Manual of Petroleum Measurement Standards Chapter 10—Sediment and Water

Section 8—Standard Test Method for Sediment in Crude Oil by Membrane Filtration

SECOND EDITION, NOVEMBER 2005

REAFFIRMED, MARCH 2010

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AMERICAN PETROLEUM INSTITUTE

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Measurement Coordination

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Designation: D 4807 – 05ε1

An American National Standard

Designation: Manual of Petroleum Measurement Standards (MPMS), Chapter 10.8

Standard Test Method for Sediment in Crude Oil by Membrane Filtration[1]

This standard is issued under the fixed designation D 4807; 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.

ε[1] NOTE—Footnote 5 was updated editorially in August 2005.

1. Scope*

1.1 This test method covers the determination of sediment in crude oils by membrane filtration. This test method has been validated for crude oils with sediments up to approximately 0.15 mass %.

1.2 The accepted unit of measure for this test method is mass %, but an equation to convert to volume % is provided (see Note 6).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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 specific warning statements, see 6.1 and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API *MPMS* Chapter 10.1)

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.1)

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.2)

D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API *MPMS* Chapter 8.3)

2.2 *API Standards:*[3]

MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D 4057)

MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D 4177)

MPMS Chapter 8.3 Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D 5854)

MPMS Chapter 10.1 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (ASTM Test Method D 473)

2.3 *ISO Standard:*[4]

ISO 5272:1979 Toluene for Industrial Use—Specifications

3. Summary of Test Method

3.1 A portion of a representative crude oil sample is dissolved in hot toluene and filtered under vacuum through a 0.45-µm porosity membrane filter. The filter with residue is washed, dried, and weighed to give the final result.

4. Significance and Use

4.1 A knowledge of the sediment content of crude oil is important both in refinery operations and in crude oil commerce.

[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, the joint ASTM-API committee on Static Petroleum Measurement. This test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.

Current edition approved June 1, 2005. Published September 2005. Originally approved in 1988. Last previous edition approved in 1999 as D 4807–88(1999).

[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 (API), 1220 L St., NW, Washington DC 20005.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

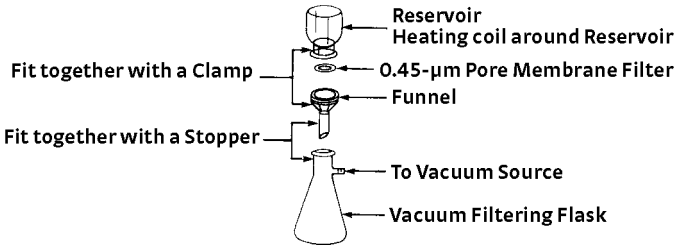

FIG. 1 Membrane Filtration Assembly

5. Apparatus

5.1 *Funnel and Filter Support Assembly*—Use an assembly designed to hold 47-mm diameter filters as was used in the development of this test method (see Fig. 1).[5]

5.1.1 *Filter Funnel*—Use a filter funnel with a 250 mL minimum capacity. The lower part of the funnel has a 40-mm inside diameter and is designed to secure the 47-mm diameter filter against the filter support. The funnel can be jacketed to facilitate heating the solvent funnel and sample during filtering.

NOTE 1—Use of a glass funnel should minimize the effect of static electricity when filtering.

5.1.2 *Filter Support*—Use a support base for the filter that has a porous scintered glass center section about 40 to 43 mm in diameter. The support base is designed to fit securely against the funnel holding the filter in place over the porous section. The filter support's stem should be long enough to extend down into the filter flask such that the end is below the vacuum connection.

5.1.3 *Clamp Assembly*—Use a spring or screw type clamp to secure the funnel to the filter support. The clamp should be tight enough to prevent the solvent from leaking through at the junction between the glass and filter membrane. The exterior dimensions of the funnel and support are designed to facilitate clamping the two pieces together.

5.1.4 *Rubber Stopper*—Use a single-hole, capable of holding the lower stem of the filter support securely onto the filtering flask.

5.1.5 *Vacuum Filtering Flask*—Use a 500 mL or larger vacuum filtering flask.

5.2 *Membrane Filter*—Use a nylon membrane filter, 47 mm in diameter with 0.45-µm pore size.[6]

5.3 *Oven*—Use an oven capable of maintaining a temperature of 105 ± 2°C (220 ± 4°F).

