Manual of Petroleum Measurement Standards Chapter 10.4

Determination of Water and/or Sediment in Crude Oil by the Centrifuge Method (Field Procedure)

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Determination of Water and/or Sediment in Crude Oil by the Centrifuge Method (Field Procedure)

1 Scope

1.1 This section describes the field centrifuge method for determining both water and sediment or sediment only in crude oil. This method may not always produce the most accurate results, but it is considered the most practical method for field determination of water and sediment. This method may also be used for field determination of sediment. When a higher degree of accuracy is required, the laboratory procedure described in API *MPMS* Ch. 10.3, *Standard Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure)* (ASTM D4007); API *MPMS* Ch. 10.2, *Standard Test Method for Water in Crude Oil by Distillation* (ASTM D4006); or API *MPMS* Ch. 10.9, *Standard Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration* (ASTM D4928); and the procedure described in API *MPMS* Ch. 10.1, *Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method* (ASTM D473) or API *MPMS* Ch. 10.8, *Standard Test Method for Sediment in Crude Oil by Membrane Filtration* (ASTM D4807) should be used.

1.2 The requirements of this standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. The user of this standard has the responsibility for establishing appropriate safety and health practices and determining the applicability of regulatory limitations prior to use.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API *MPMS* Chapter 8.1, *Manual Sampling of Petroleum and Petroleum Products*

API *MPMS* Chapter 8.2, *Automatic Sampling of Petroleum and Petroleum Products*

API *MPMS* Chapter 8.3, *Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (includes Errata 1 dated March 1996)*

API *MPMS* Chapter 10.1, *Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method*

API *MPMS* Chapter 10.2, *Standard Test Method for Water in Crude Oil by Distillation*

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

API *MPMS* Chapter 10.8, *Standard Test Method for Sediment in Crude Oil by Membrane Filtration*

API *MPMS* Chapter 10.9, *Standard Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration*

ASTM D473 [1], *Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method*

ASTM D3699, *Standard Specification for Kerosine*

ASTM D4006, *Standard Test Method for Water in Crude Oil by Distillation*

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[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, www.astm.org.

ASTM D4007, *Standard Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure)*

ASTM D4057, *Standard Practice for Manual Sampling of Petroleum and Petroleum Products*

ASTM D4177, *Standard Practice for Automatic Sampling of Petroleum and Petroleum Products*

ASTM D4807, *Standard Test Method for Sediment in Crude Oil by Membrane Filtration*

ASTM D4928, *Standard Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration*

ASTM D5854, *Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (includes Errata 1 dated March 1996)*

ASTM E542, *Standard Practice for Calibration of Laboratory Volumetric Apparatus*

3 Terms and Definitions

For the purposes of this document, the following definitions apply.

3.1
sediment
A material that normally exists in crude oils as extremely fine, well dispersed solids that originate in the reservoir from which the crude came, or in drilling fluids used to drill the wells, which may take the form of sand, clay, shale, or rock particles. Other sediments such as scale can be picked up from tubing, pipe, tanks, and other production and transportation equipment. Sediment, as defined in this standard, shall have a density greater than water and will not float at test conditions.

3.2
demulsifier-stock solution
A mixture of demulsifier and solvent in a fixed ratio (e.g. 25 % to 75 %) which is added to the test solvent to form a solution to cause a clear and distinct separation between the oil and water interface.

3.3
demulsifier-solvent solution
A mixture of any one of the approved solvents and a demulsifier, or a demulsifier-stock solution which is used as a diluent to help the separation of water and sediment from the sample being tested.

3.4
solvent
Any one of the approved liquids capable of dissolving the hydrocarbon fraction of the crude oil to form a solution. A solvent is added to reduce the viscosity of the crude oil and promote separation of the crude oil hydrocarbons from the water and sediment in the sample being tested.

4 Significance and Use

A determination of water and/or sediment content is required to accurately determine the net volumes of crude oil involved in sales, taxation, exchanges, inventories, and custody transfers. An excessive amount of water and/or sediment 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.

For determination of both water and sediment, known volumes of crude oil, demulsifier, and solvent (water-saturated if required) are placed in a centrifuge tube and heated to 60 °C ±3 °C (140 °F ±5 °F). After centrifuging, the volumes of sediment and water at the bottom of the tube are read. For some crude oils, temperatures of 71 °C (160 °F) or higher may be required to completely melt wax crystals in crude oils. Temperatures shall be maintained above the cloud point of the crude oil being tested.

For determination of sediment only, known volumes of crude oil, solvent, demulsifiers, and water are placed in a centrifuge tube and heated to 71 °C ±3 °C (160 °F ±5 °F). After centrifuging, the volume of the sediment at the bottom of the tube is read.

5 Test Solvents

5.1 General

Kerosine, Stoddard solvent, toluene, and xylene are satisfactory for use in field testing. Solvents and demulsifiers are mixed with samples in the centrifuge tubes to reduce the viscosity and to allow water and sediment to separate and fall to the tip of the tube during the testing procedure. Also, solvents are used to help dissolve paraffin and asphaltenes to prevent them from being centrifuged to the tip of the tube and included in the test results. Different types of crude oil may require the use of different solvents to obtain accurate results. For example, kerosine and Stoddard solvent will not dissolve asphaltenes.

Testing should be performed to determine the best solvent to use on a particular crude type.

Annex A contains general information regarding the physical characteristics of these solvents, as well as fire, safety, and health considerations relating to their use. Kerosine shall meet the specifications of ASTM D3699.

Information concerning physical characteristics, safety and health risks, and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

5.2 Use of Solvents

Toluene and xylene shall be saturated with water at the test temperature used, and maintained at that temperature until used. A procedure for the saturation of solvents is given in Annex B. The water-saturated solvent shall be free from suspended water at the time of use. Toluene or xylene is recommended for water and sediment determinations involving asphaltenic crude oils.

Stoddard solvent and kerosine do not have to be saturated with water, since the solubility of water in these solvents is not significant at the test temperature.

