



Designation: D 4807 – 88 (Reapproved 1999)

An American National Standard



Designation: MPMS Chapter 10.8

Standard Test Method for Sediment in Crude Oil by Membrane Filtration¹

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 test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.

This standard has been approved for use by agencies of the 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 about 0.15 mass %.

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.*

1.3 The accepted unit of measure for this test method is mass %.

2. Referenced Documents

2.1 ASTM Standards:

D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method²

D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

D 4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration³

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.

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.02 on Static Petroleum Measurement.

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

4. Significance and Use

4.1 A knowledge of the sediment content of crude oil is important both in the operation of the refinery and in the buying and selling of crude oils.

5. Apparatus

5.1 *Reservoir and Filter Support Assembly*—an assembly designed to hold 47-mm diameter filters was used in the development of this test method (see Fig. 1).⁴

5.1.1 *Filter Reservoir*—capable of holding from about 250 mL of solvent. The lower part of the reservoir has a 40-mm inside diameter and is designed to secure the 47-mm diameter filter against the filter support. The reservoir can be jacketed to facilitate heating the reservoir and sample during filtering.

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

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

5.1.3 *Clamp Assembly*—spring or screw type clamp to secure the reservoir 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 reservoir and support/funnel are designed to facilitate clamping the two pieces together.

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

5.1.5 *Vacuum Filtering Flask*, 500 mL or larger.

⁴ A filtration assembly as supplied by Millipore Corp., Ashly Rd., Bedford, MA 01730, was found acceptable.

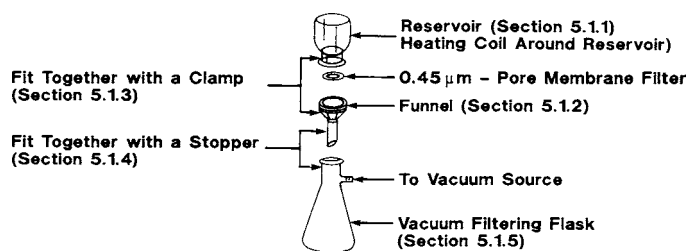


FIG. 1 Membrane Filtration Assembly

5.2 *Membrane Filter*, nylon membrane filter, 47 mm in diameter with 0.45- μm pore size.⁵

5.3 *Oven*, capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$.

5.4 *Vacuum Pump*, capable of reducing and maintaining the pressure at 200 mm Hg (-80 KPA) during the filtering.

5.5 *Analytical Balance*, capable of measuring to the nearest 0.0001 g.

5.6 *Heating Coil for Filter Assembly*—copper tubing ($1/8$ -in. diameter) wound around the reservoir on the filter apparatus and connected to a circulating bath to maintain the oil in the reservoir at $90 \pm 2^\circ\text{C}$ (see Fig. 1). Alternative methods of heating the reservoir such as heating tape or glass thermal jacket could also be used.

5.7 *Mixer*⁶—a nonaerating, high-speed mixer capable of meeting the homogenization efficiency test in Annex A1. Large volume dynamic mixing systems such as those used with automatic crude oil sampling receptacles are also acceptable providing they comply with Annex A1.

6. Reagents

6.1 *Purity of Reagents*—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 Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Toluene* (**Warning**, Flammable).

NOTE 2—**Warning**: Flammable.

7. Sampling, Test Specimens

7.1 *Sampling*, is defined as all the steps required to obtain an aliquot of the contents of any pipe, tank, or other system, and

⁵ 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.

⁶ The following mixers have been found satisfactory for samples under 300 mL: Ultra Turrax Model TP 18/10, available from Tekmar Co., Cincinnati, OH; Brinkman Polytron Model PT 35, available from Brinkman Instruments, Inc., Westbury, NY; and Kraft Apparatus Model S-25, available from SGA, Bloomfield, NJ.

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.

to place 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 or D 4177 for this test method. Analyze samples within two weeks after taking the sample. Retaining samples longer may affect the results.

7.3 *Test Samples*—The following sample 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 analysis to ensure complete homogeneity. A test sample drawn directly from a large volume dynamic mixing system analyze within 15 min or else remix as follows:

NOTE 3—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 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 , or a loss of water may occur and this affects sample composition. The type of mixer depends on the quantity of crude. Before any unknown mixer is used, the specifications for the homogenization test, Annex A, 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 Annex A1. Clean and dry the mixer between samples.

8. Procedure

8.1 Prepare nylon filters by heating in an oven at 105°C for 15 min. Cool and store the dried filters in a 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 reservoir 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 reservoir. Set the temperature of the circulating bath so as to maintain the oil in the reservoir at $90 \pm 2^\circ\text{C}$.

NOTE 4—Care should be taken not to overheat the reservoir so as to cause evaporation of the toluene and glazing of the filter.

8.5 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 $90 \pm 2^\circ\text{C}$. Maintain the temperature at $90 \pm 2^\circ\text{C}$ for about 15 min to dissolve any wax in the crude.

8.6 Start the vacuum pump and adjust the vacuum to 200 mm Hg. Carefully pour the sample mixture into the filter reservoir in three portions. Generally the sample should filter in