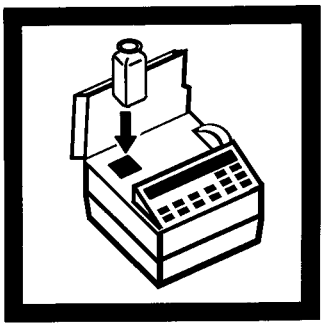


11. Drain the top benzene layer into a clean 25–mL sample cell (the prepared sample).

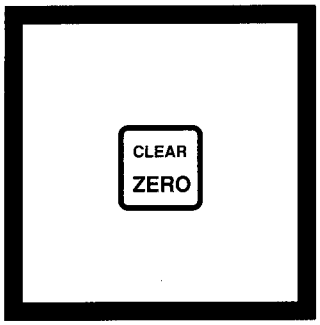
Note: *The benzene layer cannot be filtered before color measurement. Filtration removes the blue color.*

12. Fill another sample cell to the 25–mL mark with pure benzene (the blank).

Note: *The Pour–Thru Cell cannot be used with this procedure.*

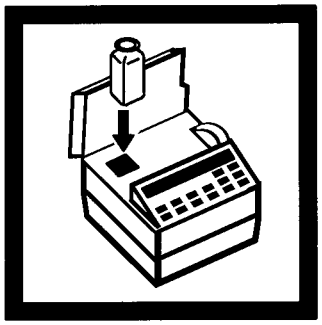


13. Place the blank in the cell holder. Close the light shield.

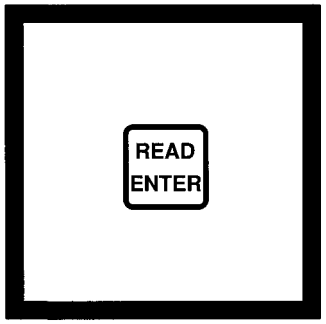


14. Press: **ZERO**

The display will show
WAIT
then:
0.000 mg/l SURF.ANION



15. Place the prepared sample into the cell holder. Close the light shield.



16. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/l anionic surfactants will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

Note: *Acetone may be used to clean benzene from glassware.*

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.

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 anion 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* in *Section I* for more information.

PRECISION

In a single laboratory, using standard solution of 0.1 mg/L LAS and two lots of reagent with a DR/2000, a single operator obtained a standard deviation of ±0.0035 mg/L LAS.

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.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
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
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

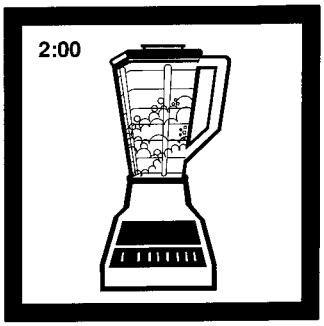
OPTIONAL APPARATUS

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

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

SUSPENDED SOLIDS (0 to 750 mg/L)

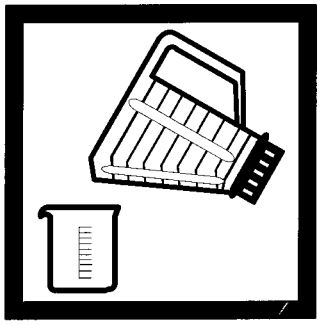
Photometric Method*(Also called Nonfilterable Residue)



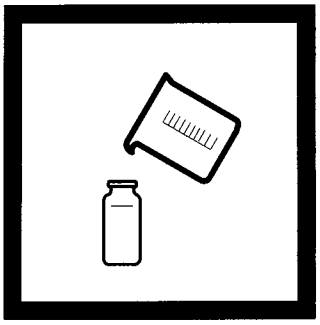
1. Blend 500 mL of sample in a blender at high speed for exactly 2 minutes.

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps.*

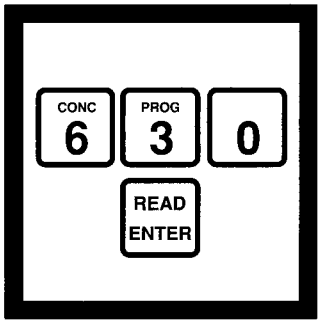
Note: *Obtain blender locally. All other apparatus is available from Hach.*



2. Pour the blended sample into a 600–mL beaker.



3. Stir the sample and immediately pour 25 mL of the blended sample into a sample cell (the prepared sample).



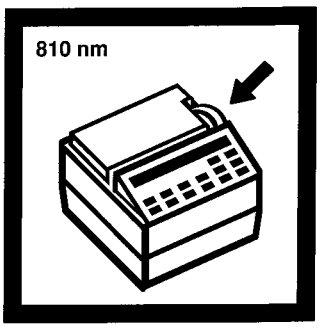
4. Enter the stored program number for suspended solids.

Press: **6 3 0 READ/ENTER**

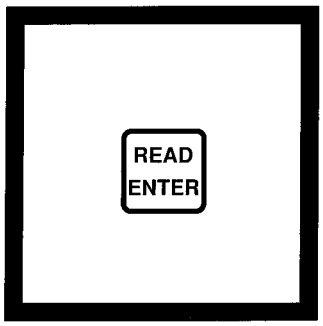
The display will show:
DIAL nm to 810

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 6. Proceed with Step 7.*

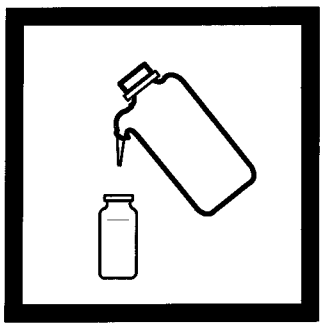


5. Rotate the wavelength dial until the small display shows:
810 nm



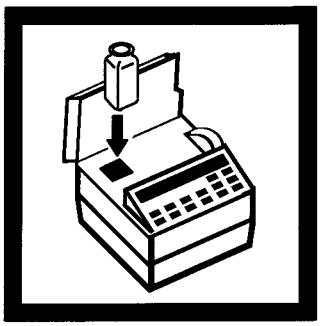
6. Press: **READ/ENTER**

The display will show:
mg/l SUSP.SOLIDS



7. Fill a sample cell with 25 mL of tap or demineralized water (the blank).

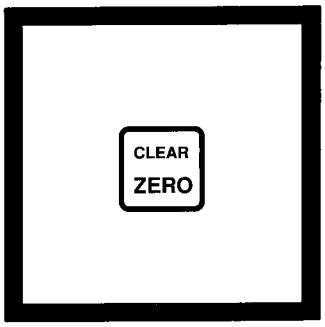
Note: *Remove gas bubbles in the tap water by swirling or tapping the bottom of the cell on a table.*



8. Place the blank into the cell holder. Close the light shield.

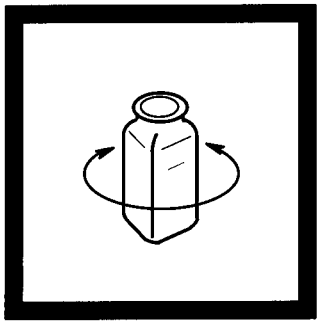
Note: *The Pour–Thru Cell cannot be used with this procedure.*

* Adapted from *Sewage and Industrial Wastes*, 31, 1159 (1959)

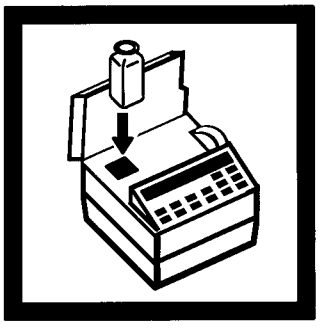


9. Press: **ZERO**

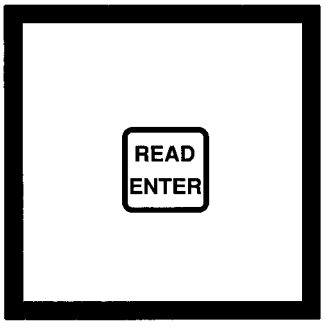
The display will show
WAIT
then:
0. mg/l SUSP.SOLIDS



10. Swirl the prepared sample cell to remove any gas bubbles and uniformly suspend any residue.



11. Place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/l suspended solids will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

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).

