



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 4006, D 4377 or D 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 Toluene²
- D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method³
- D 846 Specification for Ten-Degree Xylene²
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)²

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

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

D 3699 Specification for Kerosine⁴

D 4006 Test Method for Water in Crude Oil by Distillation⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products⁴

D 4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration⁴

E 1 Specification for ASTM Thermometers⁵

E 542 Practice for Calibration of Volumetric Ware⁶

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 \pm 3°C (140°F \pm 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 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

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.

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:

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

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

where:

rpm = rotation speed, in revolutions per minute.

rcf = relative centrifugal force,

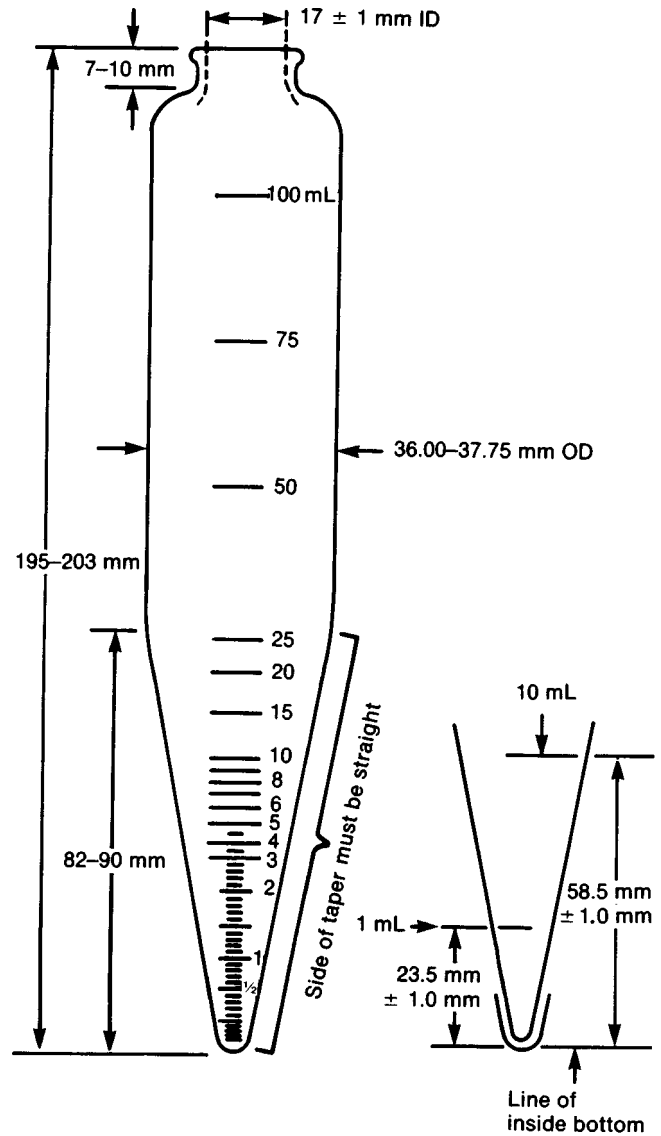
d = diameter of swing, in mm (Eq 1) or in. (Eq 2), measured between the tips of opposite tubes when the tubes are in their rotating position.

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).

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. A 200-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



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

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

^AMeasured between the tips of the opposite tubes when the tubes are in rotating position.

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.

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 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

