OXYGEN DEMAND, Chemical

✓ Method 8000

Reactor Digestion Method*

(0 to 150.0 mg/L COD)

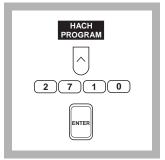
Scope and Application: For water, wastewater and seawater; digestion is required; USEPA Approved** for wastewater analyses. The estimated detection limit for program number 2710 is 1.1 mg/L COD.

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

Colorimetric Measurement



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 low range COD by pressing **2710** with the numeric keys.

Press: ENTER

Note: If samples cannot be digested immediately, see Sample Collection, Preservation and Storage following these steps.



3. The display will show: HACH PROGRAM: 2710 COD, LR

The wavelength (λ) , **420 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.

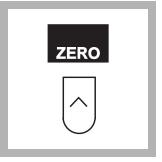
Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.



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.

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



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 **FORM:** to scroll through the available choices.

Note: For most accurate results with samples near 150 mg/L COD, repeat the analysis with a diluted sample. If the display shows 165.0 mg/L COD and an OVER! warning, the working range has been exceeded. Repeat analysis with a diluted sample or the High Range reagent vials.

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 Treatn

Vial Type Used	Maximum CI-concentration in sample (mg/L)	Suggested CI-concentration of diluted sample (mg/L)	Maximum CI ⁻ 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 (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 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 150 mg/L range with a 100-mg/L KHP 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 or, alternatively, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to produce a 100-mg/L standard.

To adjust the calibration curve using the reading obtained with the 100-mg/L standard solution, press the soft keys under **OPTIONS, MORE** then **STD: OFF**. Press **ENTER** to accept the displayed concentration, the value of which depends on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section 1.5.5 Adjusting the Standard Curve for more information.

Method Performance

Precision

Standard: 100.0 mg/L COD

Program	95% Confidence Limits		
2710	99.4–100.6 mg/L O ₂		

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

Estimated Detection Limit

Program	EDL	
2710	1.1 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: 2710

Portion of Curve	∆Abs	∆Concentration		
Entire Range	0.010	-3.45 mg/L		

See Section 1.5.3 Sensitivity Explained for more information.

OXYGEN DEMAND, Chemical, continued

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 420 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 a low range (0-150 mg/L) calibration using the reactor digestion method, use a 1000-mg/L COD Standard Solution (Cat. No. 22539-29).

Prepare calibration standards containing 20, 60, 100, 140 and 160 mg/L COD as follows:

- **a.** Into five different 100-mL volumetric flasks, pipet 2, 6, 10, 14 and 16 mL of the 1000-mg/L COD Standard Solution using Class A glassware.
- **b.** Dilute to the mark with deionized water. Stopper and invert 10 times to mix.
- **c.** Using the 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.

Alternate Reagents—COD2 Reagent Vials

For non-reporting purposes, COD2 Reagent can provide a mercury-free testing option, eliminating mercury waste and saving on disposal costs.

COD2 Reagent Vials use the same COD procedures and the same COD calibration curves programmed into the DR/4000.

COD2 Reagent is not acceptable for USEPA reporting purposes. Request Literature Code 1356 for applications where COD 2 Reagent Vials may be suitable.

Summary of Method

The mg/L COD results are defined as the mg of $\rm O_2$ 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 ($\rm Cr_2O_7^{2-}$) to green chromic ion ($\rm Cr^{3+}$). When the 0–150 mg/L colorimetric is used, the amount of $\rm Cr^{6+}$ remaining is determined. When the 0–1,500 mg/L or 0–15,000 mg/L colorimetric method is used, the amount of $\rm Cr^{3+}$ produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to control 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 1.

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.