INTERFERENCES

Calibration for this test is based on parallel samples using 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 spectrophotometric 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.

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 Per Test	Units	Cat. No.
Beaker, 600 mL, poly	1	each	1080–52
Blender	1	each	purchase locally
Cylinder, graduated, 500 mL, poly	1	each	1081–49
Pipet, serological, 25 mL	1	each	2066–40
Pipet, Filler, safety bulb	1	each	14651–00

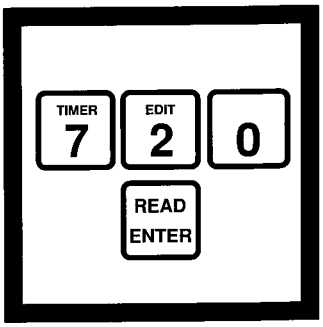
OPTIONAL APPARATUS

Stirring Rod, glass		3/pkg	1770–01

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

TANNIN AND LIGNIN (0 to 9.0 mg/L)

Tyrosine Method*



1. Enter the stored program number for tannin and lignin.

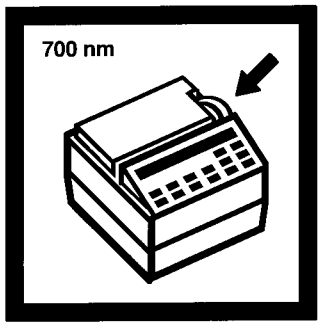
Press: **7 2 0 READ/ENTER**

The display will show:
Dial nm to 700

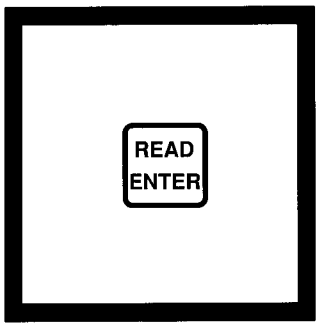
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*

Note: *If sample cannot be analyzed immediately see* Sampling and Storage *following these steps.*

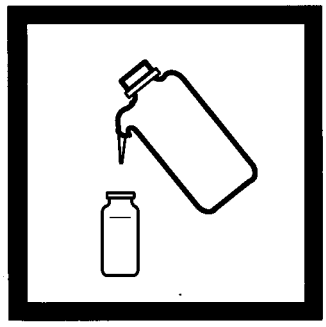


2. Rotate the wavelength dial until the small display shows:
700 nm



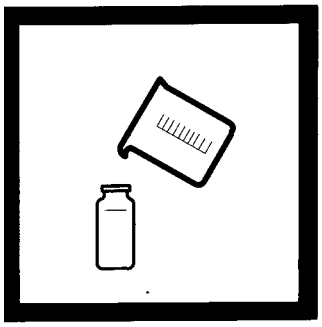
3. Press: **READ/ENTER**

The display will show:
mg/l TANNIC ACID



4. Fill a clean sample cell to the 25–mL mark with demineralized water (the blank).

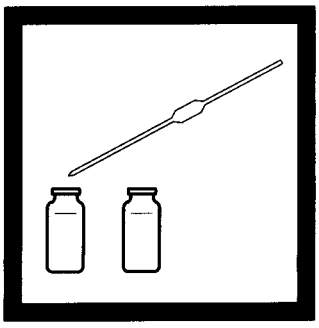
*Adapted from Kloster, M.B., *Journal American Water Works Association,* Vol. 66, No. 1, p. 44 (1974)



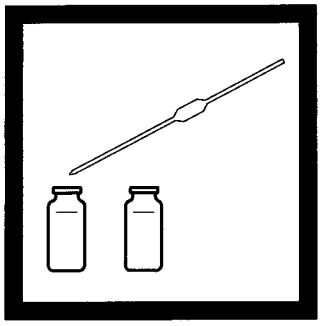
5. Fill another clean sample cell to the 25–mL mark with sample (the prepared sample).

Note: *Filter turbid samples and report results as mg/L soluble tannic acid.*

Note: *For proof of accuracy, use a 2.0 mg/L tannic acid solution in place of the sample* (see Accuracy Check).

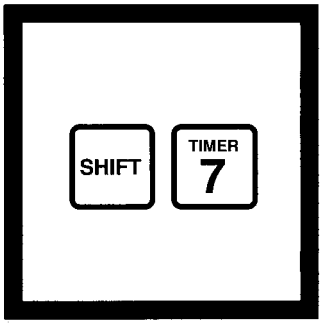


6. Pipet 0.5 mL of TanniVer 3 Tannin–Lignin Reagent into each cell. Swirl to mix.



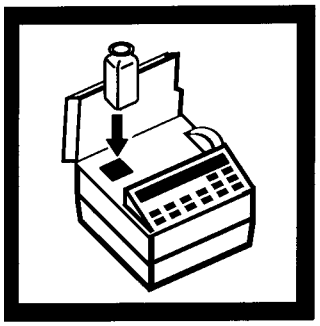
7. Pipet 5.0 mL of Sodium Carbonate Solution into each cell. Swirl to mix.

Note: *A blue color will develop if tannins and/or lignins are present.*



8. Press: **SHIFT TIMER**

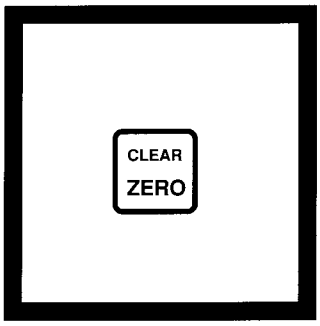
A 25–minute reaction period will begin.



9. When the timer beeps, the display will show:
mg/l TANNIC ACID

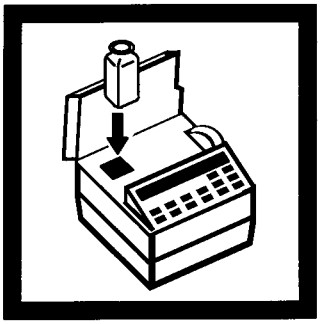
Place the blank into the cell holder. Close the light shield

Note: *The Pour–Thru Cell can be used for this procedure.*

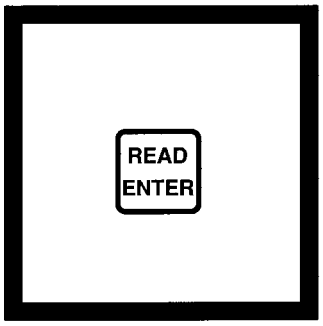


10. Press: **ZERO**

The display will show
WAIT
then:
0.0 mg/l TANNIC ACID



11. Place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L tannic acid will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

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 demineralized 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 demineralized water. Prepare this standard daily.

