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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 D4807; 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 U.S. Department of Defense.

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]
D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
2.2 *API Standards:*[3]
MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D4057)
MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)
MPMS Chapter 8.3 Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D5854)
MPMS Chapter 10.1 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (ASTM Test Method D473)
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~~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.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02 /COMQ on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API).

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[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 American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

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

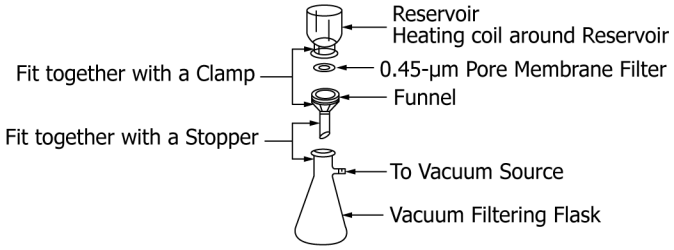

FIG. 1 Membrane Filtration Assembly

5. Apparatus

5.1 *Funnel and Filter Support Assembly*—Use an assembly designed to hold ~~47-mm~~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~~ 250 mL minimum capacity. The lower part of the funnel has a ~~40-mm~~40 mm inside diameter and is designed to secure the ~~47-mm~~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~~40 mm to ~~43 mm~~ 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~~ 500 mL or larger vacuum filtering flask.

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

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

5.4 *Vacuum Pump*—Use a vacuum pump capable of reducing and maintaining the pressure at ~~−80 kPa (−24 in.~~ −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.~~ 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~~ (3.175 mm or ⅛~~-in.~~ 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~~90 °C ± ~~2°C~~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 D5854 (API *MPMS* Chapter 8.3). Either insertion mixers or circulating mixers are acceptable provided they meet the criteria in Practice D5854 (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~~ 0.912 mm to 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

[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~~0.45 µm porosity also may be acceptable.

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 D4057 (API *MPMS* Chapter 8.1) or Practice D4177 (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)~~ 15 min) before analysis to ensure complete homogeneity. A test sample drawn directly from a large volume dynamic mixing system shall be analyzed within ~~15 min~~ 15 min or else remix as follows:

NOTE 2—Analysis should follow mixing as soon as possible. The ~~15-min~~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),~~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 D5854 (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~~50 mL to ~~300 mL,~~ 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 D5854 (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~~105 °C ± ~~2°C (220~~2 °C (220 °F ± ~~4°F)~~4 °F) for ~~15 min.~~ 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.~~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~~90 °C ± ~~2°C (195~~2 °C (195 °F ± ~~4°F).~~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~~200 mL beaker, weigh ~~10 g~~ 10 g of a thoroughly mixed sample (see Section 7) to the nearest ~~0.0001 g. Add 100 mL~~ 0.0001 g. Add 100 mL of toluene to the beaker and heat the mixture with stirring to ~~90~~90 °C ± ~~2°C (195~~2 °C (195 °F ± ~~4°F).~~4 °F). Maintain the temperature at ~~90~~90 °C ± ~~2°C (195~~2 °C (195 °F ± ~~4°F)~~4 °F) for about ~~15 min~~ 15 min to dissolve any wax in the crude.

8.6 Start the vacuum pump and adjust the vacuum to −80 kPa ~~(−24 in.~~ (−24 in. Hg). Carefully pour the sample mixture into the filter funnel in three portions. Generally the sample should filter in ~~1~~10 min to ~~15 min.~~ 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),~~ 30 min), reduce the sample size to ~~5 g~~ 5 g or less and repeat the test. Keep the volume of toluene at ~~100 mL.~~100 mL.

NOTE 4—If the filtration of a given crude typically takes less than 10 min and the sample stays at ~~90~~90 °C ± ~~2°C (195~~2 °C (195 °F ± ~~4°F)~~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~~ 50 mL of hot toluene ~~(90°C, 195°F)~~(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.~~2 min.

[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.