

Manual of Petroleum Measurement Standards Chapter 10.6

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

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Designation: D1796 – 11^{ε1}



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 Department of Defense.

^{ε1} NOTE—Referenced Documents and API information was editorially corrected in June 2013.

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NOTE 2—API MPMS Chapter 10.6 (Test Method D1796) along with API MPMS Chapter 10.3 (Test Method D4007) formerly superseded API Standard 2548.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 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

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)

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 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 D4057)

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

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 on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API).

Current edition approved June 1, 2011. Published August 2011. Originally approved in 1960. Last previous edition approved in 2009 as D1796-04(2009). DOI: 10.1520/D1796-11.

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

(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. 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.

4.2 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.

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 $60 \pm 1^\circ\text{C}$ ($140 \pm 1.8^\circ\text{F}$). 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:

$$r/min = 1335 \sqrt{rcf/d} \quad (1)$$

where:

rcf = relative centrifugal force, and

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

or

$$r/min = 265 \sqrt{rcf/d} \quad (2)$$

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:

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

where:

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

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

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

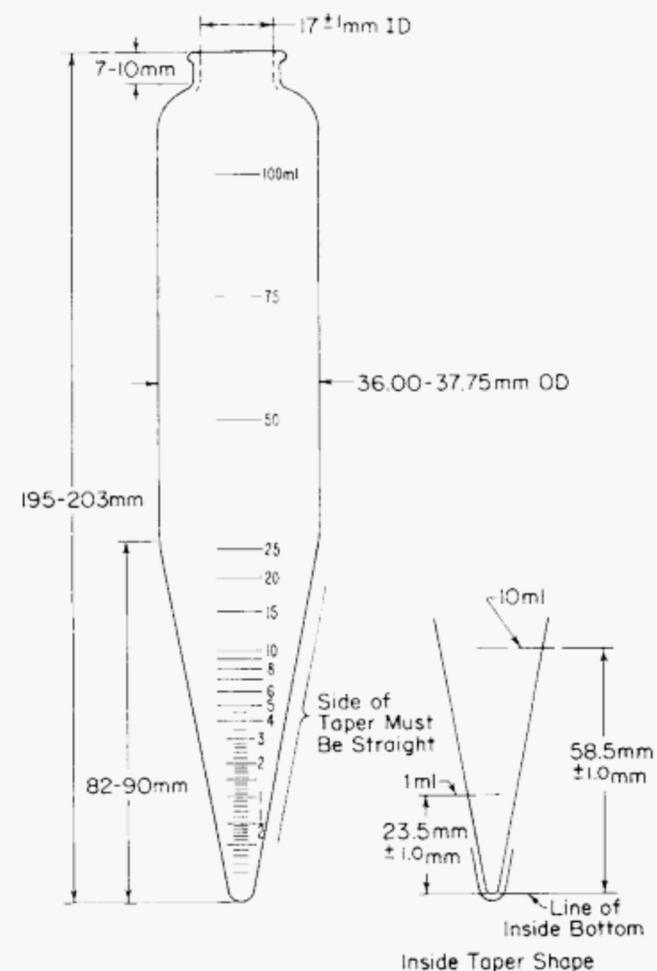


FIG. 1 Eight-Inch (203-mm) Centrifuge Tube

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, <http://www.ansi.org>.

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 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

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 **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, 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 ± 1°C (140 ± 1.8°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 C ₆ H ₅ 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 ₂ SO 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 Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

6.1.2 The solvent shall be water-saturated at 60 ± 1°C (140 ± 1.8°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 **D4057** (API MPMS Chapter 8.1) and Practice **D4177** (API MPMS Chapter 8.2) shall be used for this test method.

7.3 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).

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 ± 1°C (140 ± 1.8°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 ± 1°C (140 ± 1.8°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

TABLE 2 Rotation Speeds Applicable for Centrifuges of Various Diameters of Swing

NOTE 1—rcf = relative centrifugal force.