PRECISION
In a single laboratory, using a standard solution of 5.0 mg/L tannic acid and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.08 mg/L tannic acid.

INTERFERENCES
Sulfite interference is eliminated by adding 1 mL of formaldehyde to the sample before testing the sample.

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.

SUMMARY OF METHOD
This test measures all hydroxylated aromatic compounds, included tannin, lignin, phenol and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. The results are reported as total tannin and lignin expressed as mg/L tannic acid.

REQUIRED REAGENTS

	Cat. No.
Tannin and Lignin Reagent Set (up to 100 tests) Includes: (1) 675–16, (1) 2560–42	22446–00

Description	Quantity Required Per Test	Units	Cat. No.
Sodium Carbonate Solution	10 mL	500 mL	675–49
TanniVer 3 Tannin–Lignin Reagent	1 mL	100 mL	2560–42
Water, demineralized	25 mL	4 L	272–56

REQUIRED APPARATUS

Pipet, volumetric, 5.0 mL	1	each	515–37
Pipet, volumetric, Class A, 0.5 mL	1	each	14515–34

OPTIONAL REAGENTS

Formaldehyde	100 mL	2059–32
Sodium Pyrophosphate, ACS	50 g	784–25
Tannic Acid	113 g	791–14

OPTIONAL APPARATUS

Balance, analytical	each	22310–00
Cylinder, graduated, 25 mL	each	508–40
Filter Paper, folded, 12.5 cm	100/pkg	1894–57
Flask, volumetric, 1000 mL	each	547–53
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, volumetric, Class A, 10.00 mL	each	14515–38
Pipet, Filler, safety bulb	each	14651–00
Pour–Thru Cell Kit	each	45215–00
Spoon, measuring, 0.2 g	each	638–00

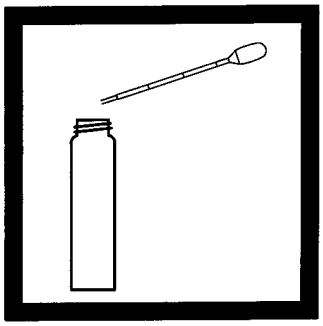
For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

TOXTRAK™ TOXICITY TEST*

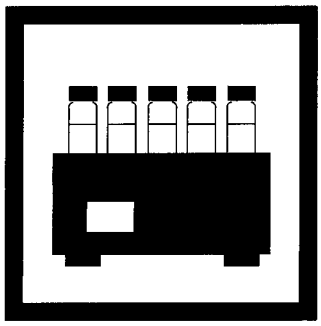
Colorimetric Method**

For wastewater

INOCULUM DEVELOPMENT
Using Indigenous Biomass

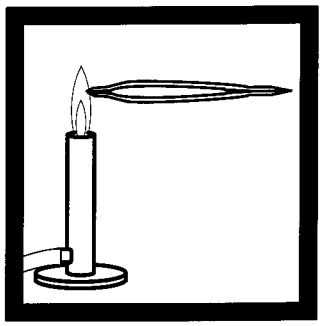


1. Using one of the dropper pipets provided, add 1.0 mL of source culture to a Tryptic Soy Broth Tube.

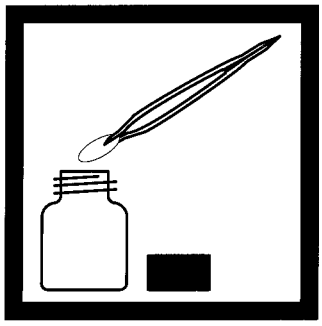


2. Incubate until the vial contents are visibly turbid (turbidty indicates bacterial growth).

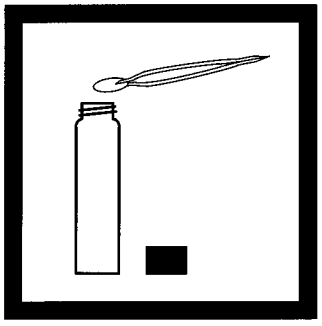
Using Bactrol Disks



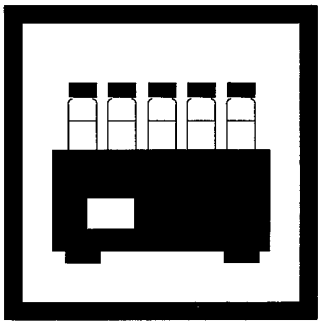
1. Flame sterilize forceps by dipping into alcohol and flame in an alcohol or bunsen burner. Let the forceps cool.



2. Remove the cap from the Bactrol inoculum bottle. Pick out one Bactrol Inoculum Disk with the sterilized forceps.



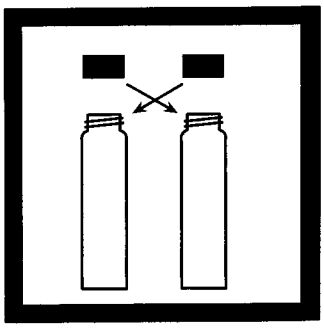
3. Remove the cap from a Lauryl Tryptose Broth Tube and drop in the Inoculum Disk. Shake to dissolve the disk.



4. Incubate the Lauryl Tryptose Broth Tube until the medium is visibly turbid. Turbidity will develop much faster if incubation is done at 35 °C instead of room temperature. At 35 °C, 12 hours is usually sufficient.

*U.S. Patent Number 5,413,916

**Liu, D., *Bull Environm. Contam. Toxicol. 26,* 145–149 (1981)

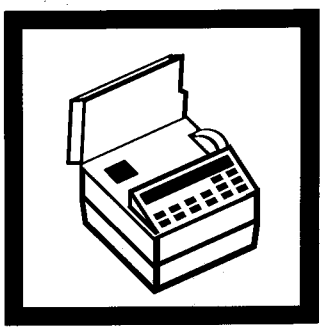


5. Inoculate a new Lauryl Tryptose Broth Tube by first inverting the tube in Step 4, and then switching the caps of the two tubes. Then invert the new tube. After incubation, this new vial may be used in subsequent tests.

Note: *In this way, several medium vials may be inoculated from one Bactrol Disk.*

Note: *If toxicity tests will be run on consecutive days, inoculum may be kept several days in the incubator or at room temperature.*

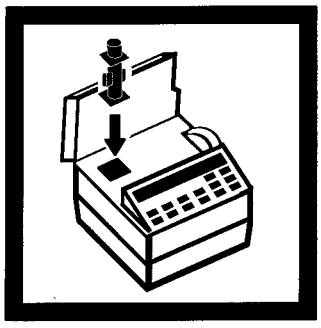
COLORIMETRIC REACTION



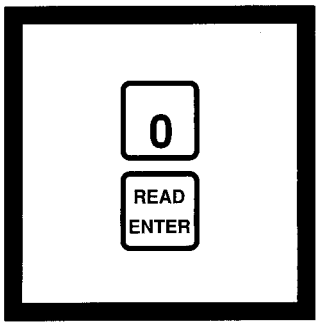
1. Turn the DR/2000 on and configure to continuous mode. Allow the instrument to warm up for at least 15 minutes.

