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Standard Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)[1]

This standard is issued under the fixed designation D 96; 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 method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

This test method has been adopted for use by government agencies to replace Method 3003 of Federal Test Method Standard No. 791b.

Annex A1 is under revision and will be included in subsequent revisions to this standard.

1. Scope

1.1 This test method covers the centrifuge method for determining sediment and water in crude oil during field custody transfers. This test method may not always provide the most accurate results, but it is considered the most practical method for field determination of sediment and water. When a higher degree of accuracy is required, the laboratory procedure described in Test Methods D 4006D 4006, D 4377D 4377 or D 473D 473 should be used.

NOTE 1—Water by distillation and sediment by extraction are considered the most accurate methods of determining sediment and water in crude oils. As such, these methods should be employed to resolve differences in results from variations of this procedure or between this procedure and other methods, or in the case of a dispute between parties.

1.2 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Drycleaning Spirits)[2]
D 362 Specification for Industrial Grade Toluene[2]
D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method[3]
D 846 Specification for Ten-Degree Xylene[2]
D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[2]
D 3699 Specification for Kerosine[4]
D 4006 Test Method for Water in Crude Oil by Distillation[4]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[4]
D 4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration[4]
E 1 Specification for ASTM Thermometers[5]
E 542 Practice for Calibration of Volumetric Ware[6]

2.2 *API Standards:*[7]

Manual of Petroleum Measurement Standards
Chapter 8, Sampling Petroleum and Petroleum Products
Chapter 10, Sediment and Water

3. Summary of Test Method

3.1 Known volumes of crude oil and solvent (water saturated if required) are placed in a centrifuge tube and heated to 60°C ± 3°C (140°F ± 5°F). After centrifugation, the volume of the sediment-and-water layer at the bottom of the tube is read.

NOTE 2—It has been observed that for some waxy crude oils, temperatures of 71°C (160°F) or higher may be required to melt the wax crystals completely so that they are not measured as sediment. If temperatures higher than 60°C (140°F) are necessary to eliminate this problem, they may be used with the consent of the parties involved. If water saturation

[1] This test method is under the jurisdiction of Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.02.OB on Sediment and Water (Joint ASTM-JP).

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from American Petroleum Institute, 1220 L St., Northwest, Washington, DC 20005.

of the solvent is required, it must be done at the same temperature.

4. Significance and Use

4.1 A determination of sediment and water content is required to determine accurately the net volumes of crude oil involved in sales, taxation, exchanges, inventories, and custody transfers. An excessive amount of sediment and water in crude oil is significant because it can cause corrosion of equipment and problems in processing and transporting and may violate federal, state, or municipal regulations.

5. Apparatus

5.1 *Centrifuge*—A centrifuge shall be capable of spinning two or more centrifuge tubes at a speed that can be controlled to give a minimum relative centrifugal force of 500 at the tip of the tubes. The rotation speed necessary to produce a relative centrifugal force of 500 for various diameters of swing can be determined from Table 1 or from one of the following equations:

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

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

where:

rpm = rotation speed, in revolutions per minute.
rcf = relative centrifugal force,
d = diameter of swing, in mm (Eq 1) or in. (Eq 2), measured between the tips of opposite tubes when the tubes are in their rotating position.

The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be 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 contain flying debris in the event a tube breaks or the centrifuge malfunctions.

5.1.1 The centrifuge shall be heated and shall be capable of maintaining the sample at a temperature of 60°C ± 3°C (140°F± 5°F). The minimum allowable temperature in the field shall be 52°C (125°F).

TABLE 1 Rotation Speeds Necessary to Produce a Relative Centrifugal Force of 500 for Centrifuges of Various Diameters of Swing

Diameter of Swing[A]		Rotation Speed (r/min)
Millimeters	Inches	
305	12	1710
330	13	1640
356	14	1580
381	15	1530
406	16	1480
432	17	1440
457	18	1400
483	19	1360
508	20	1325
533	21	1290
559	22	1260
584	23	1240
610	24	1210

[A]Measured between the tips of the opposite tubes when the tubes are in rotating position.

5.2 *Centrifuge Tubes*:

5.2.1 Centrifuge tubes shall be cone shaped and 203 mm (8 in.) or 167 mm (6 in.) in length. Tubes shall conform to the dimensions given in Fig. 1 (203 mm) or Fig. 2 (167 mm) and shall be made of thoroughly annealed glass. A200-part tube shall conform to the dimensions shown in Fig. 2, with the marking for each division multiplied by 2 (for example, 25 mL = 50 parts). The mouth of each tube shall be constricted for closure with a stopper. Graduations for the 203-mm (8-in.) and 167-mm (6-in.) tubes shall be in accordance with the requirements of Table 2 and Table 3, respectively. The scale errors for a centrifuge tube shall not exceed the tolerances specified in Table 2 and Table 3. The graduation requirements and scale-error tolerances shown in Table 2 and Table 3 apply to calibrations made by reading the bottom of the shaded meniscus of air-free water at a temperature of 20°C (68°F). The graduations on each tube shall be clearly numbered as shown in Fig. 1 and Fig. 2.