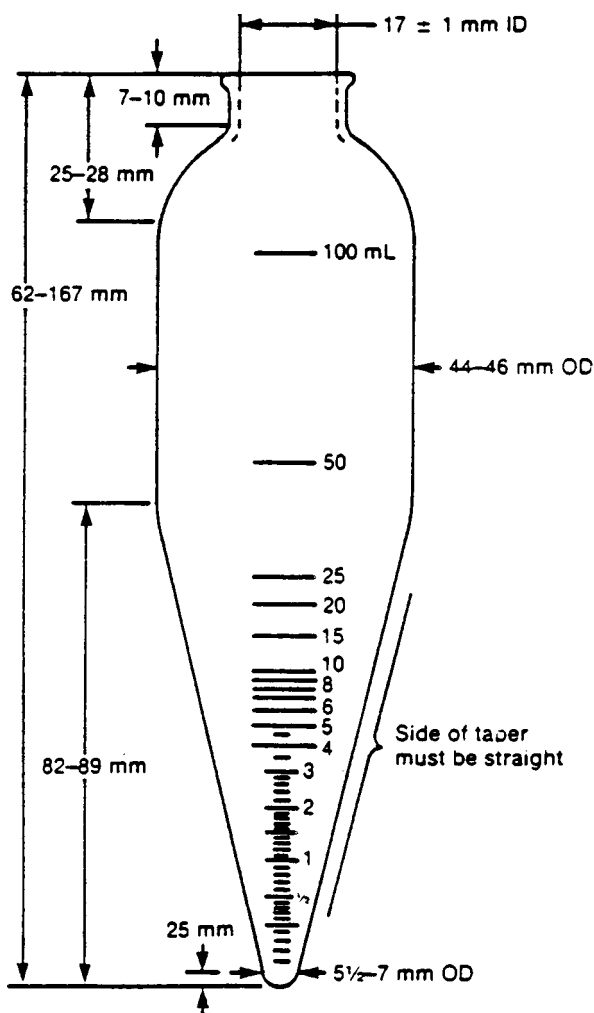


FIG. 2 Cone-Shaped Centrifuge Tube, 167 mm (6 in.)

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

if the scale error at any mark exceeds the applicable tolerance from Table 2 or 3.

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

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

^AGraduations at 50 and 100.

thermometer such as ASTM 1C or 1F is suitable as shown in Specification E 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 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 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 362):*

6.5.1 The typical characteristics of toluene are a molecular weight of 92, an American Public Health Association (APHA) color of 10 (in accordance with Test Method D1209), a boiling range (initial to dry point) of 2.0°C (3.6°F) [recorded boiling point of 110.6°C (231.1°F)], and 0.001 % residue after evaporation. Toluene passes the American Chemical Society (ACS) test for substances darkened by H₂SO₄.

NOTE 5—**Warning:** Toluene is flammable (See A1.3).

6.5.2 Toluene and xylene shall be saturated with water at

60°C ± 3°C (140°F ± 5°F) and maintained at this temperature until used. A procedure for the saturation of solvents is given in the appendix. The water-saturated solvent shall be free from suspended water at the time of use. Toluene and xylene are recommended for sediment-and-water determinations involving asphaltenic crude oils.

6.6 Xylene (Specification D 846):

6.6.1 The typical characteristics of xylene are a molecular weight of 106, an APHA color of not more than 10 (in accordance with Test Method D 1209), a boiling range of 137 to 144°C (279 to 291°F), and 0.002 % residue after evaporation. Xylene passes the ACS test for substances darkened by sulfuric acid.

NOTE 6—**Warning:** Xylene is flammable (See A1.4).

6.6.2 See 6.5.2.

7. Sampling

7.1 Sampling is defined as all steps required to obtain a representative quantity of the contents of any pipeline, tank, or other system and to place it in an appropriate centrifuge tube.

7.2 The sample shall be thoroughly representative of the crude oil in question, and the portion of the sample used for the sediment and water determination shall be thoroughly representative of the sample itself. If an automatic custody transfer (LACT) unit is involved, vigorous agitation of the sample container is required before the sample is transferred to the centrifuge tube or tubes. Only representative samples obtained as specified in Practices D 4057 or D 4177 shall be used for this test method.

8. Procedure

8.1 Fill each of two centrifuge tubes to exactly the 50-mL (100-part) mark with a sample taken directly from the sampling device (for example, a thief bottle, beaker, or LACT sample container) or the container in which the sample was collected. Then fill each tube with solvent to exactly the 100-mL (200-part) mark. Read the top of the meniscus at both the 50- and 100-mL (100- and 200-part) marks. If experience indicates that a demulsifier is required and one has not already been added to the solvent, add to each tube quantity of demulsifier-solvent stock solution that has previously been determined to be satisfactory for the crude oil under test. Stopper each tube tightly and invert the tubes a minimum of 10 times to ensure that the oil and solvent are uniformly mixed.