Note: *For software versions 2.2 and lower, press: SHIFT CONFIG. Then press the up arrow key to; 8 2 2 CONSTANT ON. Press READ/ENTER.*

Note: *For software versions 3.0 and above, press: SHIFT CONFIG. If "momentary" is displayed, press READ/ENTER, then CONFIG/METH. If "continuous" is displayed, press CONFIG/METH.*



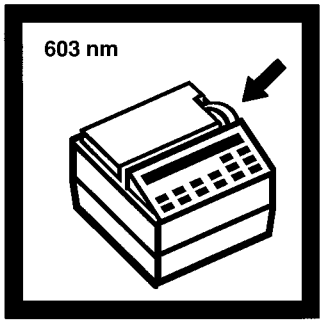
2. Place the 13 mm sample cell adapter into the sample cell compartment. Be sure the windows in the adapter allow light to pass from side to side.



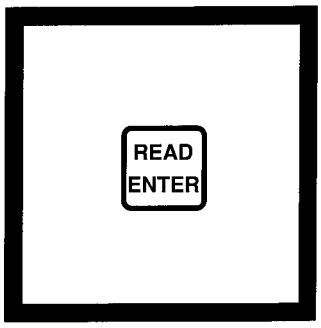
3. Enter the stored program number for absorbance.

Press: **0 READ/ENTER**

The display will show:
Abs

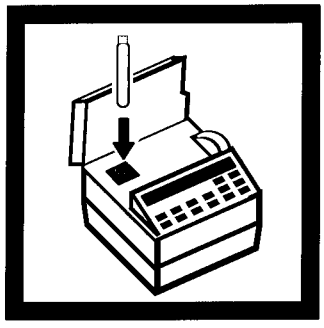


4. Rotate the wavelength dial until the small display shows:
603 nm

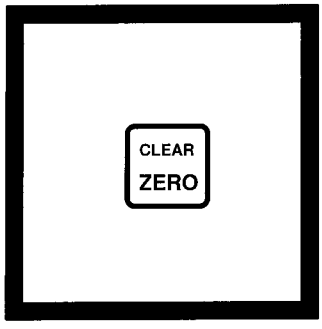


5. Press: **READ/ENTER**

The display will show:
ZERO SAMPLE

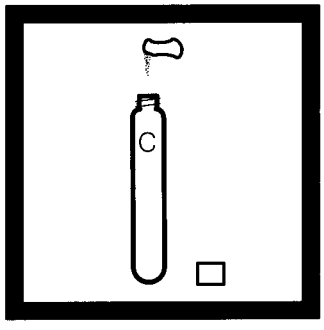


6. Fill a 13–mm reaction tube with demineralized water. Place the tube in the sample cell adapter and place the lid on the adapter.



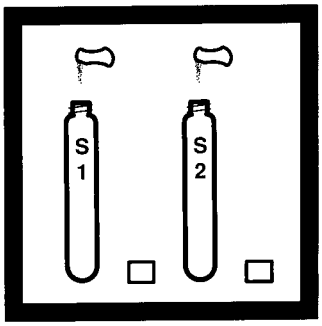
7. Press: **ZERO**

The display will show:
WAIT
then:
0.000 Abs

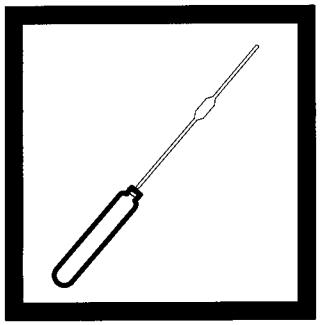


Sample Measurement

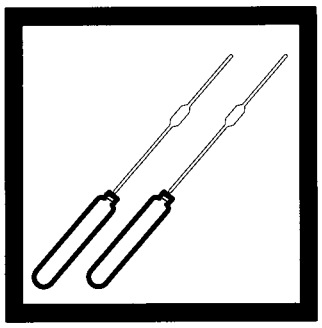
8. For each sample or dilution, open one ToxiTrak Reagent Powder Pillow and add the contents to an empty reaction tube. Label each tube clearly.



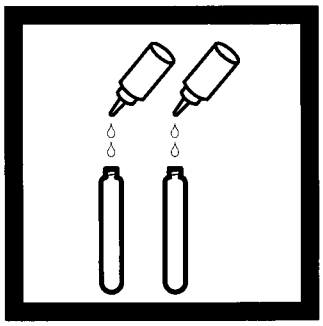
9. For each sample or dilution, repeat Step 8. Label each tube clearly.



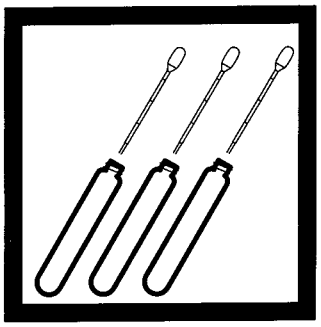
10. Add 5.0 mL of demineralized water to the negative control tube.



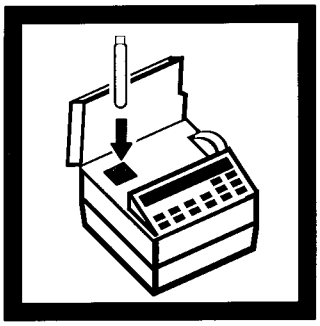
11. Add 5.0 mL of sample (or dilutions) to the sample tubes.



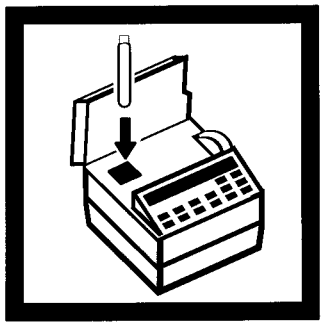
12. Add 2 drops of Accelerator Solution to each tube. Cap and invert to mix.



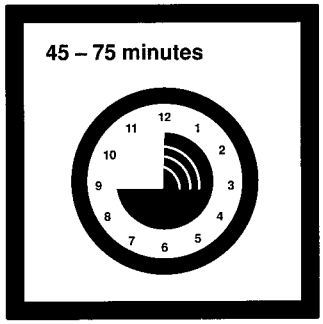
13. Add 0.5 mL of inoculum (previously prepared) to each tube. Cap and invert to mix.



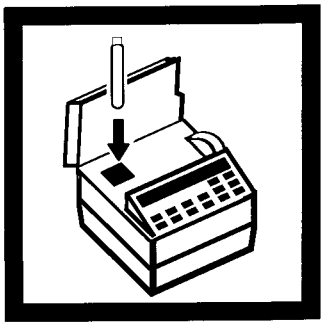
14. Place the negative control in the cell holder. Place the lid on the adapter. Record the absorbance.



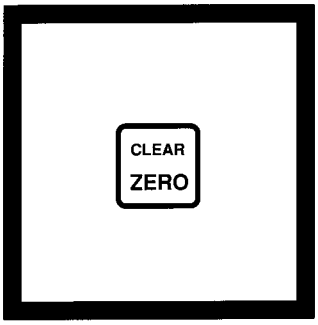
15. Repeat Step 14 for all samples and dilutions. Be sure to record each absorbance.



16. Allow the solutions in the tubes to react until the absorbance of the negative control decreases 0.60 ±0.10. This takes about 45–75 minutes.