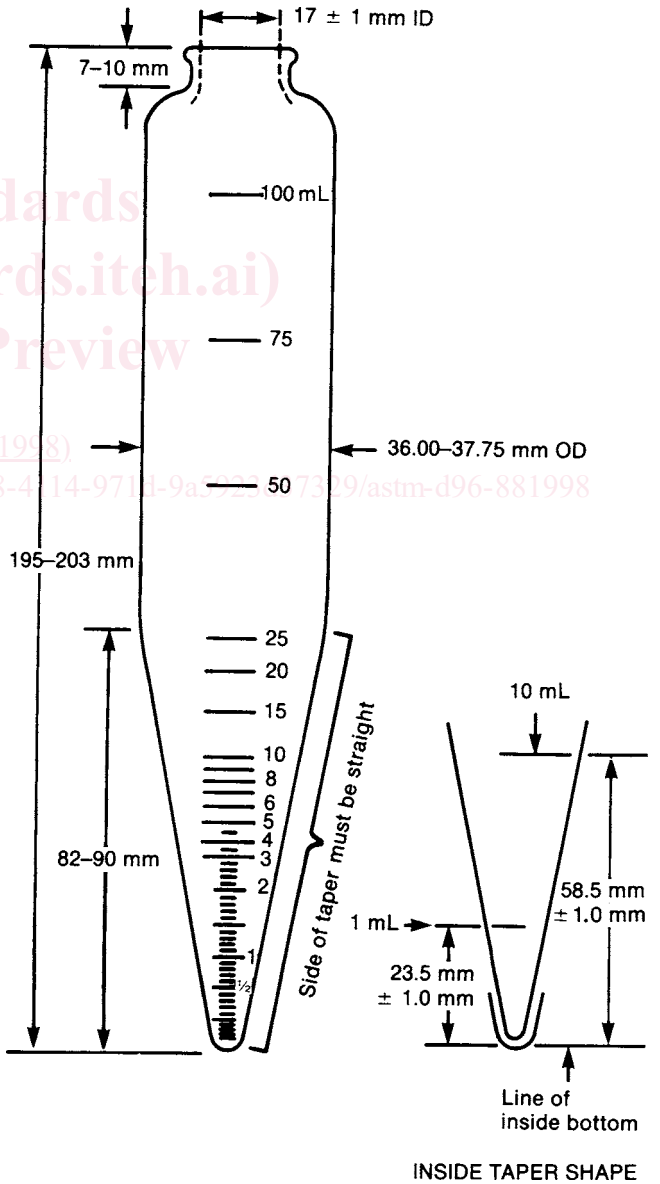

FIG. 1 Cone-Shaped Centrifuge Tube, 203 mm (8 in.)

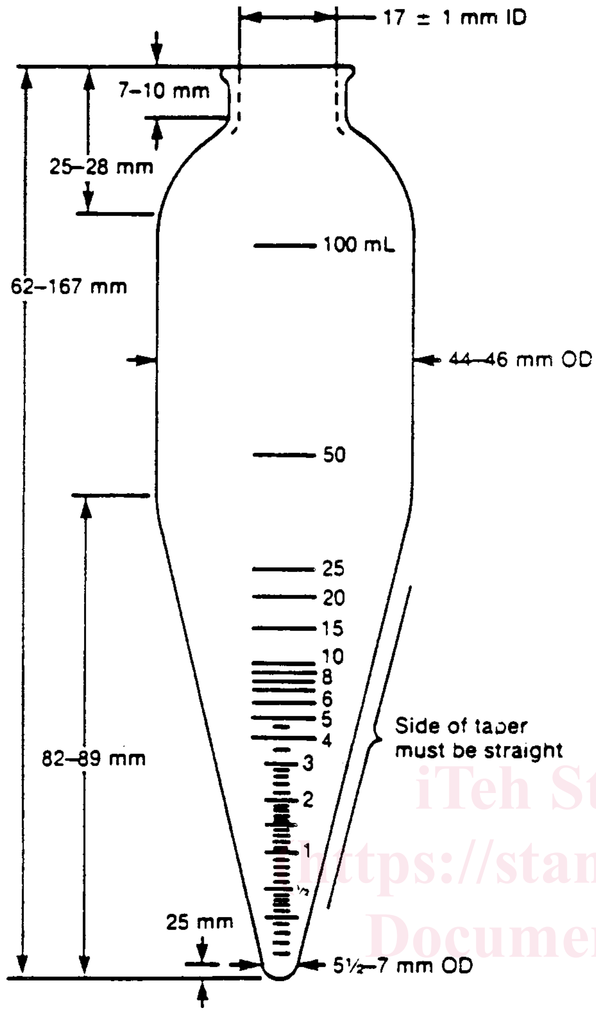

FIG. 2 Cone-Shaped Centrifuge Tube, 167 mm (6 in.)

