



Designation: D5439 – 95 (Reapproved 2012)

## Standard Test Method for Determination of Sediment in Moellon<sup>1</sup>

This standard is issued under the fixed designation D5439; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the toluene insoluble sediment contained in Moellon. This test method was derived from Test Method D473.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

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

2.2 *ISO Standard:*<sup>3</sup>

ISO 5272 Toluene for Industrial Use - Specifications

### 3. Significance and Use

3.1 This test method is intended for use in determining the toluene insoluble sediment contained in Moellon.

### 4. Apparatus

4.1 *Extraction Apparatus* (see Fig. 1 and Fig. 2), consisting of the parts described in 4.1.1 – 4.1.6.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 23–1957).

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<sup>2</sup> 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.

<sup>3</sup> Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

4.1.1 *Extraction Flask*, a wide-neck Erlenmeyer flask of 1-L capacity.

4.1.2 *Condenser*, a condenser in the form of a metal coil approximately 25 mm in diameter and 50 mm in length attached to, and with the ends projecting through a lid of sufficient diameter to cover the neck of the flask as shown in Fig. 1. The coil should be made from stainless steel, tin, tin-plated copper, or tin-plated brass tubing having an outside diameter of 5 to 8 mm and a wall thickness of 1.5 mm. If constructed of tin-plated copper or brass, the tin coating shall have a minimum thickness of 0.075 mm. The exposed surface of the coil for cooling purposes is about 115 cm<sup>2</sup>.

4.1.3 *Extraction Thimble*—The extraction thimble should be of a refractory porous material, pore size index P15, 25 mm in diameter by 70 mm in height, weighing not less than 15 g and not more than 17 g. The thimble shall be suspended from the condenser coil by means of a basket so that it hangs approximately midway between the surface of the extracting solvent and the bottom of the condenser coil.

4.1.4 *Thimble Basket*—The thimble basket shall be corrosion-resistant, shall be made of platinum, stainless steel, nickel-chromium alloy, or similar material, and shall meet the requirements of Fig. 2.

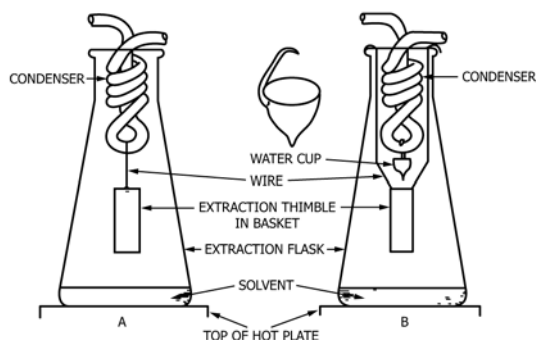
4.1.5 *Water Cup*—A water cup shall be used when testing a sample having a high-water content (see Fig. 1b). The cup shall be made of glass, shall be conical in shape, shall be approximately 20 mm in diameter and 25 mm deep, and shall have a capacity of approximately 3 mL. A glass hook fused on the rim at one side is so shaped that when hung on the condenser the cup hangs with its rim reasonably level. In this procedure, the thimble basket is suspended either as shown in Fig. 1a by means of the corrosion-resistant wire looped over the bottom of the condenser coil and attached to the basket supports or as in Fig. 1b where the wire supports of the basket are attached to hooks soldered to the underside of the condenser lid.

4.1.6 *Source of Heat*, a source of heat, preferably a hot plate, suitable for vaporizing toluene.

### 5. Solvent

5.1 *Toluene*, conforming to ISO 5272, Grade 2. (**Warning**—Flammable.)

5.1.1 The typical characteristics for the reagent are:



NOTE 1—Apparatus B shows the water cup in position.

FIG. 1 Extraction Apparatus for Determination of Sediment

Color (APHA)	10
Boiling range (initial to dry point) <sup>A</sup>	2.0°C
Residue after evaporation	0.001 %
Substances darkened by H <sub>2</sub> SO <sub>4</sub>	passes ACS test
Sulfur compounds (as S)	0.003 %
Water (H <sub>2</sub> O) (by Karl Fischer titration)	0.03 %

<sup>A</sup> Recorded boiling point 110.6°C.