5.4 *Vacuum Pump*—Use a vacuum pump capable of reducing and maintaining the pressure at −80 kPa (−24 in. Hg) during the filtering.

5.5 *Analytical Balance*—Use an analytical balance capable of measuring to the nearest 0.0001 g. Verify the balance, at least annually, against weights traceable to a national metrology institute such as the National Institute of Standards and Technology (NIST).

5.6 *Heating Coil for Filter Assembly*—Use copper tubing (3.175 mm or ⅛-in. diameter) wound around the funnel on the filter apparatus and connected to a circulating bath to maintain the oil in the funnel at 90 ± 2°C (see Fig. 1). Alternative methods of heating the funnel such as heating tape or glass thermal jacket could also be used.

5.7 *Mixer*—Use a nonaerating, high-speed mixer meeting the verification efficiency requirements specified in Practice D 5854 (API *MPMS* Chapter 8.3). Either insertion mixers or circulating mixers are acceptable provided they meet the criteria in Practice D 5854 (API *MPMS* Chapter 8.3).

5.8 *Cooling Vessel*—Use a desiccator or other type of tightly covered vessel for cooling the membrane filter before weighing. The use of a desiccant/drying agent is not recommended.

5.9 *Ground/Bond Wire*—Use a 0.912–2.59 mm (No. 10 through No. 19) bare stranded flexible, stainless steel or copper wire installed in the flask through the vacuum connection and connected to ground.

6. Reagents

6.1 *Toluene*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available,[7] or to Grade 2 of ISO 5272. Other grades may be used, provided it is first ascertained that the reagent's lot or batch is of sufficiently high purity to permit its use without lessening the accuracy of the determination. (**Warning**—Flammable. Keep away from heat, sparks and open flame. Vapor harmful. Toluene is toxic. Particular care shall be taken to avoid breathing the vapors and to protect the eyes. Keep the container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with the skin.)

7. Sampling, Test Specimens

7.1 *Sampling*, shall include all the steps required to obtain a representative portion of the contents of any pipe, tank, or other system, and to transfer the sample into the laboratory test container. The laboratory test container and sample volume shall be of sufficient dimensions and volume to allow mixing as described in 7.3.1. Mixing is required to properly disperse sediment as well as any water present in the sample.

7.2 *Laboratory Sample*—Use only representative samples obtained as specified in Practice D 4057 (API *MPMS* Chapter 8.1) or Practice D 4177 (API *MPMS* Chapter 8.2) for this test method. Analyze samples within two weeks after taking the sample. Retaining samples longer may affect the results.

7.3 *Sample Preparation*—The following sample preparation and handling procedure shall apply.

7.3.1 Mix the test sample of crude oil at room temperature in the original container immediately (within 15 min) before

[5] The following filtration assembly was used in generating the precision: Millipore Corp., Ashly Rd., Bedford, MA 01730. Other filtration assemblies also may be acceptable.

[6] The following filter was used in generating the precision: MSI Nylon 60 Membrane Filter from Fisher Scientific, Catalog Number NO-4-SP047-00. Other nylon filters of 0.45-µm porosity also may be acceptable.

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

analysis to ensure complete homogeneity. A test sample drawn directly from a large volume dynamic mixing system shall be analyzed within 15 min or else remix as follows:

NOTE 2—Analysis should follow mixing as soon as possible. The 15-min interval mentioned above is a general guideline which may not apply to all crudes, especially some light crudes which do not hold water and sediment in suspension for even this short a time.

7.3.2 Mixing of the sample should not increase the temperature of the sample more than 10°C (20°F), or a loss of water may occur affecting the sample's composition. The type of mixer depends on the quantity of crude. Before any unknown mixer is used, the specifications for the homogenization test, Practice D 5854 (API *MPMS* Chapter 8.3), must be met. The mixer must be re-evaluated following any changes in the type of crude, quantity of crude, or shape of the sample container.

7.3.3 For small test sample volumes, 50 to 300 mL, a nonaerating, high-speed, shear mixer is required. Use the mixing time, mixing speed, and height above the bottom of the container found to be satisfactory in Practice D 5854 (API *MPMS* Chapter 8.3). Clean and dry the mixer between samples.