NOTE 7—**Caution:** In general, the vapor pressures of hydrocarbons at 60°C (140°F) are approximately double those at 40°C (104°F). Consequently, tubes should always be inverted at a position below eye level.

8.1.1 Where the crude oil is very viscous and mixing of the solvent with the oil is difficult, the solvent may be added to the centrifuge tube prior to the oil to facilitate mixing. In this case, extreme care must be taken to fill the centrifuge tube to exactly the 50-mL (100-part) mark with solvent and then to exactly the 100-mL (200-part) mark with the sample.

8.2 Loosen the stoppers to prevent pressure buildup during heating and immerse the tubes to the 100-mL (200-part) mark in a preheater. Heat the contents to 60°C ± 3°C (140°F ± 5°F).

8.3 Secure the stoppers and again invert the tubes 10 times to ensure uniform mixing of the oil and solvent.

8.4 Place the tubes in the trunion cups on opposite sides of the centrifuge to establish a balanced condition. Retighten the stoppers and spin for at least 5 minutes at a minimum relative centrifugal force of 500.

8.5 Immediately after the centrifuge comes to rest, verify the temperature. Do not disturb the oil-water interface with the thermometer. The test is invalid if the final temperature after centrifugation is below 52°C (125°F).

NOTE 8—If the final temperature is found to be below 52°C (125°F), adjust the centrifuge heater to increase the final test temperature and reinitiate the procedure, beginning with 8.2.

8.5.1 Read and record the combined volume of sediment and water at the bottom of each tube as indicated in Table 4 and Fig. 3 (Table 5 and Fig. 4 for 200-part tubes). Reheat both tubes to 60°C ± 3°C (140°F ± 5°F), return the tubes without agitation to the centrifuge, and spin for another 5 min at the same rate. Repeat this operation until two consecutive consistent readings are obtained on each tube.

8.6 For the test to be considered valid, a clear interface must be observed between the oil layer and the separated water. No identifiable layering (that is, an emulsion) should be present immediately above the oil-water interface. In such cases, one or more of the following remedies may be effective:

8.6.1 Shake the mixture between whirlings in the centrifuge just enough to disperse the emulsion.

8.6.2 Use a different or an increased amount of demulsifier. (The demulsifier should not, however, contribute to the volume of sediment and water.)

8.6.3 Use a different or an increased amount of solvent. After a satisfactory procedure for a particular type of oil has been worked out, it will ordinarily be suitable for all samples of the same crude oil.

9. Calculation and Report

9.1 Compare the readings of the two tubes. If the difference between the two readings is greater than one subdivision on the centrifuge tube (see Table 2 or 3) or 0.025 mL (0.05 % for 200-part tubes) for readings of 0.10 mL (0.20 % for 200-part tubes) and below, the readings are inadmissible and the determination shall be repeated.

9.2 If tubes graduated in 100 mL have been used for the determination, record the sum of the final volumes of sediment and water in each tube obtained, as specified in Section 8, and report this sum as the percentage of sediment and water (see Fig. 3 for reading and reporting sediment and water when using 100-mL cone-shaped centrifuge tubes). Report the results as shown in Table 6.

9.3 If direct-reading 200-part tubes have been used for the determination, the percentage of sediment and water is the average, to three decimal places, of the values read directly from the two tubes. The percentage can only be read directly from a 200-part tube if the tube contains 50 mL or 100 parts of oil.