6 Demulsifier

Unless the results of testing indicate that a demulsifier is not required for a particular crude type, demulsifiers shall be used to promote the separation of water from the sample and make it easier to read the water/oil interface. Demulsifiers should be mixed according to the manufacturer's recommendations and should not add to the volume of sediment and water determined. The demulsifier shall always be used in the form of a demulsifier stock solution or be premixed with the solvent to be used in the test. The ratio of demulsifier to solvent should be recorded in order to be able to duplicate results if necessary. Testing should be done to determine which demulsifier provides the best results with a specific crude type. See Annex A and B for further information about demulsifiers.

7 Apparatus

7.1 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 g at the tip of the tubes. The rotation speed necessary to produce a relative centrifugal force of 500 g for various diameters of swing can be determined from Table 1 or from the following equation:

$$\text{rpm} = \alpha\sqrt{\frac{rcf}{d}} \tag{1}$$

where

rpm is the rotation speed in revolutions per minute,

rcf is the relative centrifugal force in multiples of gravity,

d is the diameter of swing, in millimeters or inches, measured between the tips of opposite tubes when the tubes are in their fully extended rotating position,

α is 1335 rpm for d in millimeters, and 265 rpm for d in inches.

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

Diameter of swing "*d*" [a]		Rotation Speed (revolutions per minute)
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 opposite tubes when the tubes are in their fully extended 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.

The centrifuge machine shall be heated and be capable of maintaining the sample within 8 °C (15 °F) of the test temperature. For example, the minimum acceptable temperature after centrifuging is 52 °C (125 °F) at a test temperature of 60 °C (140 °F) or 63 °C (145 °F) at a test temperature of 71 °C (160 °F).

7.2 Centrifuge Tubes

Centrifuge tubes shall be cone-shaped and 8 in. (203 mm) or 6 in. (167 mm) in length. Tubes shall conform to the dimensions given in Annex C and shall be made of thoroughly annealed glass. The tube graduation marks' accuracy shall be volumetrically verified or gravimetrically certified before field use of the tube, in accordance with ASTM E542, using National Institute of Standards and Technology (NIST) or other internationally accepted traceable equipment.

The 6 in. tubes are more commonly used in field applications due to the smaller size and weight of the centrifuge required. The 8 in. tubes are easier to read due to the increased spacing of the graduations. In either case the tubes can be 100-mL or 200-part tubes. The computation methods for the total percent of sediment and water are different based on the type of tube selected. For 100-mL tubes, the sediment and water volume readings from each tube are added. For 200-part tubes, the sediment and water volume readings from each tube are averaged. See Figure 1 for example of a 100-mL tube and Table 2 for how to read a 100-mL tube. See Figure 2 for an example of a 200-part tube and Table 3 for how to read a 200-part tube.

7.3 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 (200-part) mark and capable of heating the sample to 71 °C ±3 °C (160 °F ±5 °F).

7.4 Sample Thermometer

A sample thermometer shall have graduations at intervals of 1 °C (2 °F) or less and shall be accurate to ±0.5 °C (±1 °F). A bi-metal, dial-type thermometer is normally used.

8 Sampling

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, with care, in an appropriate analytical device, such as a centrifuge tube, to maintain its compositional integrity prior to testing.

Samples obtained and handled as specified in API *MPMS* Ch. 8.1 (ASTM D4057), Ch. 8.2 (ASTM D4177), and Ch. 8.3 (ASTM D5854) shall be used for this method.

9 Procedure—Determination of Water and Sediment

9.1 General

A test is composed of two tubes of the same sample. A centrifuge test is valid when two successive test results are obtained which are within the allowable tolerances outlined in Section 10.