Diameter of Swing		Revolutions per Minute	
Millimetres	Inches ^A	At 500 rcf	At 800 rcf
305	12	1710	2170
330	13	1650	2010
356	14	1590	2000
381	15	1530	1930
406	16	1490	1870
432	17	1440	1820
457	18	1400	1770
483	19	1360	1720
508	20	1330	1680
533	21	1300	1640
559	22	1270	1600
584	23	1240	1560
610	24	1210	1530

^A For this column, the diameter of swing is measured in inches between tips of opposite tubes when in rotating position.

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 volume as less than 0.025 mL. If no water or sediment is visible, record the volume as 0.000 mL. Return the tubes without agitation to the centrifuge and spin for another 10 min at the same rate.

8.5 Repeat this operation until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two spinings are required.

8.6 If the difference between the final volumes is not greater than one subdivision on the centrifuge tube (see Table 1), or, not greater than 0.0025 mL for estimated volumes of 0.10 mL or lower, proceed with the calculation of water and sediment content using Eq 5 as described in Section 9. If the difference is greater than one subdivision or, for readings of 0.10 or below, greater than 0.025 mL, the readings are inadmissible. If this is the case, repeat the determination starting at 8.1.

9. Calculation

9.1 Record the final volume of water and sediment in each tube. If the difference between the two readings is greater than one subdivision on the centrifuge tube (see Table 1) or 0.025 mL for readings of 0.10 mL and below, the readings are inadmissible and the determination shall be repeated.

9.2 Calculate the water and sediment of the sample as follows:

$$V = V_1 + V_2 \quad (5)$$

where:

V = water and sediment of the sample (the test result), % V/V,

V_1 = final volume of water and sediment per 50 mL of sample in the first tube, mL, and

V_2 = final volume of water and sediment per 50 mL of sample in the second tube, mL.

9.3 Express the sum of the two admissible readings as the percentage by volume of water and sediment.

10. Report

10.1 Report the result V as the water and sediment, % V/V, as shown in Table 3.

TABLE 3 Expression of Results, mL

Tube 1—Volume of water and sediment, mL	Tube 2—Volume of water and sediment, mL	Total Percent Water and Sediment, % (V/V)
No visible water and sediment	No visible water and sediment	0.00
No visible water and sediment	0.025	0.025
0.025	0.025	0.05
0.025	0.05	0.075
0.05	0.05	0.10
0.05	0.075	0.125
0.075	0.075	0.15
0.075	0.10	0.175
0.10	0.10	0.20
0.10	0.15	0.25

10.2 Round test results as follows:

10.2.1 Above 2.00 V/V, round to the nearest 0.1 % V/V.

10.2.2 In the range from 0.20 to 2.00 % V/V, round to the nearest 0.05 % V/V.

10.2.3 Below 0.20 % V/V, round as shown in Table 3.

10.3 Report the solvent used, if other than toluene (see Note 5). Report the name and amount of demulsifier, if used (see 6.2). Report the test temperature if it is not 60°C (140°F) (see Note 4).

11. Precision and Bias

11.1 *Precision*—The criteria described in 11.1.1 and 11.1.2 should be used for judging the acceptability of results (95 % probability).

11.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values in Fig. 3 in only one case in twenty.