TABLE 4 Procedure for Reading a 100-mL Cone-Shaped Tube

Volume of Sediment and Water, mL	Read to Nearest, mL
0.0–0.2	0.025
0.2–1.0	0.05
>1.0	0.10

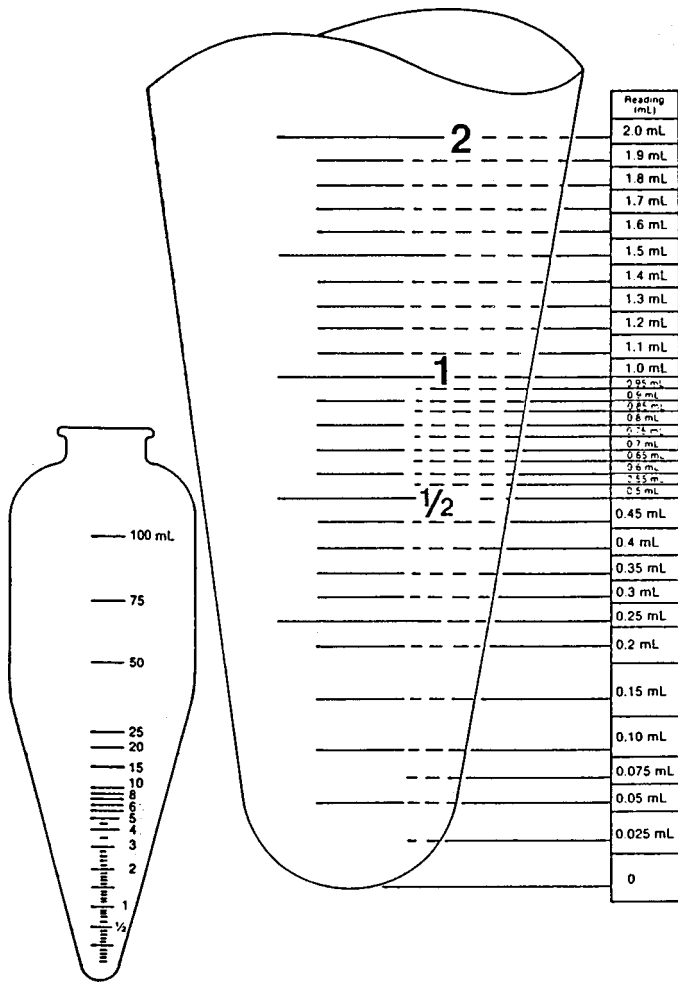


FIG. 3 Procedure for Reading Sediment and Water When Using a Tube Reading in 100 mL

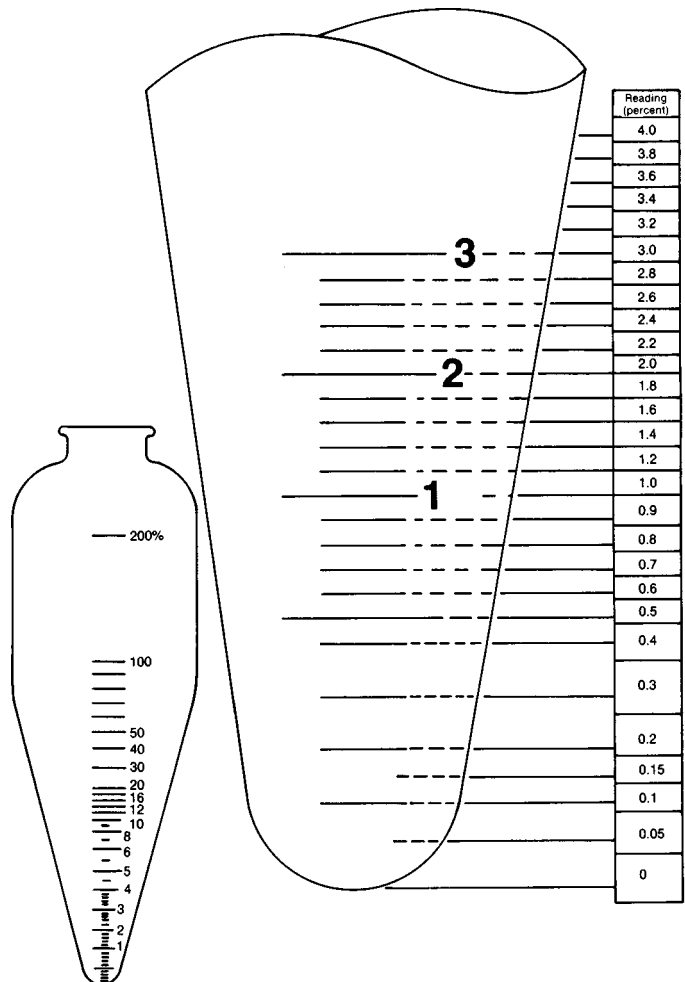


FIG. 4 Procedure for Reading Sediment and Water When Using a Tube Reading in 200 Parts