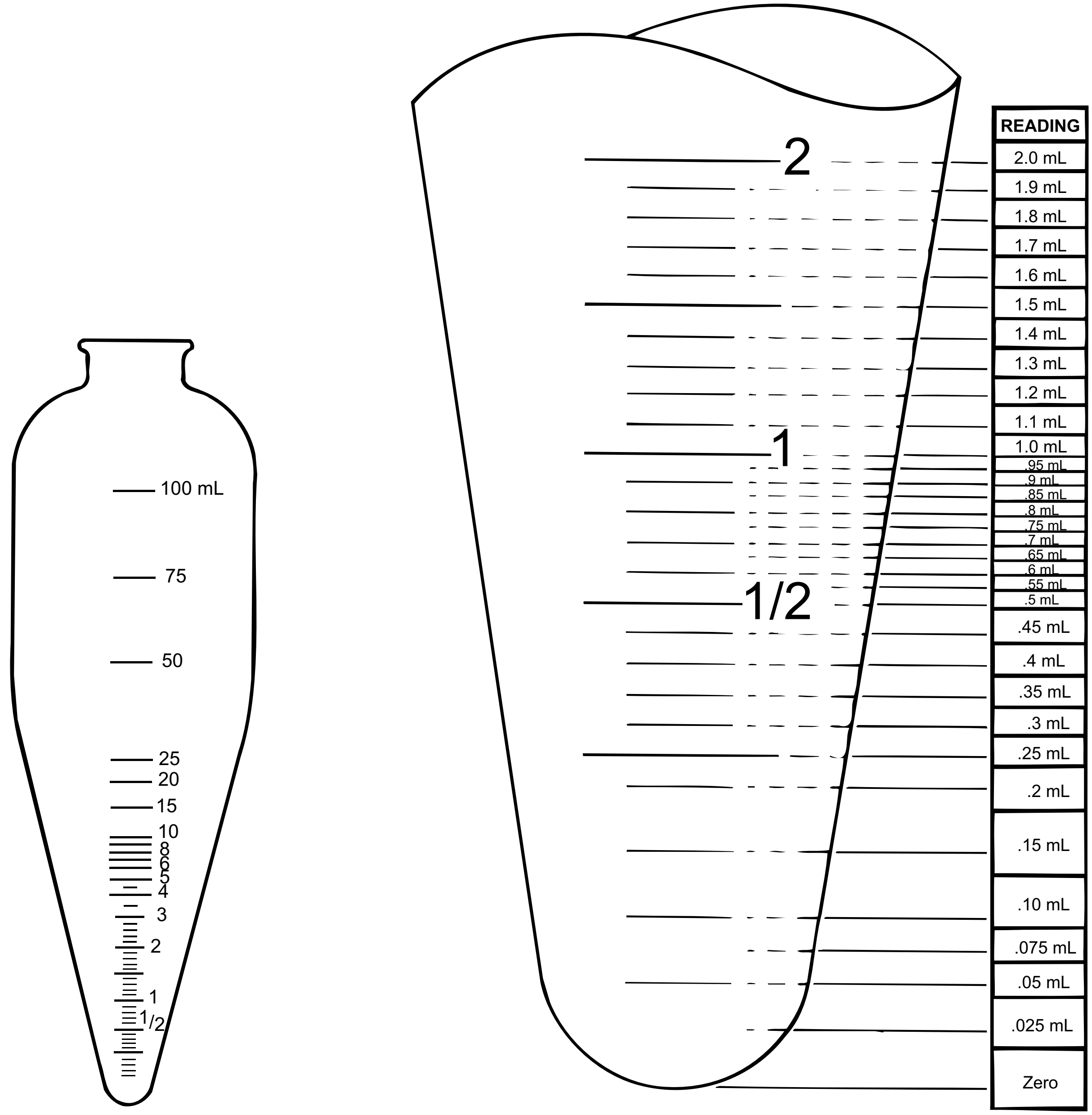

Figure 1—Reading a 100-mL Centrifuge Tube

Table 2—Procedure for Reading a 100-mL Cone-Shaped Tube

Volume (mL)	Read to Nearest (mL)
0.0 to 0.1	0.025
0.1 to 1.0	0.050
> 1.0	0.100

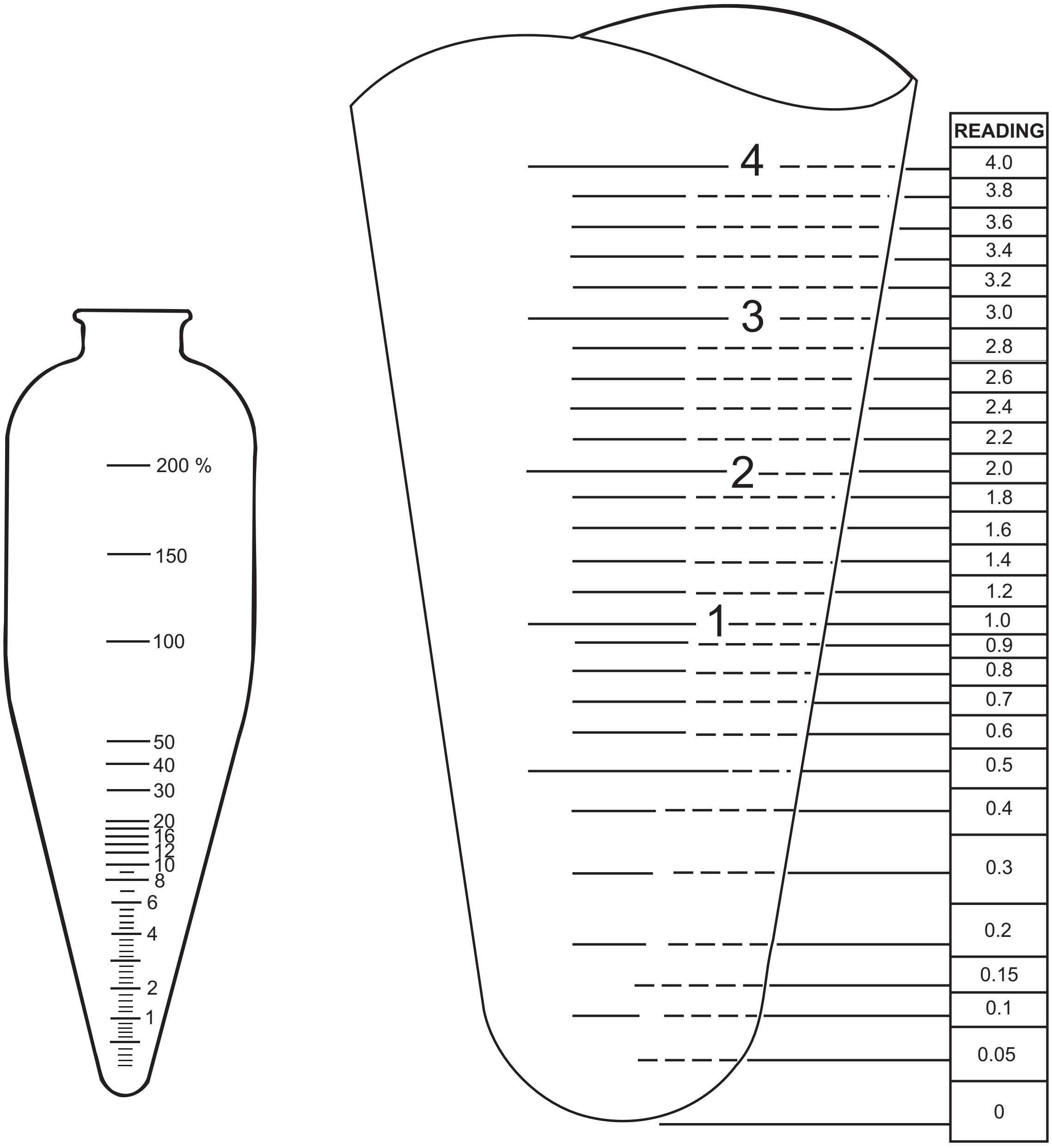

Figure 2—Reading a 200-Part Centrifuge Tube

Table 3—Procedure for Reading a 200-Part Cone-Shaped Tube

Volume (%)	Read to Nearest (%)
0.0 to 0.2	0.05
0.2 to 1.0	0.10
> 1.0	0.20

The centrifuge tube will only produce accurate results if the exact required volume of 50-mL (100-parts) of sample to be tested is in the centrifuge tube. The volume of solvent-solution, which is added to the sample, is not as critical since it is used as a diluent and should not add to or take away from the sediment and water content.

Caution should be used when using solvents that require water saturation. Water results will be affected if the volume of solvent is not properly water saturated.

NOTE Maintaining stoppered tubes throughout the entire process may help maintain the representative nature of the sample.