8. Procedure

8.1 *Filter Preparation*—Prepare nylon filters by heating in an oven at 105 ± 2°C (220 ± 4°F) for 15 min. Cool and store the dried filters in a cooling vessel (desiccator without desiccant) until needed. Use only new filters.

8.2 Weigh the filter immediately before use to the nearest 0.0001 g.

8.3 Using tweezers, place the membrane filter on the center of the filter support, which is mounted on the filtering flask with a rubber stopper. Attach the funnel to the filter support and clamp it securely.

8.4 Connect the heating coil to the circulating bath and place the coil around the lower part of the funnel. Set the temperature of the circulating bath so as to maintain the oil in the funnel at 90 ± 2°C (195 ± 4°F).

NOTE 3—Care should be taken not to overheat the funnel so as to cause evaporation of the toluene and glazing of the filter.

8.5 *Sample Addition*—Into a 200-mL beaker, weigh 10 g of a thoroughly mixed sample (see Section 7) to the nearest 0.0001 g. Add 100 mL of toluene to the beaker and heat the mixture with stirring to 90 ± 2°C (195 ± 4°F). Maintain the temperature at 90 ± 2°C (195 ± 4°F) for about 15 min to dissolve any wax in the crude.

8.6 Start the vacuum pump and adjust the vacuum to −80 kPa (−24 in. Hg). Carefully pour the sample mixture into the filter funnel in three portions. Generally the sample should filter in 10 to 15 min. If the nature of the crude (for example, heavy versus light gravity or high versus low viscosity) or the amount of sediment causes the filtration to proceed extremely slowly (for example, filtering times greater than 30 min), reduce the sample size to 5 g or less and repeat the test. Keep the volume of toluene at 100 mL.

NOTE 4—If the filtration of a given crude typically takes less than 10 min and the sample stays at 90 ± 2°C (195 ± 4°F) during this time, then external heating of the filter funnel may not be necessary.

8.7 *Filter Washing*—Before the last portion of sample has completely filtered, wash the funnel and filter with 50 mL of hot toluene (90°C, 195°F) until no oil is visible on the filter. With the vacuum on, leave the filter on the apparatus for 2 min.

8.8 *Apparatus Disassembly*—Disassemble the filter apparatus by removing the clamp and funnel. Inspect the condition of the filter. If the filter has been properly mounted, it may not be necessary to wash the edges after disassembly. However, if upon removing the funnel dark spots are observed around the edge of the filter further washing is necessary. With the vacuum on, use a dropper to wash the filter's edges with hot toluene (90°C, 195°F).

8.9 If the filter is completely or partially covered with black or dark brown crude oil residue after the washing step above, then discard the filter and repeat the test with a smaller sample size.

NOTE 5—Normally the color of the sediment on the filter is gray or light tan. A black or deep brown colored deposit on the filter is indicative of incomplete washing.

8.10 If the appearance of the filter is acceptable (as in 8.9) then carefully remove it and place it in an oven at 105°C (220°F) for 15 min. Cool in the cooling vessel to room temperature (5 to 10 min) and reweigh to the nearest 0.0001 g.

9. Calculation

9.1 Calculate the mass percent of sediment as follows:

$$S = \frac{m_2 - m_1}{m_s} \times 100 \quad (1)$$

where:

S = sediment content of the sample as a percentage by mass,
m_1 = mass of the filter, g,
m_2 = mass of the filter with the sediment, g, and
m_s = mass of the sample, g.

10. Report

10.1 Report results to the nearest 0.001 % as the mass percent of sediment by membrane filtration. The test report shall reference this Test Method D 4807 (API *MPMS* Chapter 10.8) as the procedure used.

