

Only the low range and high range vials are approved for use with method 8000.

OXYGEN DEMAND, CHEMICAL 0 to 40, 0 to 150, 0 to 15,000 mg/L COD For water, wastewater, seawater

Reactor Digestion Method*, EPA Approved†



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.



2. Turn on the COD Reactor. Preheat to 150 °C. Place the plastic shield on 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 of the desired range.

Sample Concentration Range (mg/L)	COD Digestion Reagent Vial Type
0 to 40	Ultra Low Range‡
0 to 150	Low Range
0 to 1,500	High Range
0 to 15,000	High Range Plus

Note: The reagent mixture is light sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The 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 that have been spilled. If some spills, wash with running water.

Note: For proof of accuracy, use COD standard solutions (preparation given in the accuracy check in place of the sample.

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

†Federal Register, 45(78) 26811-26812 (April 21, 1980)

‡The Ultra Low Range COD Vials are not EPA approved and may be used only with spectrophotometers with 350 nm capability

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OXYGEN DEMAND, CHEMICAL, continued

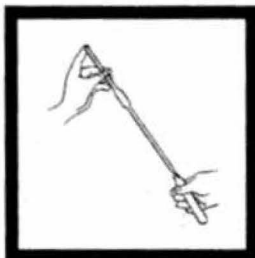


5. Replace the vial cap tightly. Rinse the COD vial with demineralized water and wipe the vial clean with a paper towel.



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.

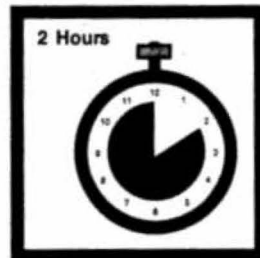
Note: The vial will become very hot during mixing.



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) deionized 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 2 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.

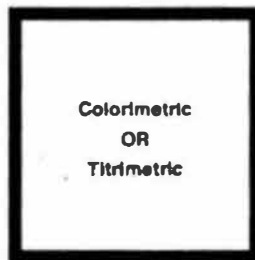


9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.



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 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:

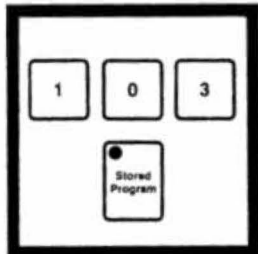
COLORIMETRIC DETERMINATION
0 to 40 mg/L COD
0 to 150 mg/L COD
0 to 1,500 mg/L COD
0 to 15,000 mg/L COD
BURET TITRATION
0 to 150 mg/L COD
0 to 1,500 mg/L COD
0-15,000 mg/L COD

OXYGEN DEMAND, CHEMICAL, continued

COLORIMETRIC DETERMINATION, 0 to 40 mg/L COD

Method 8328

For use with a DR/3000 Spectrophotometer*



1. To begin the stored program for this test, initiate the program mode by making the following keystrokes:

Press: 1 0 3

The STORED PROGRAM light will come on and a wavelength setting of 350.0 for COD testing will appear in the display. The SET WAVELENGTH and ZERO prompting lights will flash.

Note: For DR/3000 instruments with software versions prior to version 4.2, see instrument setup.



2. Turn the wavelength selector dial, located on the upper right corner of the instrument, to a setting of 350.

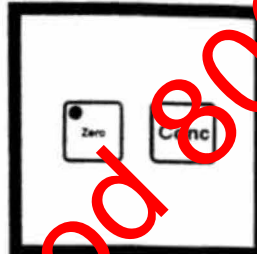
Press: Clear

The SET WAVELENGTH prompting light will turn off and the display will read 0. The ZERO prompting light will continue to flash until the instrument is zeroed in Step 4.



3. Place the COD Vial Adapter in the cell holder of the spectrophotometer with the groove in the adapter facing the front of the instrument. Wipe the vial containing the reagent blank clean with a wet towel followed by a dry towel. Insert the vial containing the reagent blank in the adapter with the Hach logo centered in the groove and cover the light shield.