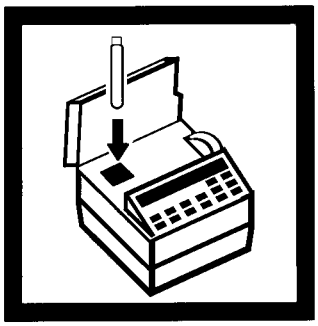


17. Place the reaction tube with demineralized water in the sample cell adaptor. Place the lid on the adaptor.

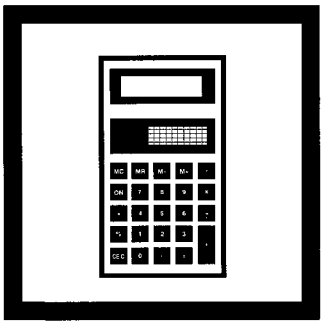


18. Press: **ZERO**

The display will show:
0.000 ABS



19. Beginning with the control, place each sample or dilution in the sample cell and place the lid on the adaptor. Record each absorbance value



20. Calculate the % inhibition (%I) as follows:

$$\%I = [\Delta A_{sample} \div \Delta A_{neg\ control})] \times 100$$

Where ΔA = Initial absorbance value – Final absorbance value

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 20 which is more negative than –10% should be considered toxic.*

TOXTRAK™ TOXICITY TEST, continued

DISPOSAL OF TEST CULTURES

Dispose of active bacterial cultures grown during incubation 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 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

This method is based on the reduction of resazurin, a redox–active dye, by bacterial respiration. When it is reduced, resazurin changes color from blue to pink. Toxic substances can inhibit the rate of resazurin reduction. A chemical accelerant has been added to shorten the reaction time.

REQUIRED REAGENTS

	Cat. No.
ToxiTrak Reagent Set	25972–00
Includes: (1) 25607–66, (1) 25608–36, (1) 22336–15, (2) 21247–10, (2) 20962–08	

Description	Quantity Required Per Test	Units	Cat. No.
ToxiTrak Reagent Powder Pillows	1 pillow	50/pkg	25607–66
ToxiTrak Accelerator Solution	2 drops	15 mL SCDB	25608–36
Tryptic Soy Broth Tubes	1	15/pkg	22336–15

REQUIRED APPARATUS

Clippers, to open powder pillows	each	936–00
Culture Tubes, 13 x 100	10/pkg	20962–08
Dropper Pipet, 1 mL	10/pkg	21247–10
Forceps, flat square tip	each	14537–00
Sample Cell Adapter	each	44798–00

OPTIONAL REAGENTS

Culture Set (incl. Bactrol Discs and Lauryl Tryptose Broth Tubes)	25 cultures	25978–00
Bactrol Discs, E. coli	25/bottle	25978–00
Isopropanol	500 mL	14459–49
Lauryl Tryptose Broth Tubes	15/pkg	21623–15

OPTIONAL APPARATUS

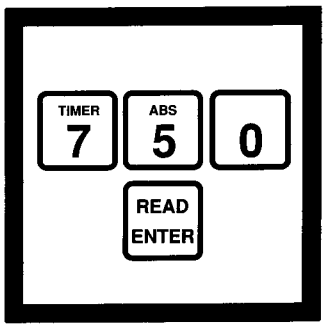
Germicidal Cloth	50/pkg	24632–00
Test Tube Rack	each	24979–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

TURBIDITY (0 to 450 FTU)

For water, wastewater and seawater

Absorptometric Method*



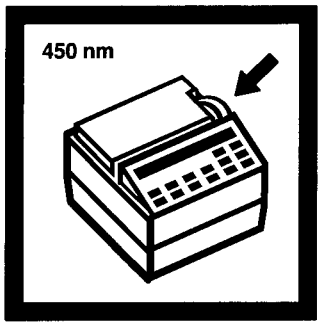
1. Enter the stored program number for turbidity.

Press: **7 5 0 READ/ENTER**

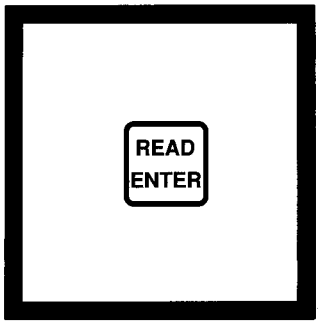
The display will show:
DIAL nm TO 450

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



2. Rotate the wavelength dial until the small display shows:
450 nm

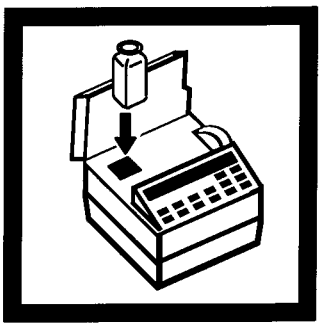


3. Press: **READ/ENTER**

The display will show:
FTU TURBIDITY

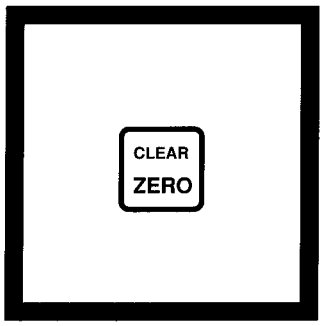
4. Pour 25 mL of demineralized water (the blank) into a sample cell.

Note: *For highly colored samples, a filtered portion of the sample is used in place of the demineralized water.*



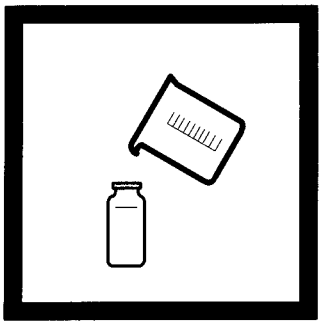
5. Place the blank into the cell holder. Close the light shield.

Note: *The Pour-Thru Cell cannot be used with this procedure.*



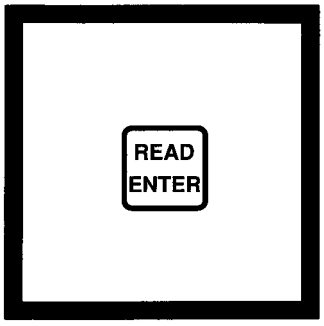
6. Press: **ZERO**

The display will show:
WAIT
then:
0. FTU TURBIDITY



7. Pour 25 mL of sample into another sample cell. Immediately place this sample cell into the cell holder. Close the light shield.

Note: *The sample must be well mixed before transferring it to the sample cell.*



8. Press: **READ/ENTER**

The display will show:
WAIT
then the result in Formazin Turbidity Units (FTU) will be displayed.

Note: *In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

*Adapted from FWPCA *Methods for Chemical Analysis of Water and Wastes*, 275 (1969)

SAMPLING AND STORAGE

Collect samples in clean plastic or glass bottles. Samples should be analyzed as soon as possible after collection but can be stored 48 hours by cooling to 4 °C (39 °F). Warm samples to room temperature before analyzing.

ACCURACY CHECK

Standard Solution Method

The stored program has been calibrated using a milky white suspension of a polymer called formazin. Standard formazin solutions for checking the accuracy of the test can be prepared using the following procedure:

a) Dissolve 1.000 gram of hydrazine sulfate in demineralized water and dilute to the mark in a 100–mL volumetric flask.

b) Dissolve 10.00 grams of hexamethylenetetramine in demineralized water and dilute to the mark in a 100–mL volumetric flask.

c) Mix 5.0 mL of each solution in a 100–mL volumetric flask and allow to stand undisturbed for 24 hours at 25 ±3 °C (77 ±5 °F). Standing temperature is important for correct polymer formation.

d) Dilute to the mark and mix.

