



Method 8000

Reactor Digestion Method*

(0 to 40.0** mg/L COD)

Scope and Application: For water, wastewater and seawater; digestion is required.
The estimated detection limit for program number 2700 is 0.2 mg/L COD.

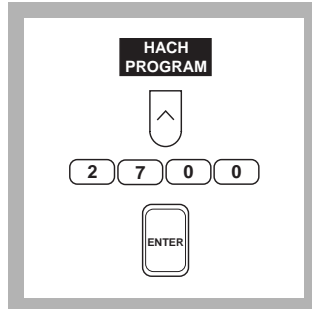
* Jirka, A.M.; Carter, M.J., *Analytical Chemistry*, 1975, 47(8), 1397

** Ultra low range vials are not USEPA approved and may be used only with spectrophotometers with 350 nm capability.

Colorimetric Measurement, 0 to 40 mg/L COD



1. Perform the digestion for this method as described in “Oxygen Demand, Chemical, Digestion Procedure” which precedes the COD colorimetric procedures.



2. Press the soft key under **HACH PROGRAM**.
Select the stored program number for ultra low range COD by pressing **2700** with the numeric keys.
Press: **ENTER**



3. The display will show:
**HACH PROGRAM: 2700
COD, ULR**
The wavelength (λ), **350 nm**, is automatically selected.

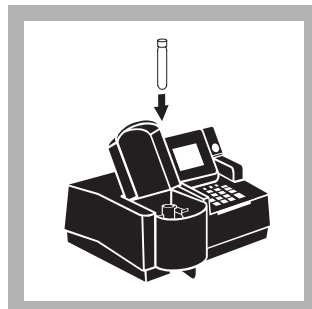


4. Insert the Test ‘N’ Tube Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

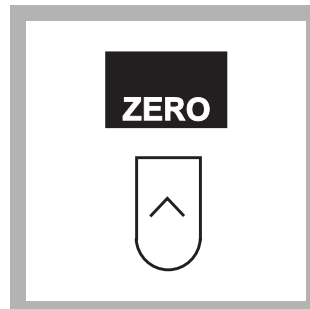
Note: The Test Tube Adapter is **NOT** designed to allow readings on hot vials (150 °C).



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.



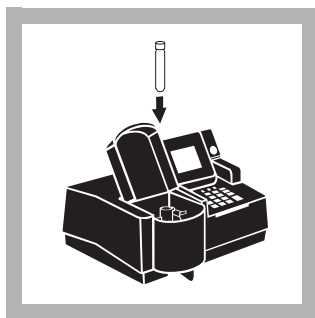
6. Place the blank into the adapter with the Hach logo facing the front of the instrument. Close the light shield.
Note: Preparation of the blank is described in the digestion procedure.
Note: The blank is stable when stored in the dark; see Blanks for Colorimetric Measurement following these procedures.



7. Press the soft key under **ZERO**.
The display will show:
0.0 mg/L COD
Note: For alternate concentration units press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



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. Close the light shield. Results in mg/L COD (or chosen units) will be displayed.

Note: Results may be expressed as mg/L COD or mg/L O₂. Press the soft keys under **OPTIONS** and then press **FORM**: to scroll through the available choices.

Note: If the display shows **45 mg/L COD** and/or **OVER!**, the upper limit of the range has been exceeded. Repeat the test with a dilute sample or use a Low Range or High Range COD Reagent Vial.

Interferences

Chloride

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 2. Samples with higher chloride concentrations should be diluted. Dilute the sample enough to reduce the chloride concentration to the level given in column 3.

Table 1 Interfering Substances and Suggested Treatments

Vial Type Used	Maximum Cl ⁻ concentration in sample (mg/L)	Suggested Cl ⁻ concentration of diluted sample (mg/L)	Maximum Cl ⁻ concentration in sample with 0.5 g HgSO ₄ Added (mg/L)
Ultra Low Range	2000	1000	NA
Low Range	2000	1000	8000
High Range	2000	1000	4000
Ultra High Range	20,000	10,000	40,000

If sample dilution will cause the COD concentration to be too low for accurate measurement, add 0.50 g of mercuric sulfate (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 4.

Bromide

Bromide interference will not be controlled by mercuric sulfate.

Sample Collection, Preservation 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 Section 1.2.2 *Correcting for Volume Additions*.

Accuracy Check

Standard Solution Method

Check the accuracy of the 0 to 40 mg/L range with a 30 mg/L standard. Using Class A glassware, prepare a 1000-mg/L solution by diluting 850 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1000 mL of organic-free deionized water. Prepare a 30 mg/L dilution by diluting 3.00 mL of this solution into a 100.0 mL volumetric flask. Dilute to volume with deionized water, stopper, and invert 10 times to mix.

Method Performance

Precision

Standard: 30.0 mg/L COD

Program	95% Confidence Limits
2700	29.9–30.1 mg/L COD

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
2700	0.2 mg/L COD

For more information on derivation and use of Hach's estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, Appendix B, see Section 1.5.1.

Sensitivity

Program Number: 2700

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	-0.52 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Preparing Organic-Free Water

Preparing organic-free water with no measurable COD:

1. Pour 1.0 liter of deionized water with low COD in a 2-liter erlenmeyer flask.
2. Add the contents of one Potassium Persulfate Powder Pillow to the flask. Swirl to dissolve.

