Designation: D1796 – 22

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 D1796; 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 U.S. 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 D95 (API *MPMS* Chapter 10.5) or Test Method D473 (API *MPMS* Chapter 10.1) may be used.

NOTE 2—API *MPMS* Chapter 10.6 (Test Method D1796) along with API *MPMS* Chapter 10.3 (Test Method D4007) formerly superseded API Standard 2548.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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 7.1.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (API *MPMS* Chapter 10.5)

D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API *MPMS* Chapter 10.1)

D4007 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (API *MPMS* Chapter 10.3)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.1)

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.2)

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API *MPMS* Chapter 8.3)

D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

E542 Practice for Gravimetric Calibration of Laboratory Volumetric Instruments

2.2 *API Standards:*[3]

MPMS Chapter 1 Terms and Definitions Database

MPMS Chapter 8.1 Practice for Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D4057)

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, 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 Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API). This test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

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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.

[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**

MPMS Chapter 8.2 Practice for Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)

MPMS Chapter 8.3 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D5854)

MPMS Chapter 10.1 Test Method for Sediment in Crude Oils by the Extraction Method (ASTM Test Method D473)

MPMS Chapter 10.3 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D4007)

MPMS Chapter 10.5 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (ASTM Test Method D95)

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. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175 and the API *MPMS* Chapter 1 Terms and Definitions Database.

4. Summary of Test Method

4.1 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.

5. Significance and Use

5.1 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.

5.2 This test method may not be suitable for products that contain alcohols that are soluble in water. In cases where the impact on the results may be significant, the user is advised to consider using another test method, such as Test Method D6304.

6. Apparatus

6.1 *Centrifuge:*

6.1.1 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 6.1.6).

6.1.2 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.

6.1.3 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 °C ± 1 °C (140 °F ± 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.

6.1.4 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.

6.1.5 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.

6.1.6 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.

6.2 *Centrifuge Tubes:*

6.2.1 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

[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, http://www.ansi.org.

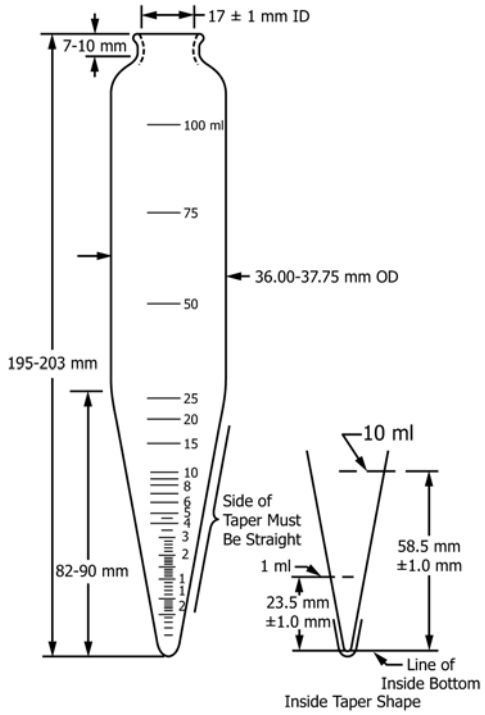

FIG. 1 Eight-Inch (203 mm) Centrifuge Tube

Table 1 and apply to calibrations made with air-free water at 20 °C (68 °F), when reading the bottom of the shaded meniscus.

6.2.2 Volumetrically verify or gravimetrically certify the accuracy of the graduation marks, in accordance with Practice E542 using equipment traceable through the National Institute 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 mL, 1.5 mL, and 2 mL marks; and of the 50 mL and 100 mL marks. Do not use the tube if the scale error exceeds the applicable tolerance in Table 1.

6.3 *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 °C ± 1 °C (140 °F ± 1.8 °F). See Note 4.

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

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

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.)

7. Reagents

7.1 *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.)

7.1.1 Typical characteristics for this reagent are:

Molecular weight $C_6H_5CH_3$	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.

7.1.2 The solvent shall be water-saturated at 60 °C ± 1 °C (140 °F ± 1.8 °F) but shall be free of suspended water. See Annex A1 for the solvent-water saturation procedure.

7.2 *Demulsifiers:*

7.2.1 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.

7.2.2 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.

8. Sampling

8.1 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.

8.2 Only representative samples obtained as specified in Practice D4057 (API *MPMS* Chapter 8.1) and Practice D4177 (API *MPMS* Chapter 8.2) shall be used for this test method.

8.3 Practice D5854 (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 D5854 (API *MPMS* Chapter 8.3).

9. Procedure

9.1 Fill each of two centrifuge tubes (6.2) 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 (7.1).