11.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material,

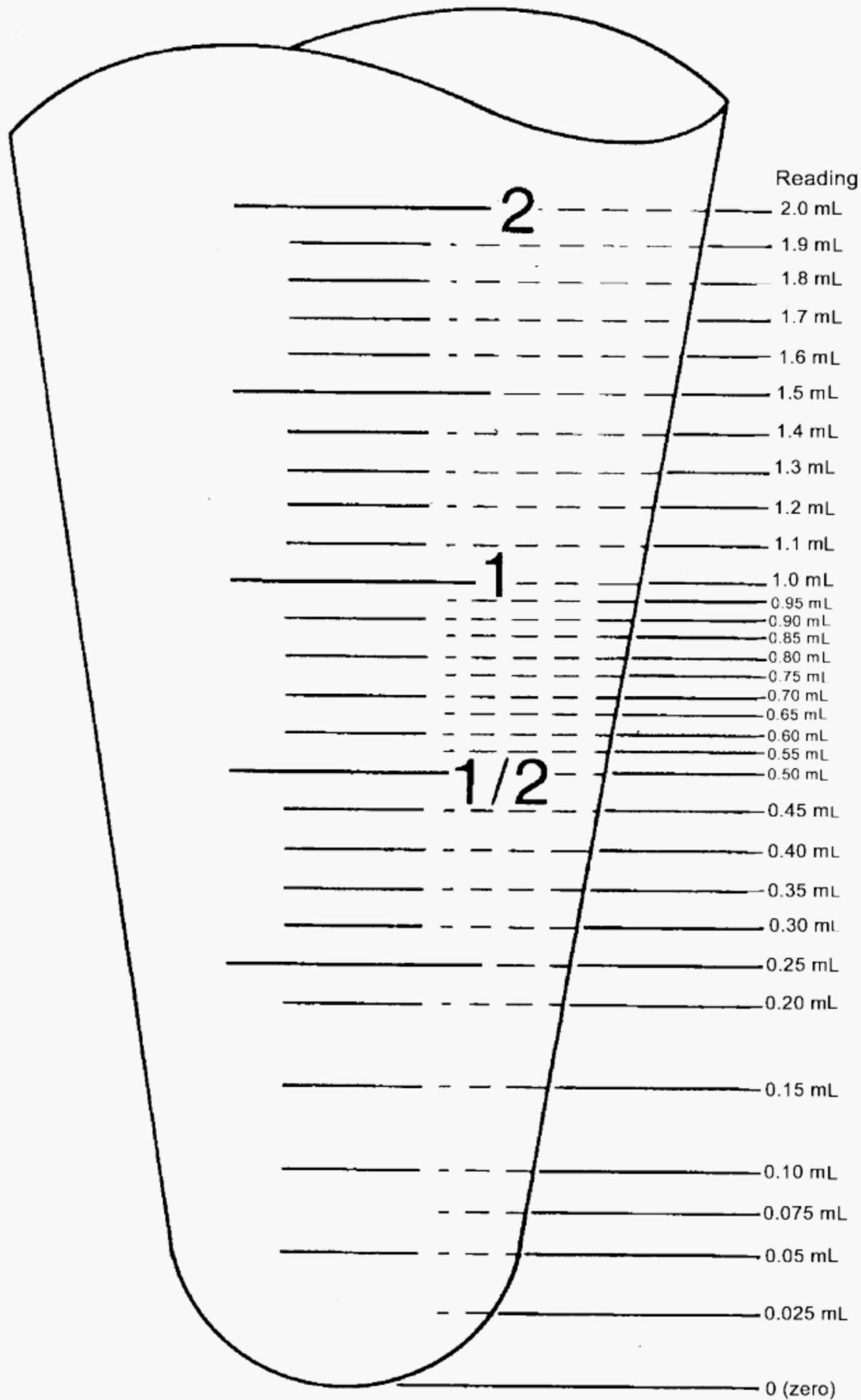


FIG. 2 Procedure for Reading Water and Sediment When Using an ASTM 100-mL Cone-Shaped Centrifuge Tube

would, in the long run, in the normal and correct operation of the test method, exceed the values in Fig. 3 in only one case in twenty.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure described in Test Method D1796 (API MPMS Chapter 10.6) for determin-

ing water and sediment in fuel oils by the centrifuge method, no statement about bias is made.