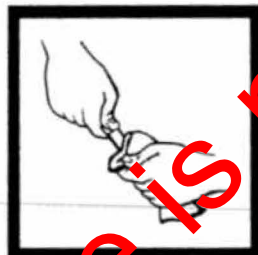
Note: See the Blanks for Colorimetric Determination Section.



4. This reagent blank is used to set the zero concentration point. Zero the instrument as follows:

Press: Zero Conc

The ZERO, CONCENTRATION and AUTO UPDATE lights will come on and a concentration value of 0 will appear in the display.



5. Wipe the vial containing the test sample clean with a wet towel followed by a dry one.



6. Place the vial in the adapter, align the Hach logo with the groove, replace the light shield and read the mg/L COD from the display.

Note: If the display flashes 44, the upper limit of the precalibrated range has been exceeded. A flashing -1 value indicates the sample has a high negative absorbance value relative to the blank. High negative values indicate a possible contamination of the blank or a procedure error by the user.

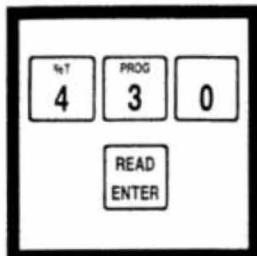
*Other spectrophotometers with 350 nm capability can be used by generating a calibration curve using known KHP standards.

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OXYGEN DEMAND, CHEMICAL, continued

COLORIMETRIC DETERMINATION, 0 to 150 mg/L COD

Method 8000



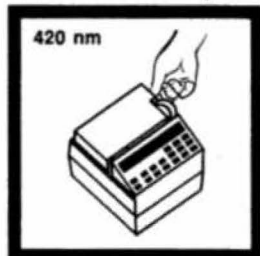
1. Enter the stored program number for chemical oxygen demand (COD), low range.

Press: 4 3 0 READ/ENTER

The display will show:
DIAL nm TO 420

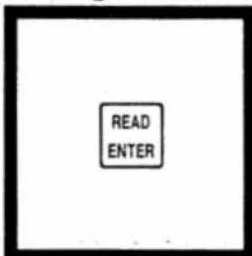
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

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

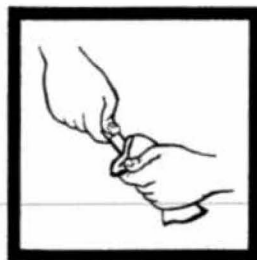
Note: Approach the wavelength setting from the higher to lower values.



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.

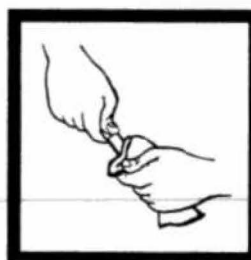


6. Place the blank into the adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.

Note: The blank is stable when stored in the dark; see Blanks for Colorimetric Determination Section.



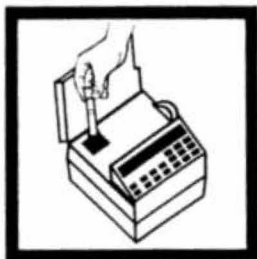
7. Press: ZERO
The display will show:
WAIT
then:
0. mg/l COD L



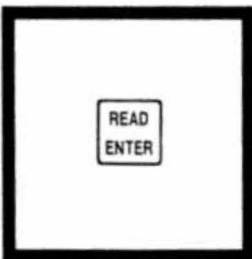
8. Clean the outside of the sample vial with a towel.

Only the low range and high range vials are approved for use with method 8000.

OXYGEN DEMAND, CHEMICAL, continued



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

Method 8000



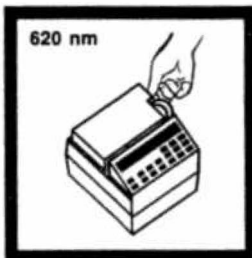
1. Enter the stored program number for chemical oxygen demand, high range.

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

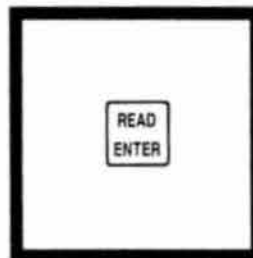
The display will show:
DIAL nm TO 620

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.

Only the low range and high range vials are approved for use with method 8000.