## 6. Sampling

6.1 Sampling is defined as all 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.

6.2 Only representative samples obtained as specified in Practices D4057 and D4177 shall be used for this test method.

## 7. Procedure

7.1 For referee tests, use a new extraction thimble. For routine tests, thimbles may be reused. Before reusing a thimble, it must be heated to a dull red heat (preferably in an electric furnace) to remove the combustible portion of the accumulated sediment. Subject the thimble to a preliminary extraction as described in 7.2 before being used for another determination.

7.2 Before using a new thimble, rub the outside surface with fine sandpaper and remove all loosened material with a stiff brush. Give the thimble a preliminary extraction with the toluene, allowing the solvent to drip from the thimble for at least 1 h. Then dry the thimble for 1 h at a temperature of 115 to 120°C; cool in a desiccator, without desiccant, for 1 h, and weigh to the nearest 0.1 mg. Repeat this extraction until the masses of the thimble, after two successive extractions, do not differ by more than 0.2 mg.

7.3 Place an estimated 10-g test portion of the sample in the thimble immediately after the sample has been mixed as described in Practices D4057 and D4177. Do not attempt to adjust this estimated 10-g portion to any exact predetermined amount. Weigh to the nearest 0.01 g. Place the thimble in the extraction apparatus, and extract with the hot toluene for 30 min after the solvent dripping from the thimble is colorless. Ensure that the rate of extraction is such that the surface of the mixture of oil and toluene in the thimble does not rise higher than to within 20 mm of the top.

7.4 When testing samples having a high water content, use the assembly shown in Fig. 1b. In this procedure, any water in the test portion is removed as its toluene azeotrope and is collected in the water cup, where it separates as a bottom layer. The toluene layer overflows into the thimble. If the cup becomes full of water, allow the apparatus to cool and empty the cup.

7.5 After the extraction is completed, dry the thimble for 1 h at 115 to 120°C; cool in a desiccator, without desiccant, for 1 h and weigh to the nearest 0.2 mg.

7.6 Repeat the extraction, allowing the solvent to drip from the thimble for at least 1 h but not longer than 1.25 h; dry, cool, and weigh the thimble as described in 7.5. Repeat this extraction for further 1-h periods, if necessary, until the masses of the dried thimble plus sediment, after two successive extractions, do not differ by more than 0.2 mg.

## 8. Calculation

8.1 Calculate the mass of the sediment as a percent of that of the original sample as follows:

$$\text{Mass \%} = \frac{MS(9.6)}{OSM(9.3)} \times 100 \quad (1)$$

where:

*M* = mass,  
*S* = sediment, and  
*O* = original sample mass.

## 9. Report

9.1 Report the results to the nearest 0.01 % as the mass percent of sediment by extraction (Note 1). The test report shall reference this test method as the procedure used.

NOTE 1—Since water and sediment values are commonly reported as volume percent, calculate the volume of the sediment as a percent of the original sample. As a major portion of the sediment probably would be sand (silicon dioxide, which has a specific gravity of 2.32) and a small amount of other naturally occurring materials (with a specific gravity lower than that of sand), use an arbitrary specific gravity of 2.0 for the resulting sediment. Then, to obtain volume percent sediment, divide the mass percent sediment multiplied by the specific gravity of the crude at 15°C (use 0.85 specific gravity, if unknown) by 2.

$$\text{Volume \%} = (MPS/2.0) \times [CSP \text{ or } 0.85 \text{ if unknown}] \quad (2)$$

where:

*M* = mass,  
*P* = percent,  
*S* = sediment, and  
*CSP* = crude specific gravity.

## 10. Precision

10.1 The precision of this test method, as based on mass percent and examination of interlaboratory test results in the range from 0 to 0.4 % is described in 10.1.1 and 10.1.2.

10.1.1 *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 value in only one case in twenty: