



Designation: D 1796 – 04

An American National Standard



Designation: Manual of Petroleum Measurement Standards (MPMS), Chapter 10.6

Standard Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)¹

This standard is issued under the fixed designation D 1796; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method describes the laboratory determination of water and sediment in fuel oils in the range from 0 to 30 % volume by means of the centrifuge procedure.

NOTE 1—With some types of fuel oils such as residual fuel oils or distillate fuel oils containing residual components, it is difficult to obtain water or sediment contents with this test method. When this situation is encountered, Test Method D 95 (API *MPMS* Chapter 10.5) or Test Method D 473 (API *MPMS* Chapter 10.1) may be used.

NOTE 2—API *MPMS* Chapter 10.6 (Test Method D 1796) along with API *MPMS* Chapter 10.3 (Test Method D 4007) formerly superseded API Standard 2548.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 6.1.

2. Referenced Documents

2.1 *ASTM Standards*:²

D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (API *MPMS* Chapter 10.5)

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02/COMQ, the joint ASTM-API committee on Static Petroleum Measurement.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API *MPMS* Chapter 10.1)

D 4007 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (API *MPMS* Chapter 10.3)

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.1)

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.2)

D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API *MPMS* Chapter 8.3)

E 542 Practice for Calibration of Laboratory Volumetric Apparatus

2.2 *API Standards*:³

MPMS Chapter 8.1 Practice for Manual Sampling of Petroleum and Petroleum Products (ASTM Practice **D 4057**)

MPMS Chapter 8.2 Practice for Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice **D 4177**)

MPMS Chapter 8.3 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice **D 5854**)

MPMS Chapter 10.1 Test Method for Sediment in Crude Oils by the Extraction Method (ASTM Test Method **D 473**)

MPMS Chapter 10.3 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method **D 4007**)

MPMS Chapter 10.5 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (ASTM Test Method **D 95**)

³ Published as Manual of Petroleum Measurement Standards. Available from the American Petroleum Institute, 1220 L St., N.W., Washington, DC 20005.

*A Summary of Changes section appears at the end of this standard.

2.3 IP Standard:⁴

Methods Book, Appendix B Specification for Methylbenzenes (Toluenes)

2.4 ISO Standard:⁵

ISO 5272:1979 Toluene for Industrial Use—Specifications

3. Summary of Test Method

3.1 Equal volumes of fuel oil and water-saturated toluene are placed in each of two cone-shaped centrifuge tubes. After centrifugation, the volume of the higher density water and sediment layer at the bottom of the tube is read.

4. Significance and Use

4.1 The water and sediment content of fuel oil is significant because it can cause corrosion of equipment and problems in processing. A determination of water and sediment content is required to measure accurately net volumes of actual fuel oil in sales, taxation, exchanges, and custody transfers.

5. Apparatus

5.1 Centrifuge:

5.1.1 Use a centrifuge capable of spinning two or more filled cone-shaped 203-mm (8-in.) centrifuge tubes at a speed that can be controlled to give a relative centrifugal force (rcf) of between 500 and 800 at the tip of the tubes (see 5.1.6).

5.1.2 The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs.

5.1.3 The centrifuge shall be heated and controlled thermostatically to avoid unsafe conditions. It shall be capable of maintaining the sample temperature during the entire process at 60 ± 1°C (140 ± 1.8°F). The thermostatic control shall be capable of maintaining the temperature within these limits and operate safely if there is a flammable atmosphere.

5.1.4 Electric powered and heated centrifuges must meet all safety requirements for use in hazardous areas.

NOTE 3—Some heated centrifuges maintain the bowl at a pressure slightly below atmospheric pressure and reduce the hazards associated with vapors and gasses, produced by samples and solvents used in the tests, by discharging any vapors to a non-hazardous area.