11.3 *Comparison of Precision of Distillation and Centrifuge Methods for Crude Oils:*

11.3.1 A round-robin testing program carried out on crude oils (see Appendix X1 of Test Method D4007/API MPMS

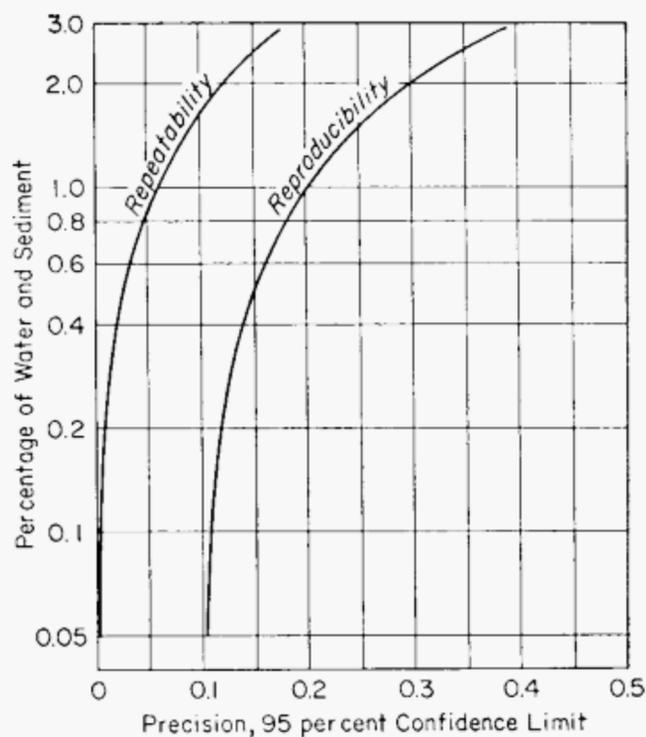


FIG. 3 Precision Curves for Centrifuge Tube Methods

Chapter 10.3) has shown that the distillation method as practiced is somewhat more accurate than the centrifuge method. The average correction for the distillation method is about 0.06, whereas the centrifuge correction is about 0.10. However, this correction is not constant nor does it correlate well with the measured concentration.

11.3.2 There is a slight improvement in the precision of the distillation method over the present Test Method D95 (API MPMS Chapter 10.5): 0.08 repeatability versus 0.1, and 0.11 versus 0.2 for reproducibility. These figures are applicable from 0.1 to 1 % water content, the maximum level studied in this program.

11.3.3 The precision of the centrifuge method is worse than the distillation method: repeatability is 0.12 and the reproducibility is 0.28.

12. Keywords

12.1 centrifuge; fuel oils; toluene saturation; water and sediment

ANNEX

(Mandatory Information)

A1. PROCEDURE TO WATER-SATURATE TOLUENE

A1.1 Scope

A1.1.1 The procedure described in this annex is satisfactory for the water saturation of toluene to be used for the determination of water and sediment in fuel oils by the centrifuge method.

A1.2 Significance and Use

A1.2.1 Fig. A1.1 shows that water is soluble in toluene to a significant extent. The percentage of water that will dissolve increases as the temperature is increased from about 0.03 % at 21°C (70°F) to about 0.17 % at 70°C (158°F). Toluene, as normally supplied, is relatively dry and, if used in the as-