TABLE 5 Procedure for Reading a 200-Part Cone-Shaped Tube

Volume of Sediment and Water, %	Read to Nearest, %
0.0–0.4	0.05
0.4–2.0	0.10
>2.0	0.20

TABLE 6 Expression of Results

Volume of Sediment and Water, mL		Total Sediment and Water, %
Tube 1	Tube 2	
None visible	Trace	0
None visible	0.025	0.025
0.025	0.025	0.5
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

9.4 If the volume of oil is greater or less than 50 mL, or 100 parts, calculate the percentage of sediment and water as follows:

$$\text{Sediment and water, percent} = (S/V) \times 100 \quad (3)$$

where:

S = volume of sediment and water, mL or parts and
 V = volume of oil tested, mL or parts.

For example, if the total volume of oil tested (V) is 20 mL and the volume of sediment and water (S) is 0.15 mL:

$$\text{Sediment and water, \%} = (0.15/20) \times 100 = 0.75 \quad (4)$$

9.5 If the test conditions outlined in Section 6 are not followed exactly, the following must also be reported:

- 9.5.1 The solvent used and the water saturation temperature.
- 9.5.2 The type and amount of demulsifier (if used).
- 9.5.3 The temperatures of the sample and solvent, the preheater temperature, the centrifuge temperature, and the

temperature of the final mixture after centrifuging.

9.5.4 The number of samples or tubes used in the determination.

10. Precision and Bias

10.1 *Precision*—The precision of this test method is being determined.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias, no statement about bias is being made.

11. Keywords

11.1 centrifuge; centrifuge tube; crude oil; field procedure; sampling; sediment and water; solvent

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 *Kerosine*—Keep away from heat, sparks, or open flame. Keep container closed when not in use. Kerosine's vapor is harmful. Provide adequate ventilation when kerosine is used. Neither an Occupational Safety and Health Administration permissible exposure limit nor an American Conference of Governmental Industrial Hygienists threshold limit value has been established for kerosine. Ingestion of kerosine may cause irritation of the digestive tract; ingestion of large amounts may cause signs of central nervous system depression. Aspiration of this material into the nervous system depression. Repetitive applications of kerosine directly to the skin of laboratory animals over their lifetimes has resulted in skin cancer in the animals. Petroleum hydrocarbons of similar composition and boiling range have been shown to produce kidney damage and tumors in laboratory animals. Avoid skin contact with kerosine. Prolonged or repeated skin contact may cause defatting and drying of the skin.

A1.2 *Stoddard Solvent*—Keep away from heat, sparks, or open flame. Its container should be kept closed when not in use. The solvent's vapor is harmful. Adequate ventilation should be provided when the solvent is used, and airborne concentrations should be kept below the established exposure limits. The permissible exposure limit established by the Occupational Safety and Health Administration for Stoddard solvent is 500 parts/million. The American Conference of Governmental Industrial Hygienists has established a threshold limit value of 100 parts per million. Inhalation of vapors or spray mist should be avoided. Acute overexposure may result in irritation of the throat and lungs. High concentrations may cause central nervous system depression. Aspiration of this material into the lungs may cause chemical pneumonia. Long-term exposure can cause chronic health effects. Chronic overexposure has resulted in liver, heart, and blood disorders. Intense and protracted exposure to the solvent may be associated with an increased risk of kidney cancer, kidney disease, and nerve and brain damage. Avoid skin contact with the solvent. Prolonged or repeated contact with the liquid can result in drying and defatting of the skin that may result in irritation and dermatitis.