9.2 Procedure

The following steps shall be performed for determination of water and sediment. See Annex D for an outline of the procedure.

1) Fill each of two centrifuge tubes with exactly 50-mL (100-parts) of the sample.

2) If the demulsifier-stock solution has not been premixed into the solvent solution, add the required demulsifier-stock solution to 50-mL (100-parts) of solvent only, to insure that the demulsifier-stock solution dissolves. Then fill each tube with the demulsifier-solvent solution to the 100-mL (200-part) mark.

   NOTE Where the crude oil is 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 shall 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.

3) Stopper each tube tightly and invert the tubes a minimum of ten times to ensure that the oil and solvent-solution are uniformly mixed.

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 so that contact will be avoided if stopper blowout occurs.

4) 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 a test temperature of 60 °C ±3 °C (140 °F ±5 °F).

   NOTE Temperatures of 71 °C ±3 °C (160 °F ±5 °F) or more may be necessary depending on the properties of the sample. Experience will provide the temperature(s) that will produce the most accurate results.

5) Secure the stoppers and again invert the tubes ten times to ensure uniform mixing of the oil and solvent-solution.

6) Place the tubes in the trunnion cups on opposite sides of the centrifuge to establish a balanced condition. Spin for at least 5 minutes at a minimum relative centrifugal force of 500 g.

   NOTE Experience may indicate that the initial spin time needs to be longer than 5 minutes, depending on the properties of the sample being tested.

7) Immediately after the centrifuge comes to rest use a sample thermometer to verify the temperature of the sample. Do not disturb the oil-water interface with the thermometer. The sample temperature shall be within 8 °C (15 °F) of the test temperature for the test to be considered valid. If the temperature meets this requirement, continue on to step 8. If the temperature does not meet the requirement reinitiate the procedure, beginning with step 4.

8) Hold the tube in an upright (vertical) position. Read and record the volume of water and sediment at the bottom of each tube as indicated in Table 2 and Figure 1 for 100-mL tubes or Table 3 and Figure 2 for 200-part tubes.

9) Reheat both tubes to the initial test temperature and return the tubes without agitation to the centrifuge, and spin for at least 5 minutes at a minimum relative centrifugal force of 500 g. Repeat step until two consecutive consistent readings are obtained in accordance with Section 10.

10) For a test to be considered valid, a clear interface shall be observed between the oil layer and the separated water/sediment. No identifiable layering such as asphaltenes, congealed paraffin, or an emulsion should be present immediately above the oil and water/sediment interface. In such cases, one or more of the following remedies may be effective:

    a) Increase the temperature of the sample to 71 °C ±3 °C (160 °F ±5 °F).

    b) Add additional demulsifier-stock solution (i.e. drops). The demulsifier shall not, however, contribute to the volume of water and sediment.

    c) Obtain new samples and retest using the centrifuge method, or other approved API/ASTM test methods.

10 Calculation and Reporting—Water and Sediment

10.1 General

A test is comprised of two tubes of the same sample. Compare the readings of the two tubes. If the difference between the two readings is greater than one subdivision on the centrifuge tube, the test is invalid and shall be repeated.

10.2 100-mL Tubes

For 100-mL tubes, read and record the volume of water and sediment in each tube. Add the readings together and report the sum as the percentage of water and sediment (see Figure 1, Table 2 for reading and reporting water and sediment when using 100-mL cone-shaped centrifuge tubes). Report the results as shown in Table 4.

10.3 200-part Tubes

For 200-part tubes, the percentage of water and sediment is the average, to three decimal places, of the values read directly from the two tubes (see Figure 2, Table 3). The percentage can only be read directly from a 200-part tube if the tube contains 100-parts (50-mL) of oil. Report the results as shown in Table 4.

Table 4—Expression of Results for 100-mL Tubes and 200-Part Tubes

100-mL Tubes Volume of water and/or sediment (mL)		200-Part Tubes Volume of water and/or sediment (%)		Read and Recorded Total water and/or sediment
Tube 1	Tube 2	Tube 1	Tube 2	(%)
< 0.0125	< 0.0125	< 0.025	< 0.025	0.000
< 0.0125	0.025	< 0.025	0.050	0.025
0.025	0.025	0.050	0.050	0.050
0.025	0.050	0.050	0.100	0.075
0.050	0.050	0.100	0.100	0.100
0.050	0.075	0.100	0.150	0.125
0.075	0.075	0.150	0.150	0.150
0.075	0.100	0.150	0.200	0.175
0.100	0.100	0.200	0.200	0.200
0.100	0.150	0.200	0.300	0.250

NOTE 1 100-mL Tubes—If the total water and/or sediment volume for both tubes is less than 0.025 % (0.0125 mL), the water and sediment volume shall be read and recorded as 0.000 % (0.000 mL).

NOTE 2 200-Part Tubes—If the total water and/or sediment average percentage for the two tubes is less than 0.025 %, the water and sediment volume shall be read and recorded as 0.000 %.

11 Procedure—Determination of Sediment Only

11.1 General

If other approved API/ASTM methods are used for water determination then the following test method for sediment may be used for the detection or quantification of sediment.

A test is comprised of two tubes of the same sample. A centrifuge test is valid when two successive test results are obtained which are within the allowable tolerances outlined in Section 12.

Solvents normally requiring water saturation do not have to be water-saturated for this procedure since water will be added to the test sample.

The centrifuge tube will only produce accurate results if the exact required volume of 50-mL (100 parts) of sample to be tested is in the centrifuge tube. The volume of solvent-solution, which is added to the sample, is not as critical since it is used as a diluent and should not add to or take away from the sediment content.

11.2 Procedure

The following steps shall be performed for determination of sediment only. See Annex E for an outline of the procedure.

1) Fill each of two centrifuge tubes with exactly 50-mL (100-parts) of the sample.

2) If the demulsifier-stock solution has not been premixed into the solvent solution, add the required demulsifier-stock solution to 50-mL (100-parts) of solvent only, to insure that the demulsifier-stock solution dissolves. Then fill each tube with the demulsifier-solvent solution to the 100-mL (200-part) mark.

   NOTE 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 shall 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.