5.1.5 Calculate the speed of the rotating head in revolutions per minute (r/min) as follows:

$$r/min = 1335 \sqrt{rcf/d} \tag{1}$$

where:

rcf = relative centrifugal force, and

d = diameter of swing measured between tips of opposite tubes when in rotating position, mm,

or

$$r/min = 265 \sqrt{rcf/d} \tag{2}$$

where:

rcf = relative centrifugal force, and

d = diameter of swing measured between tips of opposite tubes when in rotating position, in.

5.1.6 Calculate the relative centrifugal force from a measured speed (r/min) as follows:

$$rcf = d \left(\frac{r/min}{1335} \right)^2 \tag{3}$$

where:

d = diameter of swing measured between tips of opposite tubes when in rotating position, mm, or

$$rcf = d \left(\frac{r/min}{265} \right)^2 \tag{4}$$

where:

d = diameter of swing measured between tips of opposite tubes when in rotating position, in.

5.2 Centrifuge Tubes:

5.2.1 Each centrifuge tube shall be a cone-shaped tube, conforming to the dimensions given in Fig. 1, and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct, and the mouth shall be constricted in shape for closure with a cork or solvent-resistant rubber stopper. Scale error tolerances and the smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20°C (68°F), when reading the bottom of the shaded meniscus.

5.2.2 Volumetrically verify or gravimetrically certify the accuracy of the graduation marks, in accordance with Practice E 542 using equipment traceable through the National Institute

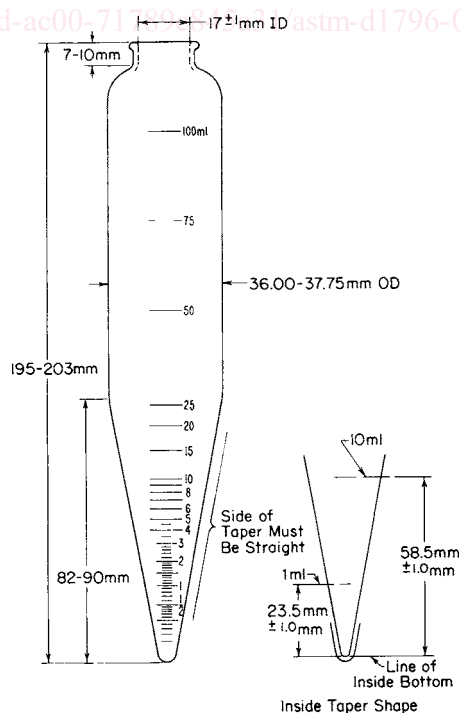


FIG. 1 Eight-Inch (203-mm) Centrifuge Tube

⁴ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

⁵ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

TABLE 1 Centrifuge Tube Calibration Tolerances for 203-mm (8-in.) Tube

Range, mL	Subdivision, mL	Volume Tolerance, mL
0 to 0.1	0.05	±0.02
Above 0.1 to 0.3	0.05	±0.03
Above 0.3 to 0.5	0.05	±0.05
Above 0.5 to 1.0	0.10	±0.05
Above 1.0 to 2.0	0.10	±0.10
Above 2.0 to 3.0	0.20	±0.10
Above 3.0 to 5.0	0.50	±0.20
Above 5.0 to 10	1.00	±0.50
Above 10 to 25	5.00	±1.00
Above 25 to 100	25.00	±1.00

for Standards and Technology (NIST)⁶ or other national standards. Include the verification or certification for each mark through the 0.5-mL mark; of the 1, 1.5 and 2-mL marks; and of the 50 and 100-mL marks. Do not use the tube if the scale error exceeds the applicable tolerance in **Table 1**.

5.3 Bath—The bath shall be either a solid metal block bath or a liquid bath of sufficient depth for immersing the centrifuge tube in the vertical position to the 100-mL mark. Provide the means for maintaining the temperature at $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$). See **Note 4**.

NOTE 4—It has been observed for some fuel oils that temperatures higher than 60°C (140°F) may be required to obtain correct sediment and water content. If temperatures higher than 60°C are necessary, they may be used only with the consent of the parties involved. Water saturation of toluene may also be carried out at this higher testing temperature. (See **Annex A1**.)