TABLE 2 Minimum Graduation Requirements and Maximum Calibration Tolerances for 203-mm (8-in.) Cone-Shaped Tubes

Range, mL	Subdivision, mL	Volume Tolerance, mL
0–0.1	0.05	±0.02
>0.1–0.3	0.05	±0.03
>0.3–0.5	0.05	±0.05
>0.5–1.0	0.10	±0.05
>1.0–2.0	0.10	±0.10
>2.0–3.0	0.20	±0.10
>3.0–5.0	0.5	±0.20
>5.0–10	1	±0.50
>10–25	5	±1.00
>25–100	25	±1.00

5.2.2 The tube graduation marks' accuracy shall be volumetrically verified or gravimetrically certified before field use of the tube, in accordance with Practice E 542E 542 using National Institute of Standards and Technology-traceable equipment. The verification or certification shall include a calibration check at each mark up through the 0.5-mL (1-part) mark; at the 1-, 1.5-, and 2-mL (2-, 3-, and 4-part) marks: and at the 50- and 100-mL (100- and 200-part) marks. The tube shall not be used if the scale error at any mark exceeds the applicable tolerance from Table 2 or 3.

TABLE 3 Minimum Graduation Requirements and Maximum Calibration Tolerances for 167-mm (6-in.) Cone-Shaped Tubes

Range, mL	Subdivision, mL	Volume Tolerance, mL
>0–0.1	0.05	±0.02
>0.1–0.3	0.05	±0.03
>0.3–0.5	0.05	±0.05
>0.5–1.0	0.10	±0.07
>1.0–1.5	0.10	±0.10
>1.5–2.0	0.10	±0.20
>2.0–3.0	0.20	±0.30
>3.0–5.0	0.50	±0.50
>5.0–10	1	±0.75
>10–25	5	±1.0
>25–100	A	±1.5

A Graduations at 50 and 100.

5.3 *Preheater*—The preheater shall be either a metal block or a liquid bath of sufficient depth to permit immersion of the centrifuge tube in the vertical position to the 100-mL litre (200-part) mark and capable of heating the sample to 60°C ± 3°C (140°F± 5°F).

5.4 Thermometer shall have graduations at intervals of 1°C (2°F) or less and shall be accurate to ±1°C (±2°F). A thermometer such as ASTM 1C or 1F is suitable as shown in Specification E 1E 1.

6. Reagents

6.1 The reagents listed in this section are satisfactory for use in field testing.

6.2 *Demulsifier*—When necessary, a demulsifier should be used to promote the separation of water from the sample, to prevent water from the sample, clinging to the walls of the centrifuge tube, and to enhance the distinctiveness of the water-oil interface. In some cases a demulsifier is required to attain agreement with the base method (see Note 1). When a demulsifier is used, it should be mixed according to the manufacturer's recommendations and should never add to the volume of sediment and water determined. The demulsifier should always be used in the form of a demulsifier-solvent stock solution or be premixed with the solvent to be used in the test.

6.3 *Kerosine (Specification D 3699D 3699)*

6.3.1 The typical characteristics of kerosine are a distillation range of 205-300°C (401-572°F), a maximum freezing point of – 30°C (–22°F), and a minimum flash point of 38°C (100°F).

6.3.2 Stoddard solvent and kerosine do not have to be saturated with water, since the solubility of water in these solvents is not significant at 60°C (140°F).

NOTE 3—**Warning:** Kerosine is combustible (See A1.1).

6.4 *Stoddard Solvent (Specification D 235D 235)*:

6.4.1 The typical characteristics of Stoddard solvent are a distillation range of 149–208°C (300–407°F), a minimum flash point of 38°C (100°F), and aromatics plus olefins content of less than 20 % by volume.

NOTE 4—**Warning:** Stoddard solvent is combustible (See A1.2).

6.4.2 See 6.3.2.

6.5 *Toluene (Specification D 362D 362)*:

6.5.1 The typical characteristics of toluene are a molecular weight of 92, an American Public Health Association (APHA)