OXYGEN DEMAND, CHEMICAL, continued



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 Section.



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 10.

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.

Only the low range and high range vials are approved for use with method 8000.

OXYGEN DEMAND, CHEMICAL, continued

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

Method 8000



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

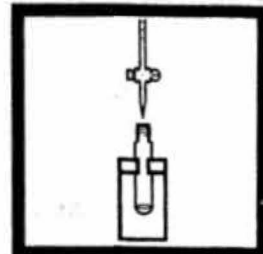


2. Add a small Teflon-coated stirring bar and one drop of the appropriate Ferrous Indicator Solution. When using the Low Range COD Digestion Reagent Vials, use Low Range Ferrous Indicator Solution. When using the High Range or High Range Plus COD Digestion Reagent Vials, use High Range Ferrous Indicator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



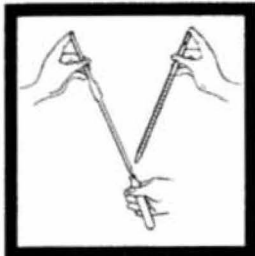
4. Titrate with the appropriate Ferrous Ammonium Sulfate Standard Solution (FAS) until the sample color changes sharply from greenish-blue to orange-brown. When using the Low Range COD Digestion Reagent Vials, use 0.0125 N FAS. When using the High Range or High Range Plus COD Digestion Reagent Vials, 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 bottle well before using.

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

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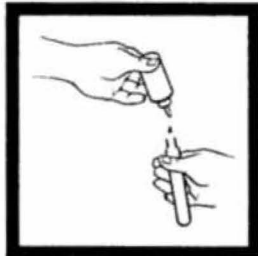
OXYGEN DEMAND, CHEMICAL, continued



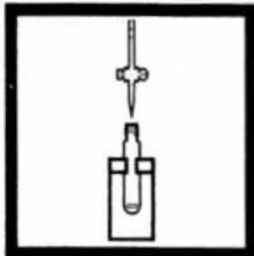
5. Pipet 2.00 mL of Potassium Dichromate Standard Solution into an empty vial. When using the Low Range COD Digestion Reagent Vials, use a 0.025 N solution. When using High Range or High Range Plus COD Digestion Reagent Vials, use a 0.25 N solution.

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: Steps 5 through 7 need only be done 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 bottle 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.

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

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

COD mg/L =

$$(3.95 - 2.00) \times \frac{2000}{4.00} \times 0.1 = 97.5$$

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OXYGEN DEMAND, CHEMICAL, continued

INSTRUMENT SETUP

For a DR/3000 with a software version prior to version 4.2, manually enter the Ultra Low Range COD (0-40 mg/L) procedure program by making the following entries:

1. Press:



The Zero, Conc Factor and Standard 1 lights will flash.

2. Set the wavelength by turning the wavelength selector dial, located on the upper right corner of the instrument, to a setting of 350.0 nm.

3. Place the COD Vial Adapter in the cell holder of the spectrophotometer with the groove in the adapter facing the front of the instrument. Wipe the vial containing the reagent blank clean. Insert the vial in the adapter with the Hach logo centered on the groove and cover with the light shield.

4. The reagent blank is used to set zero absorbance. Zero the instrument as follows:

Press:



5. When measuring the vials at room temperature, enter the following concentration factor. This factor will change if the vials are measured hot.

Press:



Press:



Wipe the vial containing the test sample with a paper towel. Place the vial in the adapter, align the Hach logo with the groove, replace the light shield and set the instrument into the concentration mode.

Press:



Concentration values can now be read directly from the instrument display. Periodically re-zero the instrument with the blank solution.

Note: At very low COD values (1-5 mg/L), run three to five replicates, and average the results. Good technique and the elimination of contamination are necessary for accurate results.

*Note: After the digestion is complete and while the vials are still hot (150 °C), the samples can be read directly from the digestion block. This will save the time allotted for the samples to cool. The method is entered into the DR/3000 using the same procedure as used for room temperature samples above with the following exceptions. The blank used to zero the instrument is hot (150 °C) and the concentration factor is -53.6. Samples can now be read directly from the digestion block. **Caution!** The sample vials are hot and must be handled by the caps only.*

Note: If the display flashes 44, the upper limit of the precalibrated range has been exceeded. A flashing -1 value indicates the sample has a high negative absorbance value relative to the blank. High negative values indicate a possible contamination of the blank or a procedure error by the user.