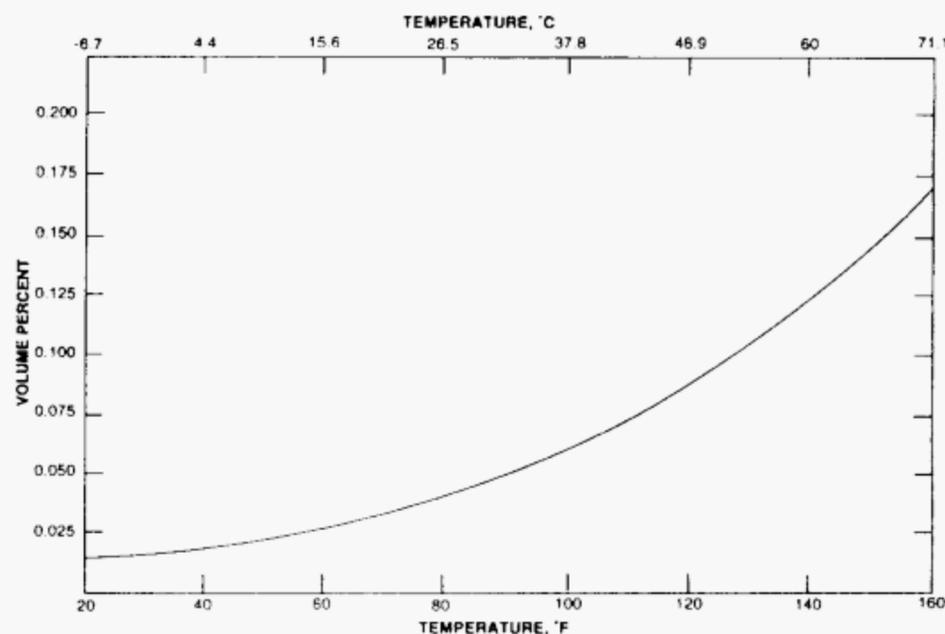


FIG. A1.1 Solubility of Water in Toluene

received condition, will dissolve a portion or even all the water present in a fuel oil sample. This dissolution would reduce the apparent sediment and water level in the fuel oil sample. To determine water and sediment accurately by centrifuge on a fuel oil sample, the toluene must first be saturated at the centrifuge test temperature.

A1.3 Reagents

A1.3.1 Toluene, conforming to the IP Specification for Methylbenzenes (Toluenes) or ISO 5272.

A1.3.2 Water, either distilled or tap water.

A1.4 Apparatus

A1.4.1 Liquid-Heating Bath, of sufficient depth for immersing a 1-qt or 1-L bottle to its shoulder. Provide a means for maintaining the temperature at 60 ± 1°C (140 ± 1.8°F).

A1.4.2 Bottle, 1-qt or 1-L, with screw top.

A1.5 Procedure

A1.5.1 Adjust the heating bath to the temperature at which the centrifuge test is to be run. Maintain the temperature to an accuracy of ±1°C.

A1.5.2 Fill the glass bottle with 700 to 800 mL of toluene. Add sufficient water (at least 2 mL but not more than 25 mL) to maintain a visual indication of excess water. Screw the cap on the bottle and shake vigorously for 30 s.

A1.5.3 Loosen the cap and place the bottle into the bath for 30 min. Remove the bottle, tighten the cap, and shake

cautiously for 30 s. (**Warning**—In general, the vapor pressures of hydrocarbons at 60°C (140°F) are approximately double those at 40°C (104°F). Consequently bottles should always be inverted at a position below eye level so that contact will be avoided if the cap is blown off.)

A1.5.4 Repeat the procedure in A1.5.3 three times.

A1.5.5 Allow the bottle with the water-toluene mixture to sit in the bath for 48 h before using it. This will ensure complete equilibrium between the toluene and the free water as well as complete saturation at the desired temperature. If it is necessary to use the water-saturated toluene before the 48-h

equilibrium time has been completed, pour the solvent into centrifuge tubes and centrifuge in the same equipment at the same relative centrifugal force and temperature that is used for the centrifuge test. The toluene must be carefully pipetted from the centrifuge tube so that any free water that may be at the bottom of the tube is not disturbed.

A1.5.6 Saturation is time- and temperature-dependent. It is recommended that bottles of the toluene-water mixture be kept at test temperature in the bath at all times so that saturated solvent will be available whenever tests are to be run.

SUMMARY OF CHANGES

Subcommittee D02.02.08 has identified the location of selected changes to this standard since the last issue (D1796–04(2009)) that may impact the use of this standard.

(1) Added Test Method D6304 to the Referenced Documents and standard text.

(2) Added Subsection 4.2.

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