6. Reagents

6.1 Toluene, conforming to the IP Specification for Methylbenzenes (Toluenes) or to **ISO 5272**. (**Warning**—Flammable. Keep away from heat, sparks, and open flame. Vapor harmful. Toluene is toxic. Particular care must be taken to avoid breathing the vapor and to protect the eyes. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with the skin.)

6.1.1 Typical characteristics for this reagent are:

Molecular weight $\text{C}_6\text{H}_5\text{CH}_3$	92.14
Color (APHA)	10
Boiling range (initial to dry point) ^A	2.0°C (3.6°F)
Residue after evaporation	0.001 %
Substances darkened by H_2SO_4	passes ACS test
Sulfur compounds (as S)	0.003 %

^A Recorded boiling point 110.6°C .

NOTE 5—Some oils may require other solvents or solvent-demulsifier combinations. Those agreed upon between the purchaser and the seller may be used.

6.1.2 The solvent shall be water-saturated at $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$) but shall be free of suspended water. See **Annex A1** for the solvent-water saturation procedure.

6.2 Demulsifiers:

6.2.1 Where necessary, use a demulsifier to promote the separation of water from the sample, to prevent water from

clinging to the walls of the centrifuge tube, and to enhance the distinctness of the water-oil interface.

6.2.2 When using a demulsifier, it should be mixed according to the manufacturer's recommendations and should never be added to the volume of sediment and water determined. Always use the demulsifier in the form of a demulsifier-solvent stock solution or be premixed with the solvent to be used in the test.

7. Sampling

7.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place them into the laboratory test container.

7.2 Only representative samples obtained as specified in Practice **D 4057** (API **MPMS Chapter 8.1**) and Practice **D 4177** (API **MPMS Chapter 8.2**) shall be used for this test method.

7.3 Practice **D 5854** (API **MPMS Chapter 8.3**) contains additional information on sampling and homogenization efficiency of an untested mixer. Do not use this test method without strict adherence to Practice **D 5854** (API **MPMS Chapter 8.3**).

8. Procedure

8.1 Fill each of two centrifuge tubes (**5.2**) to the 50-mL mark with the well-mixed sample directly from the sample container. Using a pipette, add 50 mL of the water-saturated solvent (**6.1**). Read the top of the meniscus at both the 50 and 100 mL marks. Stopper the tubes tightly and shake vigorously until the contents are thoroughly mixed. Loosen the stoppers on the tubes and immerse the tubes to the 100-mL mark for 10 min in the bath maintained at $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$).

8.2 Tighten the stoppers and again invert the tubes to ensure that the oil and solvent are uniformly mixed and shake cautiously. (**Warning**—In general, the vapor pressures of hydrocarbons at 60°C (140°F) are approximately double those at 40°C (104°F). Consequently, invert the tubes at a position below eye level so that contact will be avoided if the stopper is blown out.)

8.3 Place the tubes in the trunnion cups on opposite sides of the centrifuge to establish a balanced condition and ensure that the tubes and stoppers do not touch adjacent or opposite tubes when in the extended position. Spin for 10 min at a rate, calculated from the equation given in **5.1.6**, sufficient to produce a relative centrifugal force (rcf) of between 500 and 800 at the tip of the whirling tubes (see **Table 2** for the relationship between diameter of swing, relative centrifugal force, and revolutions per minute). Maintain the temperature of the sample during the entire centrifuging procedure at $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$). (See **Note 4**.)

8.4 Immediately after the centrifuge comes to rest following the spin (ensure the tubes are immediately brought to a vertical position after the centrifuge stops because the results may be affected if the tubes come to rest at an angle), read and record the combined volume of water and sediment at the bottom of each tube to the nearest 0.05 mL from 0.1 to 1-mL graduations and to the nearest 0.1 mL above the 1-mL graduations. Below 0.1 mL, estimate to the nearest 0.025 mL (see **Fig. 2**). If less than 0.025 mL of water and sediment is visible and it is not a great enough volume to be considered 0.025 mL, record the

⁶ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.