✓ Method 8000

OXYGEN DEMAND, Chemical

Reactor Digestion Method*

(For all ranges)

Scope and Application: For water, wastewater and seawater. USEPA Approved** for wastewater analyses

* Jirka, A.M.; Carter, M.J., Analytical Chemistry, 1975, 47(8), 1397 ** Federal Register, April 21, 1980, 45(78), 26811–26812

DR/4000

PROCEDURE

Digestion



1. Homogenize 100 mL of sample for 30 seconds in a blender.

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

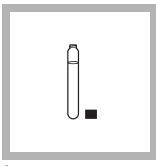
Note: Mix the sample prior to homogenization. To improve accuracy and reproducibility, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate. For samples containing large amounts of solids, increase the homogenization time.

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



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

Note: 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 40	Ultra Low Range
0 to 150	Low Range
0 to 1,500	High Range
0 to 15,000	Ultra High Range

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.

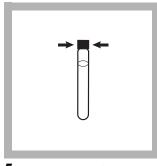
Note: Pipet only 0.20 mL of sample, not 2.00 mL, using a TenSette Pipet. For greater accuracy a minimum of three replicates should be analyzed and the results averaged.

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 some spills, wash with running water.

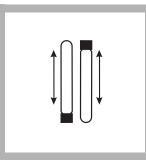
Note: For proof of accuracy, use COD standard solutions (preparation given in the Accuracy Check section of the individual colorimetric procedure) in place of the sample.

Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. 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.

OXYGEN DEMAND, Chemical, continued



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



6. Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated 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. Place the blank in the COD Reactor.

Note: Use a clean or wellrinsed 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.



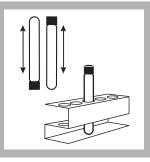


8. Heat the vials for 2 hours.

Note: Although many wastewater samples are digested completely in less than two hours, the DR/4000 Test Tube Adapter is NOT designed to measure hot vials (150 °C). If preliminary readings are desired, cool vial before reading.



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 or alternate reagent vial. Colorimetric or Titrimetric

11. Use one of the analytical techniques in *Method 8140, Iron Reduction Method for Oxygen Scavengers* to determine the sample concentration:

Colorimetric measurement, 0 to 40 mg/L COD

Colorimetric measurement, 0 to 150 mg/L COD

Colorimetric measurement, 0 to 1,500 mg/L COD and 0 to 15,000 mg/L COD

Note: A titrimetric procedure is also available. Contact Hach Customer Service for details.

Sample Collection, Preservation and Storage

Refer to the appropriate COD colorimetric measurement procedure.

Summary of Method

Refer to the appropriate COD colorimetric measurement procedure.

REQUIRED REAGENTS AND STANDARDS

	Quantity Required		
Description	per test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Ultra Low Range, 0 to 40 mg/L	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, deionized	varies	4 liters	

REQUIRED EQUIPMENT AND SUPPLIES

	Quantity Required		
Description	per test	Unit	Cat. No
Blender, 2 speed, 120 VAC	1	each	
Blender, 2 speed, 240 VAC	1	each	
Cap Tool, COD	1	each	45587-00
COD Reactor, 115/230 VAC, North American Plug		each	45600-00
COD Reactor, 230 VAC, 50 Hz, European plug		each	45600-02
Pipet, TenSette, 0.1 to 1.0 mL		each	
Pipet, volumetric, Class A, 2-mL		each	14515-36
Pipet Filler, safety bulb		each	
Test Tube Rack	1 to 2 racks	each	

OPTIONAL REAGENTS AND STANDARDS

COD Digestion Reagent Vials, 0 to 40 mg/L COD	150/pkg	
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 Digestion Reagent Vials, 0 to 15,000 mg/L COD	150/pkg	24159-15
COD Standard Solution, 300-mg/L		
COD Standard Solution, 1000-mg/L		
Mercuric Sulfate, ACS		
Potassium Acid Phthalate, ACS	500 g	
Potassium Persulfate Powder Pillows	100/pkg	
Sulfuric Acid, ACS, concentrated		979-49

OPTIONAL EQUIPMENT AND SUPPLIES

Beaker, 250-mL	each	500-46
Cylinder, graduated, 5-mL	each	508-37
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	
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	
Spoon, measuring, 0.5-g	each	907-00

^{*} Contact Hach for larger sizes.



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 FAX: (970) 669-2932