The turbidity of this stock solution is 400 FTU and it should be prepared monthly. Dilutions used for standard solutions must be prepared fresh daily. A more convenient prepared formazin stock solution, 4000 NTU (or 4000 FTU), is available from Hach.

PRECISION

In a single laboratory, using a standard solution of 140 FTU and one representative lot of reagent with the DR/2000, a single operator obtained a standard deviation of ±2 FTU.

SUMMARY OF METHOD

The turbidity test measures an optical property of the water sample which results from the scattering and absorbing of light by the particulate matter present. The amount of turbidity registered is dependent on such variables as the size, shape and refractive properties of the particles.

This procedure is calibrated using formazin turbidity standards and the readings are in terms of formazin turbidity units (FTU). This test cannot be used for EPA reporting purposes but may be used for day to day in–plant monitoring. [A formazin turbidity unit (FTU) is equivalent to a nephelometric turbidity unit (NTU) when readings are made on a nephelometer.]

OPTIONAL REAGENTS

Description	Units	Cat. No.
Formazin Stock Solution, 4000 NTU	500 mL	2461–49
Hexamethylenetetramine	500 g	1878–34
Hydrazine Sulfate	100 g	742–26
Water, demineralized	4 L	272–56

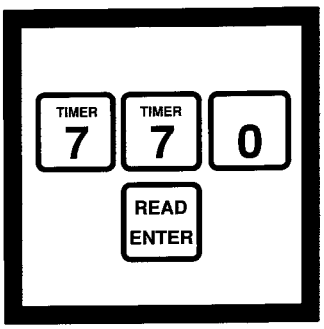
OPTIONAL APPARATUS

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
Pipet Filler, safety bulb	each	14651–00
Pipet, volumetric, Class A, 5.0 mL	each	14515–37
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 additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

VOLATILE ACIDS (0 to 2800 mg/L)

Esterification Method*



1. Enter the stored program number for volatile acids as acetic acid.

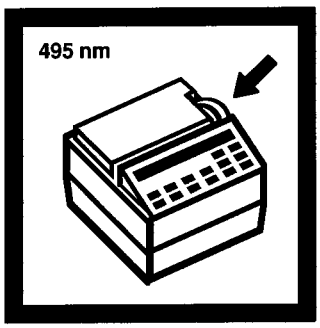
Press: **7 7 0 READ/ENTER**

The display will show:
DIAL nm TO 495

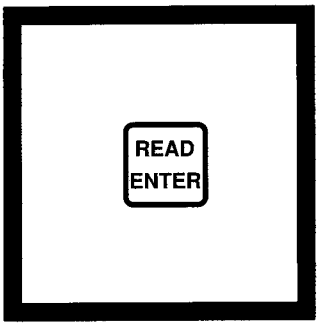
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: If the sample cannot be analyzed immediately, see Sampling and Storage *below. Adjust the pH of stored samples before analysis.*

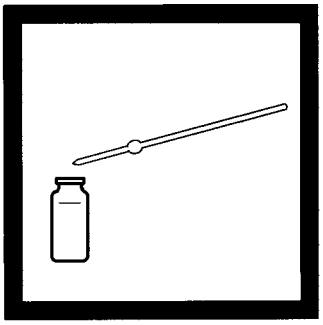


2. Rotate the wavelength dial until the small display shows:
495 nm



3. Press: **READ/ENTER**

The display will show:
mg/l VOL. ACID

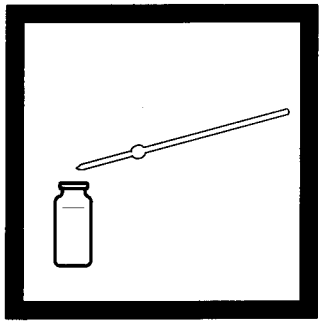


4. Pipet 0.5 mL of demineralized water into a dry 25–mL sample cell (the blank).

*Adapted from *The Analyst*, 87 949 (1962)

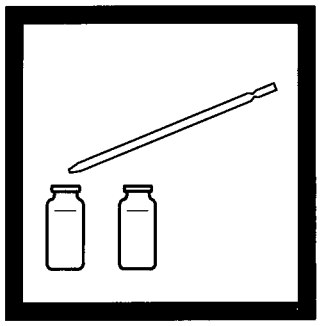
5. Filter or centrifuge 25 mL of the sample using labware listed under *Required Apparatus.*

Note: *Centrifugation is faster than filtration.*

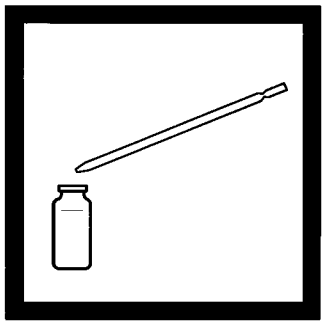


6. Pipet 0.5 mL of the filtrate or supernatant into another dry 25–mL sample cell (the prepared sample).

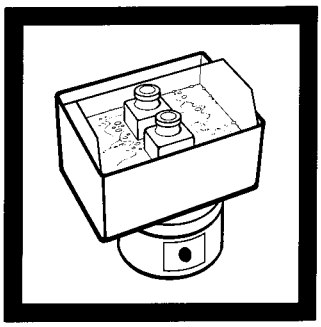
Note: *For proof of accuracy, use 0.5 mL of a 500 mg/L volatile acid solution (preparation given in the* Accuracy Check*) in place of the sample.*



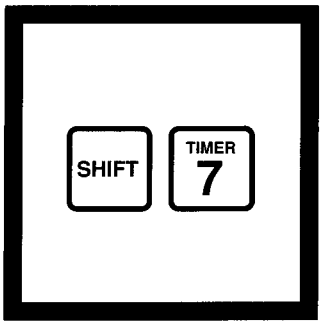
7. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



8. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.

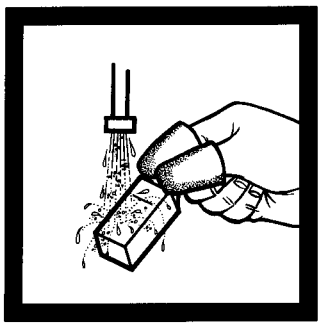


9. Place both cells into a boiling water bath.

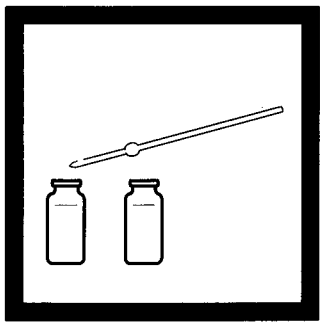


10. Press: **SHIFT TIMER**

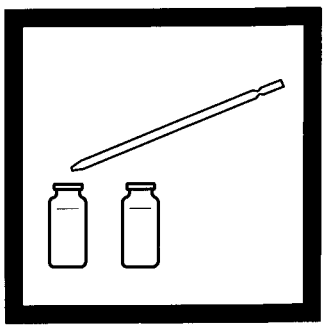
A 3–minute reaction period will begin.



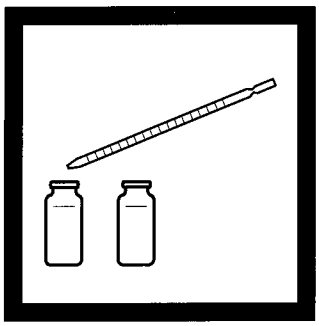
11. When the timer beeps, cool solutions to 25 °C (until cell feels cold) with running tap water.