NOTE 6—Since water and sediment values are commonly reported as volume percent, calculate the volume of the sediment as a percentage of the original sample. As a major portion of the sediment probably would be sand (silicon dioxide, which has a density of 2.32) and a small amount of other naturally occurring materials (with a relative density lower than that of sand), use an arbitrary density of 2.0 for the resulting sediment. Then, to obtain volume percent sediment, divide the mass percent sediment by 2.0 and multiply by the relative density of the crude oil. (Note that this calculation is provided for convenience only, and the precision and bias for this standard are based on mass percent sediment and not on volume percent of sediment.)

$$S_V = \frac{S}{2.0} \times \text{relative density of the oil} \quad (2)$$

TABLE 1 Precision Intervals

Mass Sediment	Repeatability	Reproducibility
0.0050	0.0031	0.0083
0.0100	0.0044	0.0118
0.0150	0.0054	0.0144
0.0200	0.0062	0.0166
0.0250	0.0069	0.0186
0.0300	0.0076	0.0204
0.0350	0.0082	0.0220
0.0400	0.0088	0.0235
0.0450	0.0093	0.0249
0.0500	0.0098	0.0263
0.0600	0.0107	0.0288
0.0700	0.0116	0.0311
0.0800	0.0124	0.0333
0.0900	0.0132	0.0353
0.1000	0.0139	0.0372
0.1250	0.0155	0.0416
0.1500	0.0170	0.0455

where:

S_V = the sediment content of the sample as a percentage by volume, and

S = the sediment content of the sample as a percentage by mass.

10.2 Report that the sample mixing procedure was performed in accordance with the procedures specified in Practice D 5854 (API *MPMS* Chapter 8.3). Report the temperature of the sample before and after mixing.

10.3 The test report shall also contain all details necessary for complete identification of the product tested; any deviation, by agreement or otherwise, from the procedure specified; and the date of the test.

11. Precision and Bias

11.1 *Repeatability*—The difference between successive 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 following values in only one case in twenty (see Table 1).

$$0.04388(X^{1/2}) \quad (3)$$

where:

X = sample mean in mass percent.

11.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty (see Table 1).

$$0.1176(X^{1/2}) \quad (4)$$

where:

X = sample mean in mass percent.

11.3 *Bias*—The data generated during the development of this test method showed that more sediment is reported for crude oil using this test method compared to the results of Test Method D 473 (API *MPMS* Chapter 10.1).

12. Keywords

12.1 crude oil; membrane filtration; sediment

ANNEX

(Mandatory Information)

A1. SAFETY PRECAUTIONS TO AVOID STATIC DISCHARGE

A1.1 In Guide D 4865 it is noted that micro-filters are prolific generators of electrostatic charge. This is particularly true in the case of membrane filters used in this procedure.

A1.1.1 The flow of crude oil through the membrane in performing this type of test causes charges to separate due to the presence of ionic impurities or additives in the crude oil. Charges of one polarity are carried with the moving oil while the opposite charges accumulate within the membrane and its holder. The surface charges seek a path to ground.

A1.2 The rate at which these charges recombine depends on the conductivity of the oil. Relaxation time could be of the order of 10-100 s with low conductivity oil. In membrane filtration, very little time is available for charge recombination due to high velocities through the membrane. As a consequence even high conductivity oils may cause charges to accumulate in the membrane holder and receiver and develop significant voltage differences between the oil and apparatus. Using a glass receiving flask and placing a grounding wire in the receiver will minimize the development of voltage in the fuel.

A1.3 Although grounding the apparatus will not prevent charge separation or accumulation of charges in the oil, it is necessary to bond all parts of the filtration apparatus together and provide a grounding wire. It is essential that no unbonded metal components are present during filtration since they concentrate charge and develop voltage sufficient to cause static discharge within the apparatus.

A1.4 To verify that bonding of all parts of the filtration apparatus is complete the method requires that an electrical conductivity test be conducted using a multimeter. There must be 10 Ω or less resistance between any two points.

SUMMARY OF CHANGES

Subcommittee D02.02 has identified the location of selected changes to this standard since the last issue (D 4807–88(1999)) that may impact the use of this standard.

(*1*) Corrected the requirements for the vacuum pump to −80 kPa (−24 in. Hg).

(*2*) Added the requirement to verify the analytical balance at least annually.

(*3*) Added a cooling vessel to the apparatus and time to cool back to room temperature—5 to 10 min.

(*4*) Added ground/bond wire to the apparatus to dissipate static charge.

(*5*) Added Note 6 to allow conversion of mass percent of sediment to volume percent of sediment.

(*6*) Deleted original Annex A1, Homogenization Efficiency of Unknown Mixers since this is now part of Practice D 5854 (API *MPMS* Chapter 8.3) which is referenced.

(*7*) Added Annex A1, Safety Precautions to Avoid Static Discharge.

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