3) Add approximately 3 to 4 drops of demineralized or distilled water to the centrifuge tube to allow for a clear and distinct reading.

4) Stopper each tube tightly and invert the tubes a minimum of ten times to ensure that the oil and solvent-solution are uniformly mixed.

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 so that contact will be avoided if stopper blowout occurs.

5) 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 a test temperature of 71 °C ±3 °C (160 °F ±5 °F).

6) Secure the stoppers and again invert the tubes ten times to ensure uniform mixing of the oil and solvent-solution.

7) Place the tubes in the trunnion cups on opposite sides of the centrifuge to establish a balanced condition. Spin for at least 5 minutes at a minimum relative centrifugal force of 500 g.

   NOTE Experience may indicate that the initial spin time needs to be longer than 5 minutes depending on the properties of the sample being tested.

8) Immediately after the centrifuge comes to rest use a sample thermometer to verify the temperature of the sample. Do not disturb the oil-water interface with the thermometer. The sample temperature shall be within 8 °C (15 °F) of

the test temperature for the test to be considered valid. If the temperature meets this requirement, continue on to step 9. If the temperature does not meet the requirement reinitiate the procedure, beginning with step 5.

9) Hold the tube in an upright (vertical) position. Read and record the volume of sediment at the bottom of each tube as indicated in Table 2 and Figure 1 for 100-mL tubes or Table 3 and Figure 2 for 200-part tubes.

10) Reheat both tubes to the initial test temperature and return the tubes without agitation to the centrifuge, and spin for at least 5 minutes at a minimum relative centrifugal force of 500 g. Repeat step until two consecutive consistent readings are obtained in accordance with Section 12.

11) For a test to be considered valid, a clear interface shall be observed between the oil layer and the separated water/sediment. No identifiable layering such as asphaltenes, congealed paraffin, or an emulsion should be present immediately above the oil and water/sediment interface. In such cases, one or more of the following remedies may be effective.

   a) Increase the temperature of the sample.

   b) Add additional demulsifier-stock solution (i.e. drops). The demulsifier shall not, however, contribute to the volume of sediment.

   c) Obtain new samples and retest using the centrifuge method, or other approved API/ASTM test methods.

12 Calculation and Reporting—Sediment Only

12.1 General

A test is comprised of two tubes of the same sample. If the test is used for determination of quantity then compare the readings of the two tubes. If the difference between the two readings is greater than one subdivision on the centrifuge tube, the test is invalid and shall be repeated. If the test is used for detection only, and sediment is detected, then the sample may be retested using an API/ASTM approved test method to determine the quantity of the sediment.

12.2 100-mL Tubes

For 100-mL tubes, read and record the volume of sediment in each tube. Add the readings together and report the sum as the percentage of sediment (see Figure 1, Table 2 for reading and reporting sediment when using 100-mL cone-shaped centrifuge tubes). Report the results as shown in Table 4.

12.3 200-part Tubes

For 200-part tubes, the percentage of sediment is the average, to three decimal places, of the values read directly from the two tubes (see Figure 2, Table 3). The percentage can only be read directly from a 200-part tube if the tube contains 100-parts (50-mL) of oil. Report the results as shown in Table 4.

13 Precision

No precision for the test method described herein has been established. This is a field method. Experience has shown that the determination of both water and sediment using this test method has sometimes been as precise as other accepted test methods.

14 Bias

Since there is no accepted reference material suitable for determining the bias for the procedure described in these sections for measuring water and/or sediment in crude oil by the centrifuge method (field procedure), no statement about bias is being made.

Annex A
(informative)

Precautionary Information

A.1 Physical Characteristics and Fire Considerations

Information regarding particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

A.1.1 Kerosine (ASTM D3699)

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

Caution—Kerosine is combustible and should be kept away from heat, sparks, or open flame. Kerosine containers should be kept closed when they are not in use. Fires should be extinguished with foam, dry chemical, or carbon dioxide. Spills should be soaked up with clay, diatomaceous earth, or a similar material.

A.1.2 Stoddard Solvent

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

Caution—Stoddard solvent is combustible and should be kept away from heat, sparks, or open flame. Stoddard solvent containers should be kept closed when they are not in use. Fires should be extinguished with foam, dry chemical, or carbon dioxide. Spills should be soaked up with clay, diatomaceous earth, or a similar material.

A.1.3 Toluene

The typical characteristics of toluene are a minimum flash point of 4 °C (40 °F), an ignition temperature of 480 °C (896 °F), a boiling point of 111 °C (231 °F), an upper flammability limit of 7.1 percent by volume, a lower flammability limit of 1.2 percent by volume, a molecular weight of 92, an American Public Health Association (APHA) color of ten (per ASTM 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 H_2SO_4.

Caution—Toluene is flammable and should be kept away from heat, sparks, or open flame. Toluene containers should be kept closed when they are not in use. Fires should be extinguished with water spray, foam, dry chemical, or carbon dioxide. Spills should be soaked up with clay, diatomaceous earth, or a similar material.

A.1.4 Xylene

The typical characteristics of xylene are a minimum flash point of 27 °C (81 °F), an ignition temperature of 527 °C (982 °F), a boiling point of 139 °C (282 °F), an upper flammability limit of 7.0 percent by volume, a lower flammability limit of 1.1 percent by volume, a molecular weight of 106, an APHA color of not more than ten (per ASTM D1209), a boiling range of 137 °C to 144 °C (279 °F to 291 °F), and 0.002 percent residue after evaporation. Xylene passes the ACS test for substances darkened by H_2SO_4.

Caution—Xylene is flammable and should be kept away from heat, sparks, or open flame. Xylene containers should be kept closed when they are not in use. Fires should be extinguished with water spray, foam, dry chemical, or carbon dioxide. Spills should be soaked up with clay, diatomaceous earth, or a similar material.