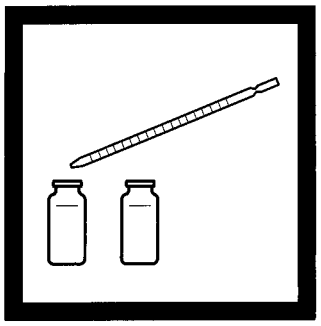
12. Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



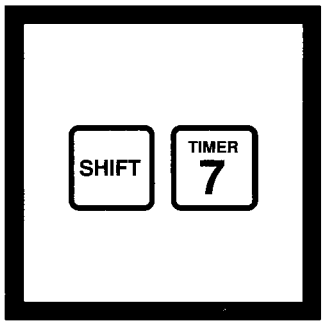
13. Pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Swirl to mix.



14. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Swirl to mix.



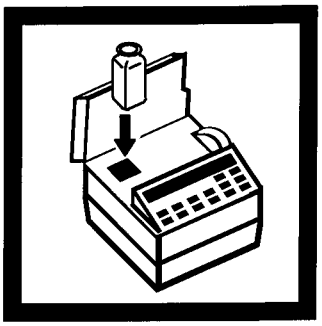
15. Add 10 mL of demineralized water to each cell. Swirl to mix.



16. Press: **SHIFT TIMER**

A 3–minute reaction period will begin.

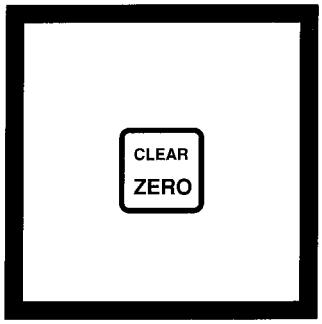
Note: *After this three–minute reaction period proceed immediately through the Steps 17–20 of this procedure.*



17. When the timer beeps, the display will show:

mg/l VOL. ACID

Immediately place the blank into the cell holder. Close the light shield.



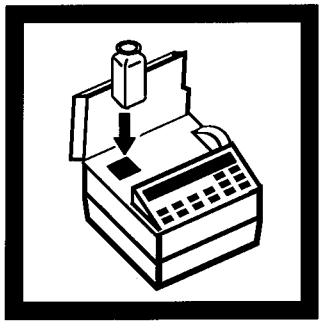
18. Press: **ZERO**

The display will show:

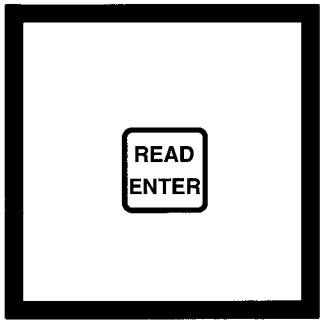
WAIT

then:

0. mg/l VOL. ACID



19. Place the prepared sample into the cell holder. Close the light shield.



20. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L volatile acids as acetic acid will be displayed.

Note: *In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.*

SAMPLING AND STORAGE

Collect samples in plastic or glass bottles. Analyze sample 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 running the test.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Volatile Acids Voluette 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 to three 25–mL graduated mixing cylinders 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 a sample cell. All three samples can be analyzed along with the original test sample beginning with Step 7 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 I* for more information.

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 Voluette Ampule Standard Solution to a 100–mL volumetric flask. Dilute to volume with demineralized water.

PRECISION

In a single laboratory, using a standard solution of 1550 mg/L volatile acids as acetic acid and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±34 mg/L.

SUMMARY OF METHOD

The volatile acids test is designed specifically for the determination of volatile acids in digestor 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.

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 Per Test	Units	Cat. No.
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, demineralized	20.5 mL	4 L	272–56

REQUIRED APPARATUS

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, 3–1/2" diam.	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
Water Bath and Rack	1	each	1955–55

OPTIONAL REAGENTS

Volatile Acids Standard Solution, Voluette ampule, 62,500 mg/L as acetic acid, 10 mL	16/pkg	14270–10
Water, demineralized	4 L	272–56

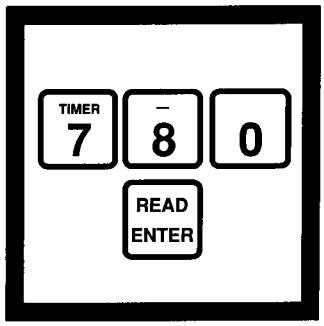
OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968–00
Cylinder, graduated, mixing, 25 mL	each	1896–40
Pipet, TenSette, 0.1 to 1.0 mL	each	19700–01
Pipet Tips, for 19700–01 TenSette Pipet	50/pkg	21856–96
Sample Cells, matched pair	each	20950–00

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

ZINC (0 to 2.00 mg/L)

Zincon Method*; USEPA approved for wastewater analysis (digestion is required; see *Section I*)**



1. Enter the stored program number for zinc (Zn).

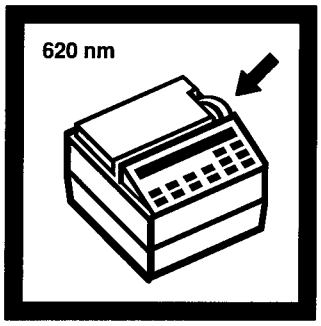
Press: **7 8 0 READ/ENTER**

The display will show:
DIAL nm TO 620

Note: *If samples cannot be analyzed immediately, see* Sampling and Storage *following these steps. Adjust pH of stored samples before analysis.*

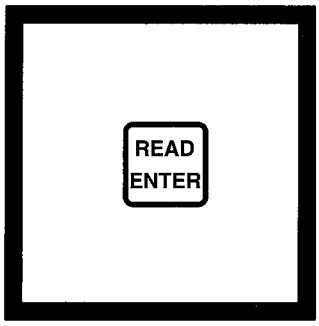
Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.*



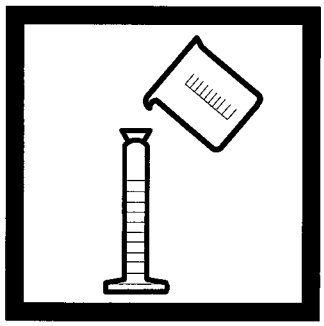
2. Rotate the wavelength dial until the small display shows:
620 nm

Note: *Total zinc determination needs a prior digestion; use either the Digesdahl or mild digestion (*Section I*). Adjust the digested sample to 4 to 5 pH; see* Sampling and Storage *following these steps.*



3. Press: **READ/ENTER**

The display will show:
mg/l Zn

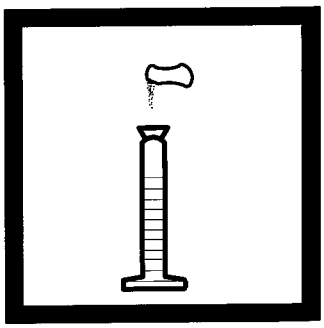


4. Fill a 50–mL graduated mixing cylinder to the 50–mL mark with sample.

Note: *Use only glass stoppered cylinders in this procedure. Rinse with 1:1 Hydrochloric Acid and demineralized water before use.*

Note: *For proof of accuracy, use a 0.5 mg/L zinc standard solution (preparation given in the* Accuracy Check*) in place of the sample.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*
**Federal Register,* 45 (105) 36166 (May 29, 1980)



5. Add the contents of one ZincoVer 5 Reagent Powder Pillow. Stopper. Invert several times to completely dissolve powder.