Description		Quantity Required				
Water, deionized varies 272-56 REQUIRED EQUIPMENT AND SUPPLIES	•	per test				
REQUIRED EQUIPMENT AND SUPPLIES COD Reactor, 115/230 VAC, North American plug						
COD Reactor, 115/230 VAC, North American plug 1	Water, deionized	varies	4 liters	272-56		
COD Reactor, 115/230 VAC, North American plug 1	DECLUDED EQUIDMENT AND GUDDI IEG					
COD Reactor, 230 VAC, 50 Hz, European plug		1	1	45,000,00		
Pipet, volumetric, Class A, 2.00-mL						
Pipet Filler, safety bulb.	COD Reactor, 230 VAC, 50 Hz, European plug	1	each	45600-02		
Test Tube Rack 1 to 2 racks .each .18641-00 DR/4000 Test Tube Adapter 1 .each .48189-00 OPTIONAL REAGENTS AND STANDARDS Description Unit Cat. No. COD Digestion Reagent Vials, 0 to 150 mg/L COD 150/pkg .21258-15 COD Standard Solution, 300-mg/L .236 mL .12186-31 COD Standard Solution, 1000-mg/L .236 mL .2259-93 COD Standard Solution, 1000-mg/L .236 mL .2259-mL Cob Standard Solution, 1000-mg/L .236 mL .2259-mL COD Standard Solution, 1000-mg/L .236 mL .2259-mL COD Total Standard Solution, 1000-mg/L .236 mL .2278-m OPTIONAL EQUPMENT AND SUPLIES <td <="" colspan="2" td=""><td>Pipet, Volumetric, Class A, 2.00-mL</td><td>1</td><td>eacn</td><td>14515-36</td></td>	<td>Pipet, Volumetric, Class A, 2.00-mL</td> <td>1</td> <td>eacn</td> <td>14515-36</td>		Pipet, Volumetric, Class A, 2.00-mL	1	eacn	14515-36
DR/4000 Test Tube Adapter	Pipet Filler, Safety builb	1 4 - 2 1	eacn	14651-00		
OPTIONAL REAGENTS AND STANDARDS Description Unit Cat. No. COD Digestion Reagent Vials, 0 to 150 mg/L COD 150/pkg 21258-15 COD Digestion Reagent Vials, 0 to 1,500 mg/L COD 150/pkg 21259-15 COD Standard Solution, 300-mg/L 236 mL 12186-31 COD Standard Solution, 1000-mg/L 236 mL 22539-31 COD2 Reagent Vials, Low Range, 0-150 mg/L 25/pkg 25650-25 Mercuric Sulfate, ACS 28 g* 1915-20 Potassium Acid Phthalate, ACS 500 g 315-34 Sulfuric Acid, ACS, concentrated 500 mL* 979-49 OPTIONAL EQUIPMENT AND SUPPLIES Beaker, 250-mL each 500-46H Cylinder, graduated, 5-mL each 500-46H Cylinder, graduated, 5-mL each 508-37 Culture Tube, 16 x 100 mm, borosilicate glass 6/pkg 22758-06 Culture Tube Cap (for 22758-06) 6/pkg 227158-06 Culture Tube Cap (for 22758-06) 6/pkg 32411-06 Flask, volumetric, Class A, 100-mL each 1457-42						
Description Unit Cat. No. COD Digestion Reagent Vials, 0 to 150 mg/L COD 150/pkg 21258-15 COD Digestion Reagent Vials, 0 to 1,500 mg/L COD 150/pkg 21259-15 COD Standard Solution, 300-mg/L 236 mL 12186-31 COD Standard Solution, 1000-mg/L 236 mL 22539-31 COD2 Reagent Vials, Low Range, 0-150 mg/L 25/pkg 25650-25 Mercuric Sulfate, ACS 28 g* 1915-20 Potassium Acid Phthalate, ACS 500 g 315-34 Sulfuric Acid, ACS, concentrated 500 mL* .979-49 OPTIONAL EQUIPMENT AND SUPPLIES Beaker, 250-mL each 500-46H Cylinder, graduated, 5-mL each 508-37 Culture Tube, 16 x 100 mm, borosilicate glass 6/pkg 22758-06 Culture Tube Cap (for 22758-06) 6/pkg 22718-06 Culture Tube Cap (for 22758-06) 6/pkg 22411-06 Flask, volumetric, Class A, 1000-mL each 14574-42 Flask, volumetric, Class A, 1000-mL each 14574-53 pipet, velumetric, Class A, 532-37 each	DR/4000 Test Tube Adapter	1	eacn	48189-00		
Description Unit Cat. No.	OPTIONAL REAGENTS AND STANDARDS					
