



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)³

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.

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.

5.2 *Membrane Filter*, nylon membrane filter, 47 mm in

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.

Current edition approved March 25, 1988. Published May 1988.

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

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

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

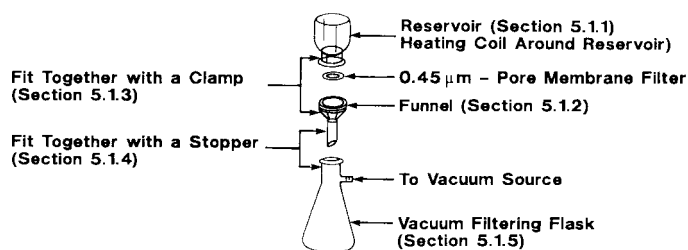


FIG. 1 Membrane Filtration Assembly

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 ($\frac{1}{8}$ -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 to place the sample into the laboratory test container. The

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.

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 10 to 15 min. If the nature of the crude 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 5—If the filtration of a given crude typically takes less than 10 min and the sample stays at $90 \pm 2^\circ\text{C}$ during this time, then external heating of the filter reservoir may not be necessary.

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

8.8 Disassemble the filter apparatus by removing the clamp and reservoir. 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 reservoir 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).

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 6—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 for 15 min. Cool in a desiccator and reweigh to the nearest 0.0001 g.

9. Calculation

9.1 Calculate the mass percent of sediment as follows:

$$\text{Mass \%} = \frac{G}{S} \times 100 \quad (1)$$

where:

G = gain in mass of filter, g, and

S = sample mass, 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 as the procedure used.

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 test method, exceed the following values in only one case in twenty (see Table 1).

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

where X is sample mean

11.2 *Reproducibility*—The difference between two single and independent results obtained by different operations 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).

$$0.1176 (X^{1/2}) \quad (3)$$

where X is sample mean

11.3 *Bias*—The data generated during the development of this test method showed that more sediment is recovered from crude oil using this test method compared to the results of Test Method D 473.

12. Keywords

12.1 apparatus; crude oil; membrane filtration; procedure; reagents; sampling; sediment

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

ANNEX**(Mandatory Information)****A1. HOMOGENIZATION EFFICIENCY OF UNKNOWN MIXERS**

A1.1 The homogenization efficiency of each unknown mixer shall be evaluated before use. The grade of crude oil, the sample size, and the sample container expected to be used with the mixer should be used in this test. The specifications of this test should be met before running this test method and any changes in the mixing procedure should be re-evaluated by this test. The mixing efficiency is measured by following the water content as described in Test Method D 4377. The crude oil used in this test should be dry (less than 0.1 % water), otherwise perform a preliminary distillation to remove the water as described in Test Method D 2892.

A1.2 Weigh the sample container to the nearest 0.01 g. Fill the container halfway (or the level normally used) with the dry crude. Immerse the mixer into the crude with the bottom of the mixer 5 mm above the bottom of the container and mix the crude at the speed and for the amount of time you expect to use. Suggested mixing time is between 1 and 5 min at 5 to 7000 r/min. Immediately determine the water content in duplicate (see Test Method D 4377) of the dry crude. Obtain the average of the duplicate results.

A1.3 Weigh the crude and container to the nearest 0.01 g. Immerse the mixer into the crude as in A1.2. Knowing the weight of the crude, add enough water to increase the water

content 1 % above the base level found in A1.2. From a water-filled syringe previously weighed (nearest 0.1 mg), inject the water below the surface of the crude near the inlet to the mixer. Reweigh the syringe (to the nearest 0.1 mg) and determine the amount of water added. Any oil on the needle should be wiped off before weighing. Mix the sample in the same manner as in A1.2. Determine the water content of the crude immediately after mixing. Sample the crude just below the liquid level.

A1.4 Without additionally mixing the crude, determine the water content of the crude 15 and 30 min after the initial mixing in A1.3.

A1.5 Remix the sample in the same manner as A1.2. Immediately after mixing, determine the water content in duplicate.

A1.6 The water contents of the crude determined in A1.3, A1.4, and A1.5 minus the base determined in A1.2 should agree within 0.05 % absolute of the added water and to each other. If they do not agree, this test should be repeated while changing the mixing time, the mixing speed, or the height of the mixer in the crude, or a combination thereof until these conditions are met.

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