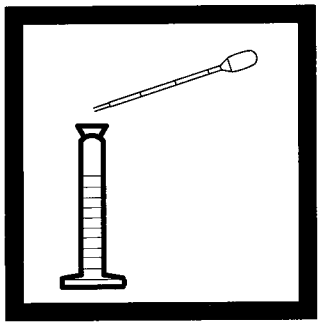
Note: Inconsistent readings may result for low zinc concentrations if all the particles are not dissolved.

Note: At this point the sample color should be orange. If the color is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high, or an interfering metal is present.

Caution: This reagent contains cyanide and is very poisonous if taken internally or inhaled. Do not add to an acidic sample. Store away from water and acids.

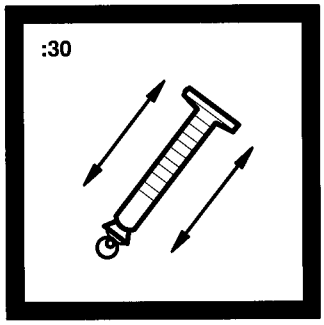
6. Measure 25 mL of the solution into a sample cell (the blank).

Note: The Pour–Thru Cell cannot be used with this procedure.



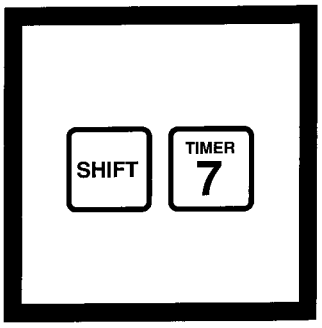
7. Add 1.0 mL of cyclohexanone to the remaining solution in the cylinder.

Note: Use a plastic dropper, as rubber bulbs may contaminate the cyclohexanone.



8. Stopper the cylinder (the prepared sample). Shake for 30 seconds.

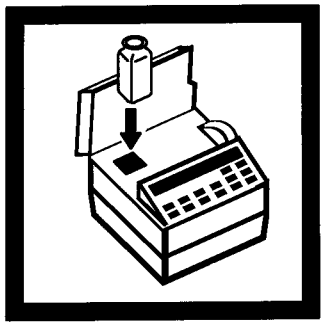
Note: The sample color will be reddish–orange, brown or blue, depending on the zinc concentration.



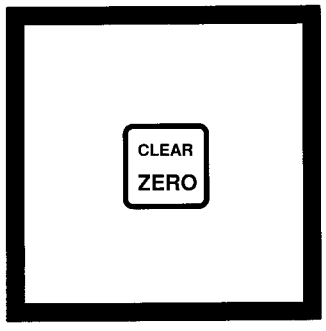
9. Press: **SHIFT TIMER**

A 3–minute reaction period will begin.

10. Pour the solution from the cylinder into a sample cell.



11. When the timer beeps, place the blank into the cell holder. Close the light shield.



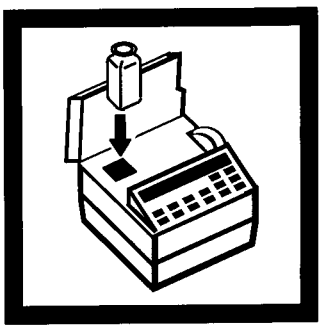
12. Press: **ZERO**

The display will show:

WAIT

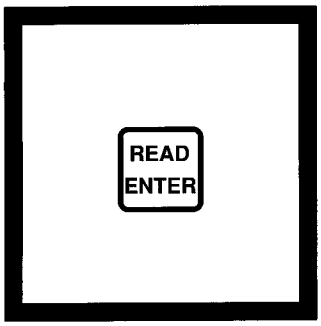
then:

0.00 mg/l Zn



13. Within ten minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.

Note: If more than five minutes elapse after the timer beeps, ZERO SAMPLE may appear. Remove the prepared sample. Insert the blank. Press: ZERO. Insert the prepared sample.



14. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L zinc will be displayed.

Note: In the constant–on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

Note: Determine a reagent blank for each lot of reagent by running the procedure on demineralized water. Subtract this value from all following results obtained in Step 14.

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 for 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, (Section I)* for more information. If only dissolved zinc is to be determined, filter the sample before acid addition.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Zinc Voluette Ampule Standard Solution, 25 mg/L.

b) Use the TenSette Pipet to add 0.2, 0.4, and 0.6 mL of standard to three 50–mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.2 mL of standard added.

d) If these increases do not occur, see Standard Additions in Section I for more information.

Standard Solution Method

Prepare a 0.5 mg/L zinc standard solution by diluting 0.50 mL of zinc standard solution, 100 mg/L as Zn, to 100 mL with demineralized water. Prepare this solution daily.

PRECISION

In a single laboratory, using a standard solution of 1.00 mg/L zinc and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ±0.008 mg/L zinc.

INTERFERENCES

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

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

Large amounts of organic material may interfere. Perform the mild digestion (*Section I*), 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 I).*

WASTE MANAGEMENT

Collect all cyanide–containing waste for proper disposal. To prevent release of hydrogen cyanide gas, store cyanide wastes in a strong solution of sodium hydroxide. In the event of a spill or release, clean up the area by the following 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.

SUMMARY OF METHOD

Zinc and other metals in the sample are complexed with cyanide. The addition of cyclohexanone causes a selective release of zinc. The zinc then reacts with 2–carboxy–2'–hydroxy–5'–sulfoformazyl benzene (zincon) indicator. The zinc concentration is proportional to the resulting blue color.

REQUIRED REAGENTS

			Cat. No.
Zinc Reagent Set (100 Tests*)			22448–00
Includes: (1) 14033–32, (4) 14032–68			
Description	**Quantity Required Per Test**	**Units**	**Cat. No.**
Cyclohexanone	1 mL	100 mL MDB	14033–32
ZincoVer 5 Reagent Powder Pillows	1 pillow	25/pkg	14032–68

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968–00
Cylinder, graduated, mixing, 50 mL	1	each	196–41

OPTIONAL REAGENTS

Bleach, household	1 gal	obtain locally
Hydrochloric Acid, 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	59 mL** SCDB	2450–26
Sodium hydroxide, 50% w/w	500 mL	2180–49
Water, demineralized	4 L	272–56
Zinc Standard Solution, 100 mg/L	100 mL	2378–42
Zinc Standard Solution, Voluette ampule, 25 mg/L as Zn, 10 mL	16/pkg	14246–10

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968–00
Aspirator, vacuum	each	2131–00
Beaker, glass, 1000 mL	each	500–53
Cylinder, graduated, 100 mL	each	508–42
Dropper, plastic, 0.5 & 1.0 mL	10/pkg	21247–10
Filter discs, glass, 47 mm	100/pkg	2530–00
Filter holder, 47 mm	each	2340–00
Flask, erlenmeyer, 250 mL	each	505–46
Flask, volumetric, Class A, 100 mL	each	14574–42
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, EC10, portable	each	50050–00
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, volumetric, Class A, 5.00 mL	each	14515–37
Pipet, volumetric, Class A, 0.5 mL	each	14515–34

For additional ordering information, see final section.
In the U.S.A. call 800–227–4224 to place an order.

*100 Tests equals 100 samples and 100 blanks.
**Contact Hach for larger sizes.