



Standard Test Method for Determination of Sediment in Moellon¹

This standard is issued under the fixed designation D 5439; 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.

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 D 473.

1.2 The values stated in SI units are to be regarded as the 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:

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

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

2.2 ISO Standard:

5272 Toluene-Specifications⁴

3. Significance and Use

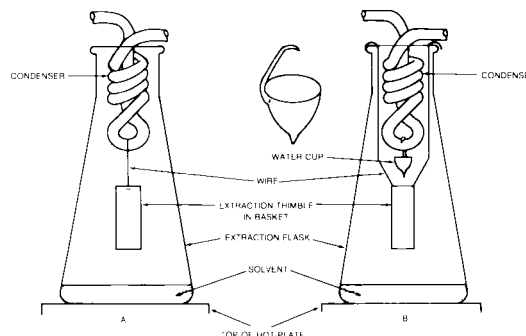
3.1 This test method is intended for use in determining the toluene insoluble sediment contained in Moellon.

4. Apparatus

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

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



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

FIG. 1 Extraction Apparatus for Determination of Sediment

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 cm².

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.

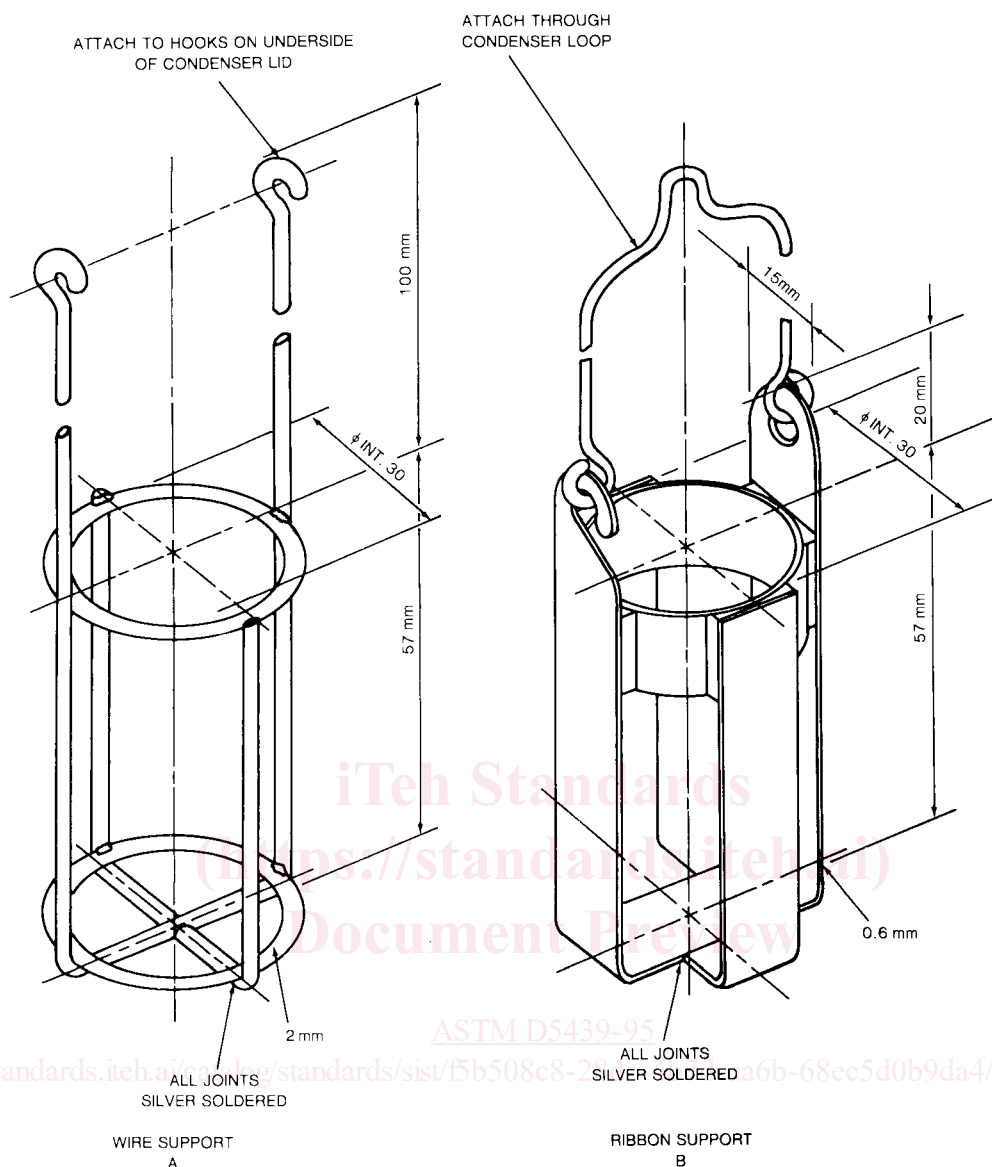
¹ This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 23-1957).

Current edition approved May 15, 1995. Published July 1995. Originally published as D 5439 – 93. Last previous edition D 5439 – 93.

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

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

⁴ Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.



NOTE 1—Silver solder should be used on all joints.

FIG. 2 Basket Thimble Support

5. Solvent

5.1 Toluene, conforming to ISO 5257, Grade 2.

NOTE 1—**Warning:** Flammable.

5.1.1 The typical characteristics for the reagent are:

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

[^] 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 D 4057 and D 4177 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