A.2 Safety and Health Considerations

A.2.1 General Considerations

Potential health effects that result from chemical exposure depend on the chemical concentration and the length of exposure. Everyone should minimize exposure to chemicals. The following general precautions are suggested.

a) Minimize skin and eye contact and breathing of vapors.

b) Keep chemicals away from the mouth, since they can be harmful or fatal if swallowed or aspirated.

c) Keep chemical containers closed when they are not in use.

d) Keep work areas well ventilated and as clean as possible.

e) Promptly clean up spills in accordance with pertinent safety, health, and environmental regulations.

f) Observe established exposure limits and use proper protective clothing and equipment.

NOTE For permissible exposure limits (PELs), consult the most recent edition of the Occupational Safety and Health Standards, 29 *Code of Federal Regulations* Subpart Z, “Toxic and Hazardous Substances” Section 19101000, and following. Also consult the most recent edition of the ACGIH booklet, *Threshold Limit Values and Biological Exposure*. (See Section 2 for complete reference information.)

A.2.2 Kerosine

Health effects can result from exposure to kerosine through contact with the skin and eyes, breathing of vapors, swallowing, or aspiration. Kerosine can exhibit local irritant properties that may be manifested by dermatitis of the skin, stinging of the eyes, nose, or throat, or respiratory irritation. Acute exposure to kerosine may result in adverse systemic effects including effects on the central nervous, respiratory, or gastrointestinal systems. Symptoms of systemic effects may include vomiting, diarrhea, and in severe cases, drowsiness and central nervous system depression progressing to coma and death.

There may also be long-term (chronic) health effects of varying severity from exposure to kerosine.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

A.2.3 Stoddard Solvent

Health effects can result from exposure to Stoddard solvent through contact with the skin and eyes, breathing of vapors, swallowing, or aspiration. Stoddard solvent exhibits local irritant properties that may be manifested by dermatitis of the skin, stinging of the eyes, nose, or throat, or respiratory irritation. Acute exposure to Stoddard solvent may result in adverse systemic effects including effects on the central nervous, respiratory, or gastrointestinal systems. Symptoms of systemic effects may include headache, fatigue, nausea, vomiting, cough, and gastrointestinal or respiratory difficulties.

There may also be long-term (chronic) health effects of varying severity from exposure to Stoddard solvent.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

A.2.4 Toluene

Health effects can result from exposure to toluene through contact with the skin and eyes, breathing of vapors, swallowing, or aspiration. Toluene exhibits local irritant properties that may be manifested by dermatitis of the skin, stinging of the eyes, nose, or throat, or respiratory irritation. Acute exposure to toluene may result in adverse systemic effects including effects on the central nervous, cardiovascular, respiratory, or gastrointestinal systems.

Symptoms of acute systemic effects may include headache, fatigue, disturbed vision, dizziness, confusion, numbness of the hands and feet, heart palpitations, anorexia, and gastrointestinal or respiratory difficulties. Overexposure may lead to coma and death.

There may also be long-term (chronic) health effects of varying severity from exposure to toluene.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

A.2.5 Xylene

Health effects can result from exposure to xylene through contact with the skin and eyes, breathing of vapors, swallowing, or aspiration. Xylene exhibits local irritant properties that may be manifested by dermatitis of the skin, stinging of the eyes, nose, or throat, or respiratory irritation. Acute exposure to xylene may result in adverse systemic effects including effects on the central nervous, cardiovascular, respiratory, or gastrointestinal systems. Symptoms of acute systemic effects may include headache, fatigue, disturbed vision, dizziness, confusion, numbness of the hands and feet, heart palpitations, anorexia, and gastrointestinal or respiratory difficulties. Overexposure may lead to coma and death.

There may also be long-term (chronic) health effects of varying severity from exposure to xylene.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

A.2.6 Demulsifiers

Various chemical demulsifiers may be used in the procedures described in this manual.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the Material Safety Data Sheet.

Annex B
(informative)

Demulsifiers and Water Saturation of Solvents

B.1 Water Saturation of Solvents

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

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

B.1.3 Place the tubes containing the water/solvent mixture into a sample preheater. Heat the tubes to 60 °C (140 °F) or other test temperature and maintain this temperature for a minimum of 30 minutes.

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

B.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.

B.2 Demulsifiers

B.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. Oil-soluble demulsifiers, which are generally used in this procedure, normally consist of an aromatic base (usually xylene or toluene) plus a proprietary active ingredient. Water-soluble demulsifiers normally consist of an alcohol base plus a proprietary active ingredient. For specific information regarding demulsifiers, consult Annex A and the Material Safety Data Sheet for the particular demulsifier in use.

B.2.2 Excessive amounts of demulsifier will cause errors. Demulsifiers do not, in general, contain water, but they do have a limited solubility in the solvent-sample mix 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-stock solution or should be premixed with the solvent to be used in the test. The ratio of demulsifier to solvent should be recorded in order to be able to duplicate results using the solution if necessary. In either case, the quantity of demulsifier to be added to the solvent should be based on tests for the particular demulsifier-solvent solution combination. The demulsifier manufacturer's instructions should be followed when a demulsifier-solvent solution is prepared.

B.2.3 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 manufacturer's recommended shelf life.

Annex C
(normative)

Specifications for Centrifuge Tubes

Centrifuge tubes shall be cone-shaped and 8 in. (Figure C-1) or 6 in. (Figure C-2) in length and conform to the dimensions shown. The tubes shall be made of thoroughly annealed glass.

A 100-mL tube shall conform to the dimensions shown in Figure C-1. A 200-part tube shall conform to the dimensions shown in Figure C-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 8-in. (203-mm) and 6-in. (167-mm) tubes shall be in accordance with the requirements of Table C-1 and Table C-2, respectively. The scale errors for a centrifuge tube shall not exceed the tolerances specified in these tables. The graduation requirements and scale error tolerances shown in Table C-1 and Table C-2 apply to calibrations made by reading the bottom of the shaded meniscus of air-free water at a temperature of 68 °F (20 °C). The graduations on each tube shall be clearly numbered as shown in Figure C-1 and Figure C-2.

The tube graduation marks' accuracy shall be volumetrically verified or gravimetrically certified before field use of the tube, in accordance with ASTM E 542, using National Institute of Standards and Technology (NIST)-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 Tables 2 or 3. Tubes may have additional marks added at the 0.025-mL and 0.075-mL (0.05- and 0.15-parts) which shall also be volumetrically verified or gravimetrically certified.