3. Suspend a UV lamp in the flask so the glass portion of the bulb is immersed and the black bakelite portion is above the solution. Follow the safety and operation instructions recommended in the UV lamp kit. Safety UV goggles should be worn for eye protection.
4. Irradiate the solution with UV light for at least two hours (overnight is fine).
5. Remove the lamp from the solution. Add one level 0.05-gram scoop of Nickel Sulfate to the solution.
6. Heat the water to a boil. Remove the flask from the hot plate and cover it with a watch glass.
7. Let the flask cool to room temperature. The water will have zero oxygen demand. Seal the flask top with aluminum foil to prevent organic contamination. The water should stay free of oxygen demand for one week if properly sealed.

Blanks for Colorimetric Measurement

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 350 nm. Zero the instrument in the absorbance mode, using a culture tube (see *OPTIONAL EQUIPMENT AND SUPPLIES*) containing 5 mL of deionized water. Measure the absorbance of the blank and record the value. Prepare a blank when the absorbance has changed by about 0.010 absorbance units.

Calibration Standard Preparation

To perform an ultra low range calibration using the reactor digestion method, prepare a 500-mg/L COD stock solution by pipetting 50.00 mL of a 1000-mg/L COD Standard Solution (Cat. No. 22539-29) into a 100-mL volumetric flask using Class A glassware. Dilute to the mark with organic-free deionized water and mix thoroughly.

Prepare calibration standards containing 5, 15, 25, 35 and 40 mg/L COD as follows:

- a. Into five different Class A 100-mL volumetric flasks, pipet 1.0, 3.0, 5.0, 7.0 and 8.0 mL of the 500-mg/L COD stock solution using Class A glassware.
- b. Dilute to the mark with deionized water. Stopper and invert 10 times to mix.
- c. Using the ultra low range reactor digestion method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

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 two 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 or 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.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to Section 3.

Pollution Prevention and Waste Management

Final samples will contain mercury (D009), silver (D011), and chromium (D007) at concentration levels regulated as hazardous waste by the Federal RCRA. Please see Section 1 for further information on proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS

Description	Quantity Required per test	Unit	Cat. No.
Ultra Low Range COD Digestion Vials, 0 to 40 mg/L COD.....	1 to 2 vials.....	25/pkg.....	24158-25
Water, deionized.....	varies.....	4 liters.....	272-56

REQUIRED EQUIPMENT AND SUPPLIES

COD Reactor, 115/230 VAC, North American plug.....	1.....	each.....	45600-00
COD Reactor, 230 VAC, 50 Hz, European plug.....	1.....	each.....	45600-02
Description	per test	Unit	Cat. No.
DR/4000 Test Tube Adapter.....	1.....	each.....	48189-00
Pipet, volumetric, Class A, 2.00-mL.....	1.....	each.....	14515-36
Pipet Filler, safety bulb.....	1.....	each.....	14651-00
Test Tube Rack.....	1-2.....	each.....	18641-00

OPTIONAL REAGENTS AND STANDARDS

COD Digestion Reagent Vials, 0 to 40 mg/L COD.....	150/pkg.....	24158-15
COD Digestion Reagent Vials, 0 to 150 mg/L COD.....	150/pkg.....	21258-15
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD.....	150/pkg.....	21259-15
COD Standard Solution, 300-mg/L.....	200 mL.....	12186-29
COD Standard Solution, 1000-mg/L.....	200 mL.....	22539-29
Mercuric Sulfate, ACS.....	28 g*.....	1915-20
Nickel Sulfate, ACS.....	25 g.....	11264-24
Potassium Acid Phthalate, ACS.....	500 g.....	315-34
Potassium Persulfate Powder Pillows.....	100/pkg.....	20847-69
Sulfuric Acid, ACS, concentrated.....	500 mL*.....	979-49

* Contact Hach for larger sizes.

OXYGEN DEMAND, Chemical, continued

OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Beaker, 250 mL	each.....	500-46
Culture Tube, 16 x 100 mm, borosilicate glass	each.....	23800-00
Culture Tube Cap (for 23800-00).....	each.....	22411-00
Cylinder, graduated, 5-mL	each.....	508-37
Flask, Erlenmeyer, 2000-mL.....	each.....	505-54
Flask, volumetric, Class A, 100-mL	each.....	14574-42
Flask, volumetric, Class A, 1000-mL	each.....	14574-53
Goggle, safety.....	each.....	21134-00
Hot Plate, 120 VAC	each.....	23441-00
Hot Plate, 240 VAC	each.....	23441-02
pH Paper, pH 1.0 to 11.0	5 rolls/pkg.....	391-33
Pipet, serological, 5-mL	each.....	532-37
Pipet, volumetric, Class A, 1.00-mL.....	each.....	14515-35
Pipet, volumetric, Class A, 3.00-mL.....	each.....	14515-03
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 7.00-mL.....	each.....	14515-07
Pipet, volumetric, Class A, 8.00-mL.....	each.....	14515-08
Pipet, volumetric, Class A, 10.00-mL.....	each.....	14515-38
Pipet, volumetric, Class A, 50.00-mL.....	each.....	14515-41
Safety shield, for COD reactor.....	each.....	23810-00
Spoon, measuring, 0.5-g.....	each.....	907-00
UV Lamp Kit, 115 VAC.....	each.....	20828-00
UV Lamp Kit, 230 VAC.....	each.....	20828-02

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

Title	Literature Code No.
COD Disposal Information Brochure	4144



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
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