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. For more information see Volume Additions in Section I.

ACCURACY CHECK

- Check the accuracy of the 0 to 40 mg/L range with a 30 mg/L COD standard solution. Prepare by diluting 3.00 mL of a 1000 mg/L COD standard solution to 100.0 mL. The 1000 mg/L COD standard solution can be prepared by dissolving 850 mg of oven dried (120 °C, overnight) potassium acid phthalate (KHP) in deionized water and diluting to 1 L with high quality deionized water.
- Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2 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 deionized water.

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OXYGEN DEMAND, CHEMICAL, continued

- Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD. Prepare this solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water.

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. Samples with higher chloride concentrations should be diluted. 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) Suggested Cl ⁻ concentration of diluted samples (mg/L)	(3) Maximum Cl ⁻ concentration in sample when 0.50 g HgSO ₄ added (mg/L)
Ultra Low Range	2000	1000	---
Low Range	2000	1000	8000
High Range	2000	1000	4000
High Range Plus	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 HgSO₄ 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 (350, 420 or 620 nm). Zero the instrument in the absorbance mode, using a vial containing deionized 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 O₂ consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for 2 hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion (Cr₂O₇²⁻) to green chromic ion (Cr³⁺). When the 0-40 mg/L colorimetric method or the 0-150 mg/L colorimetric or titrimetric method is used, the amount of Cr⁶⁺ remaining is determined. When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr³⁺ produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. For more information, see *Chemical Procedures Explained*, Appendix A.

REQUIRED REAGENTS (For Colorimetric Analysis)

Description	Quantity Required Per Test	Unit	Cat. No.
Pick the appropriate COD Digestion Reagent Vial			
Ultra Low Range, 0 to 40 mg/L COD	1 to 2 vials	25/pkg	24158-25
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	3.78 L	272-17

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OXYGEN DEMAND, CHEMICAL, continued

REQUIRED REAGENTS (For Buret Titration):

Pick one or both Potassium Dichromate Standard Solutions

0.025 N	2 mL	473 mL	164-11
0.25 N	2 mL	946 mL	1809-16
Sulfuric Acid, ACS	3 mL	500 mL*	979-49
Water, demineralized	varies	946 mL*	272-16

Pick 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

Pick one or both Ferrioin Indicator Solutions

Low Range	1 to 2 drops	29 mL DB	20551-33
High Range	1 to 2 drops	29 mL DB	1812-33

Pick one or both Ferrous Ammonium Sulfate Standard Solutions†

0.0125 N	varies	946 mL	14237-16
0.125 N	varies	500 mL	20548-49

REQUIRED APPARATUS (For Colorimetric Analysis)

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
Safety Shield, laboratory bench	1	each	23810-00
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
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
Safety Shield, laboratory bench	1	each	23810-00
Stir Bar	1	each	20549-59
Stir Bar Retriever	1	each	15232-00
Stirrer, magnetic, 120 Vac, 60 Hz	1	each	23444-00
Support Stand	1	each	563-00
Test Tube Rack, 8 place	1 (2 recommended)	each	18641-00
Titration Stand, test tube	1	each	18642-00

*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.

‡Does not include reagent or apparatus for reagent blanks or standardization.

Only the low range and high range vials are approved for use with method 8000.

OXYGEN DEMAND, CHEMICAL, continued

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	500 g	315-34
Sulfuric Acid, ACS	500 mL*	979-49
Mercuric Sulfate	28 g*	1915-20
Potassium Dichromate Standard Solution, 0.250 N	946 mL*	1809-16

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
Spoon, measuring, 0.5 g	each	907-00
Stir Bar, 7/8" x 3/16"	each	45315-00
Stir Bar Retriever	each	15232-00

For Technical Assistance, Price and Ordering

*In the U.S.A.—Call 800-227-4224 toll-free for more information
Outside the U.S.A.—Contact the Hach office or distributor serving you.*