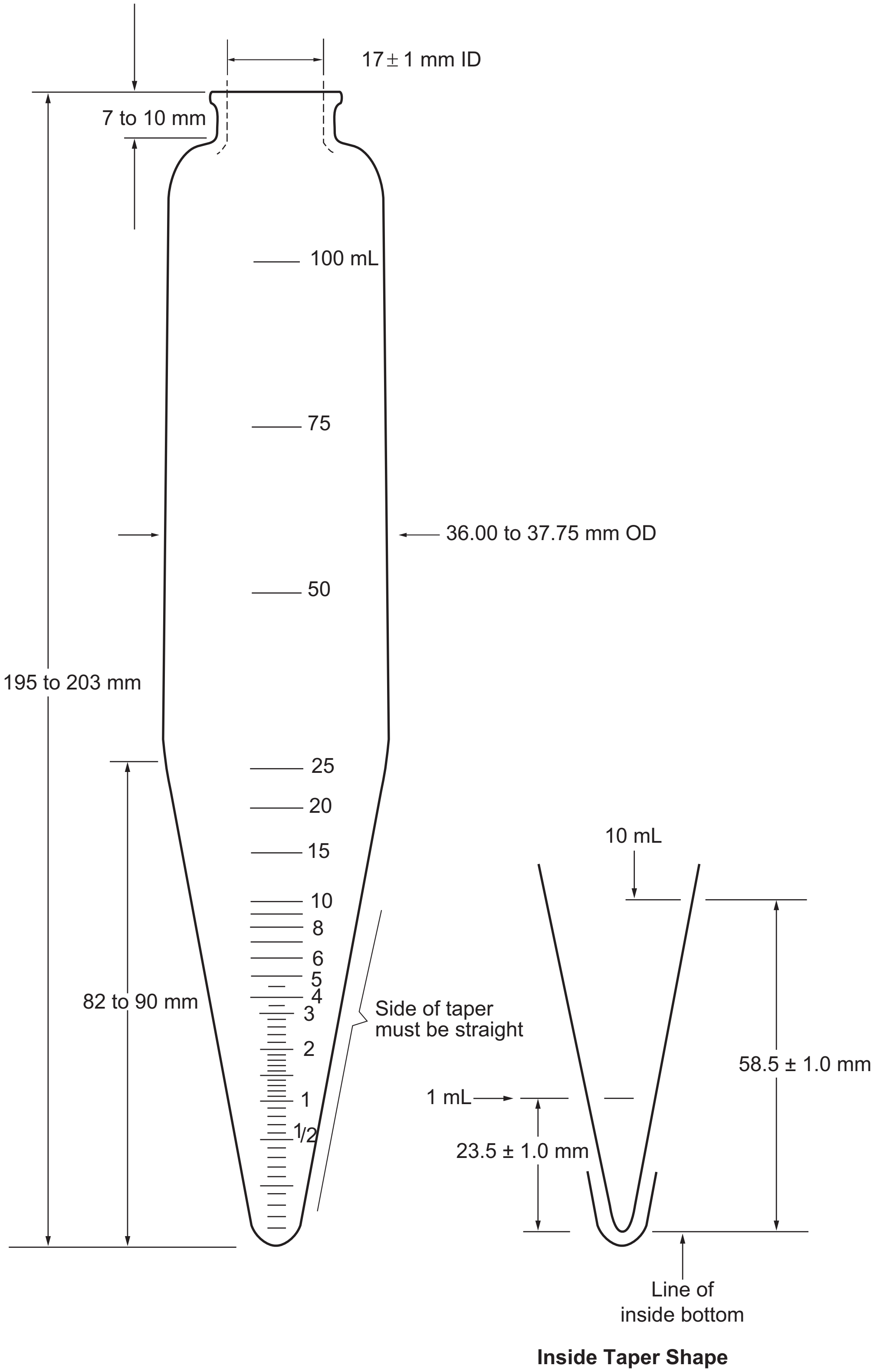

Figure C-1—8-in. (203-mm) Cone-Shaped Centrifuge Tube

Table C-1—Minimum Graduation Requirements and Maximum Calibration Tolerances for 8-in. (203-mm) Cone-Shaped Tubes

Range	Subdivision (mL)	Volume Tolerance (mL)
Above 0.0 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 10.0	1.0	± 0.50
Above 10.0 to 25.0	5.0	± 1.00
Above 25.0 to 100.0	25.0	± 1.00