A1.3 *Toluene*—Keep away from heat, sparks, or open flame. Keep container closed when not in use. Toluene's vapor

is harmful. Provide adequate ventilation when toluene is used, and airborne concentrations should be kept below the established exposure limits. The Occupational Safety and Health Administrator has established a permissible exposure limit of 200 parts/million, with an acceptable ceiling of 300 parts/million and an acceptable maximum peak of 500 parts/million for 10 min. The American Conference of Governmental Industrial Hygienists has established a threshold limit value of 100 parts/million, with a short-term exposure limit of 150 parts/million for 1 min. Prolonged overexposure through inhalation may cause coughing, shortness of breath, dizziness and intoxication. Aspiration of this material into the lungs may cause chemical pneumonia. Long-term exposure to this material may cause chronic health effects. Toluene may remove fats from the skin and cause chronic dermatitis. Other potential hazards include possible liver, kidney, and nervous system damage and cardiac sensitization to epinephrine. In addition, toluene has been shown to be toxic to the embryo and fetus at high concentrations in animal experiments, however, such studies have failed to demonstrate frank birth defects. Prolonged or repeated skin contact may cause skin to become dry or cracked.

A1.4 *Xylene*—Keep away from heat, sparks, or open flame. Keep container closed when not in use. Xylene's vapor is harmful. Provide adequate ventilation when xylene is used, and keep airborne concentrations below established exposure limits. The Occupational Safety and Health Administration's permissible exposure limit for xylene is 100 parts/million. The American Conference of Governmental Industrial Hygienists has established a threshold limit value of 100 parts/million with a 15-min short-term exposure limit of 150 parts/million. Overexposure through inhalation may cause shortness of breath, dizziness, intoxication, and collapse. Aspiration of this material into the lungs may cause chemical pneumonia. Long-term exposure to this material can cause chronic health effects. Prolonged, repeated exposure to high levels of xylene can induce central nervous system problems and may cause liver and kidney damage. Avoid prolonged or repeated skin contact with xylene. Skin contact may result in delayed skin irritation and blistering.

APPENDIX

(Nonmandatory Information)

X1. DEMULSIFIERS AND WATER SATURATION OF SOLVENTS**X1.1 Water Saturation of Solvents:**

X1.1.1 Fill each of two centrifuge tubes to the 2-mL (4-part) mark with water and then to the 100-mL (200-part) mark with the solvent to be saturated.

X1.1.2 Stopper the tubes and shake vigorously for 30 s, *holding the tubes below eye level*, to disperse the water in the solvent. Loosen the stoppers.

X1.1.3 Place the tubes containing the water/solvent mixture into a sample preheater or heated (nonspinning) centrifuge maintained at a temperature of 60°C (140°F) for a minimum of 30 min.

X1.1.4 Inspect the water/solvent mixture for suspended water droplets before use. If any suspended water is visible, the tubes must be centrifuged at a temperature of 60°C (140°F) for 5 min at a speed sufficient to give a relative centrifugal force of 500 at the tube tip.

X1.1.5 Use the top 50 mL (100 parts) of the mixture from each tube for test purposes. Take particular care not to pour any of the free water in the tip of each tube into the sample.

X1.2 Demulsifiers:

X1.2.1 Although a good commercial crude-oil demulsifier will work effectively with a wide range of crude oils, there are

some crudes for which one demulsifier is more effective than another. If the selected demulsifier does not provide the desired results, others should be tried.

X1.2.2 Overtreatment with a demulsifier can add erroneously to the apparent sediment and water level. Demulsifiers do not in general contain water, but they do have a limited solubility in the solvent-sample and, if added in excessive quantities, can show up after centrifuging as a separate immiscible component at the bottom of the tube. To prevent this problem, the demulsifier should always be used in the form of a demulsifier-solvent stock solution or should be premixed with the solvent to be used in the test. In either case, the quantity of demulsifier to be added to the solvent should be based on tests for the particular demulsifier solvent combination. The demulsifier manufacturer's instructions should be followed when a demulsifier solution is prepared.

X1.3 Demulsifiers and demulsifier-solvent solutions should always be stored in accordance with the manufacturer's recommendations. Each container used to store a demulsifier or demulsifier-solvent solution should be dated, and the contents should be discarded when they reach the demulsifier manufacturer's recommended shelf life.

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