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 COD2 Reagent Vials, Low Range, 0-150 mg/L 25/pkg 25650-25 Mercuric Sulfate, ACS 28 g* 1915-20 Potassium Acid Phthalate, ACS 500 g 315-34 Sulfuric Acid, ACS, concentrated 500 mL* .979-49 OPTIONAL EQUIPMENT AND SUPPLIES Beaker, 250-mL each .500-46H Cylinder, graduated, 5-mL each .508-37 Culture Tube, 16 x 100 mm, borosilicate glass .6/pkg .22758-06 Culture Tube, 20 (for 22758-06) .6/pkg .22715-06 Flask, volumetric, Class A, 100-mL each .14574-42 Flask, volumetric, Class A, 1000-mL each .14574-53 Pipet, serological, 5-mL each .532-37 Pipet, volumetric, Class A, 2.00-mL each .14515-36 Pipet, volumetric, Class A, 6.00-mL	Description					
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD	COD Digestion Reagent Vials, 0 to 150 mg/L COD		150/pkg	21258-15		
COD Standard Solution, 1000-mg/L 236 mL 22539-31 COD2 Reagent Vials, Low Range, 0–150 mg/L 25/pkg 25650-25 Mercuric Sulfate, ACS 28 g* 1915-20 Potassium Acid Phthalate, ACS 500 g 315-34 Sulfuric Acid, ACS, concentrated 500 mL* 979-49 OPTIONAL EQUIPMENT AND SUPPLIES Beaker, 250-mL each 508-37 Culture Tube, 16 x 100 mm, borosilicate glass 6/pkg 22758-06 Culture Tube Cap (for 22758-06) 6/pkg 22411-06 Flask, volumetric, Class A, 100-mL each 14574-42 Flask, volumetric, Class A, 1000-mL each 14574-53 pH Paper, pH 1.0 to 11.0 5 rolls/pkg 391-33 Pipet, serological, 5-mL each 532-37 Pipet, volumetric, Class A, 2.00-mL each 14515-36 Pipet, volumetric, Class A, 6.00-mL each 14515-06 Pipet, volumetric, Class A, 8.00-mL each 14515-08 Safety shield, for COD reactor each 907-00 RELATED LITERATURE—Ask for your copy by literature code number. Title COD Dispo						
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Pipet, serological, 5-mL each 532-37 Pipet, volumetric, Class A, 2.00-mL each 14515-36 Pipet, volumetric, Class A, 6.00-mL each 14515-06 Pipet, volumetric, Class A, 7.00-mL each 14515-07 Pipet, volumetric, Class A, 8.00-mL each 14515-08 Safety shield, for COD reactor each 50030-00 Spoon, measuring, 0.5-g each 907-00 RELATED LITERATURE—Ask for your copy by literature code number. Title Literature Code No. COD Disposal Information Brochure 4144						
Pipet, volumetric, Class A, 2.00-mL each 14515-36 Pipet, volumetric, Class A, 6.00-mL each 14515-06 Pipet, volumetric, Class A, 7.00-mL each 14515-07 Pipet, volumetric, Class A, 8.00-mL each 14515-08 Safety shield, for COD reactor each 50030-00 Spoon, measuring, 0.5-g each 907-00 RELATED LITERATURE—Ask for your copy by literature code number. Title Literature Code No. COD Disposal Information Brochure 4144						
Pipet, volumetric, Class A, 6.00-mL each 14515-06 Pipet, volumetric, Class A, 7.00-mL each 14515-07 Pipet, volumetric, Class A, 8.00-mL each 14515-08 Safety shield, for COD reactor each 50030-00 Spoon, measuring, 0.5-g each 907-00 RELATED LITERATURE—Ask for your copy by literature code number. Title Literature Code No. COD Disposal Information Brochure 4144						
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Safety shield, for COD reactor						
Spoon, measuring, 0.5-g	•					
RELATED LITERATURE—Ask for your copy by literature code number. Title COD Disposal Information Brochure	•					
TitleLiterature Code No.COD Disposal Information Brochure4144	Spoon, measuring, 0.5-g	•••••	each	907-00		
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^{*} Contact Hach for larger sizes.