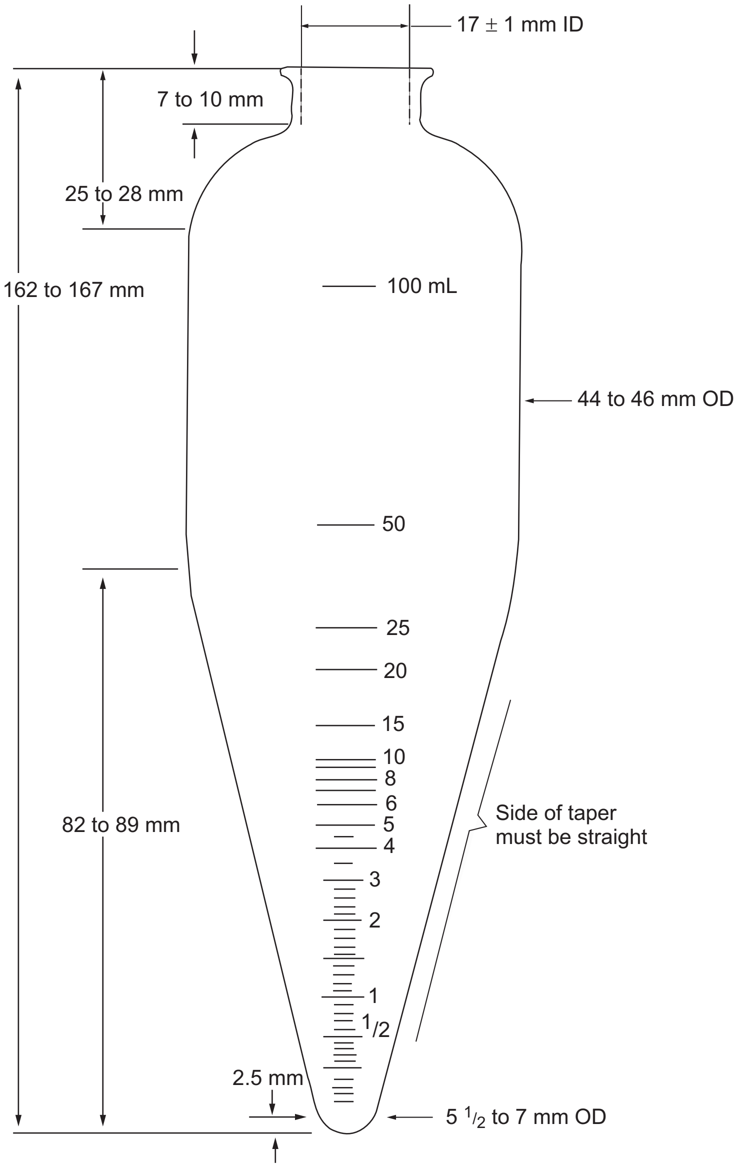

Table C-2—6-in. (167-mm) Cone-Shaped Centrifuge Tube

Table C-2—Minimum Graduation Requirements and Maximum Calibration Tolerances for 6-in. (167-mm) Cone-Shaped Tubes

Range	Subdivision (mL)	Volume Tolerance (mL)
Above 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 to1.0	0.10	± 0.075
Above 1.0 to1.5	0.10	± 0.10
Above 1.5 to 2.0	0.10	± 0.20
Above 2.0 to 3.0	0.20	± 0.30
Above 3.0 to 5.0	0.50	± 0.50
Above 5.0 to10.0	1.00	± 0.75
Above 10.0 to 25.0	5.00	± 1.00
Above 25.0 to 100.0	[a]	± 1.50

[a] Graduations at 50 and 100.

Annex D
(informative)

Procedure Outline—Determination of Water and Sediment

1. Fill each of the two tubes with exactly 50 mL (100 parts) of the sample.
2. Fill each tube with the solvent solution to the 100-mL (200-part) mark.
3. Stopper each tube tightly and invert 10 times.
4. Loosen the stoppers and immerse the tubes in a pre-heater. Heat the contents to a test temperature of 60 °C ±3 °C (140 °F ±5 °F).
5. Stopper each tube tightly and invert 10 times.
6. Place the tubes in the centrifuge machine, in a balanced condition, and spin for 5 minutes.
7. Immediately after the centrifuge comes to rest, use a sample thermometer to verify that the sample temperature is within 8 °C (15 °F) of the test temperature.

Note: If the sample temperature is within 8 °C (15 °F) then go to step 8. If the sample temperature is not within 8 °C (15 °F) then reinitiate the procedure beginning with step 4.

8. Read and record the volume of water and sediment at the bottom of each tube.
9. Reheat the tubes to the initial test temperature and return them, without agitation, to the centrifuge machine. Spin for an additional 5 minutes. Repeat the step until two consecutive consistent readings are obtained.

Note: For the test to be considered valid, a clear interface shall be observed between the oil layer and the separated water/sediment. No emulsions should be present immediately above the oil and water/sediment interface. A test is comprised of TWO TUBES of the SAME SAMPLE. Compare the readings of the two tubes. If the difference is greater than one subdivision on the centrifuge tube, the test is invalid and shall be repeated.

10. Calculating and reporting.

For 100-mL tubes: read and record the volume of water and sediment in each tube. Add the readings together and report the sum as the percentage of water and sediment.

For 200-part tubes: the percentage of water and sediment is the average, to three decimal places, of the values read directly from the two tubes.

Annex E
(informative)

Procedure Outline—Determination of Sediment Only

1. Fill each of the two tubes with exactly 50 mL (100 parts) of the sample.
2. Fill each tube with the solvent solution to the 100-mL (200-part) mark.
3. Add approximately 1 mL (3 to 4 drops) of demineralized or distilled water to the centrifuge tube.
4. Stopper each tube tightly and invert 10 times.
5. Loosen the stoppers and immerse the tubes in a pre-heater. Heat the contents to a test temperature of 71 °C ±3 °C (160 °F ±5 °F).
6. Stopper each tube tightly and invert 10 times.
7. Place the tubes in the centrifuge machine, in a balanced condition, and spin for 5 minutes.
8. Immediately after the centrifuge comes to rest, use a sample thermometer to verify that the sample temperature is within 8 °C (15 °F) of the test temperature.

Note: If the sample temperature <u>is</u> within 8 °C (15 °F) then go to step 9. If the sample temperature <u>is not</u> within 8 °C (15 °F) then reinitiate the procedure beginning with step 5.

9. Read and record the volume of sediment at the bottom of each tube.
10. Reheat the tubes to the initial test temperature and return them, without agitation, to the centrifuge machine. Spin for an additional 5 minutes. Repeat the step until two consecutive consistent readings are obtained.

Note: For the test to be considered valid, a clear interface shall be observed between the oil layer and the separated water/sediment. No emulsions should be present immediately above the oil and water/sediment interface. A test is comprised of TWO TUBES of the SAME SAMPLE. Compare the readings of the two tubes. If the difference is greater than one subdivision on the centrifuge tube, the test is invalid and shall be repeated.

11. Calculating and reporting.

    For 100- mL tubes: read and record the volume of water and sediment in each tube. Add the readings together and report the sum as the percentage of water and sediment.

    For 200-part tubes: the percentage of water and sediment is the average, to three decimal places, of the values read directly from the two tubes.

Bibliography

[1] OSHA 29 C*FR* Subpart Z [2], "Toxic and Hazardous Substances," Section 1910.1000

[2] API *MPMS* Chapter 8.4, *Standard Practice for Sampling and Handling Fuels for Volatility Measurement*

[3] ACGIH—*Threshold Limit Values and Biological Exposure Indices for 1987–1988*

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[2] U.S. Department of Labor, Occupational Safety and Health Administration, 200 Constitution Avenue, NW, Washington, DC 20210, www.osha.gov.

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