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Standard Test Method for Pentane Insolubles by Membrane Filtration¹

This standard is issued under the fixed designation D 4055; 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 pentane insolubles for particles exceeding 0.8 μm in new and used lubricating oils.

NOTE 1—Pentane insolubles with particle sizes less than ~~0.8 μm~~ 0.8 μm may be studied with appropriate size membrane filters. Particle sizes above or below 0.8 μm can be studied. The precision of this test method has been determined only at 0.8 μm .

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.* For specific ~~hazards~~ warning statements, see 7.1, 8.2.1, and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*²

D 4057 [Practice for Manual Sampling of Petroleum and Petroleum Products](#)

D 4175 [Terminology Relating to Petroleum, Petroleum Products, and Lubricants](#)

D 4177 [Practice for Automatic Sampling of Petroleum and Petroleum Products](#)

3. Terminology

3.1 *Definitions:*

3.1.1 *pentane insolubles, in used oil analysis, n*—separated matter resulting when a used oil is dissolved in pentane.

3.1.1.1 *Discussion*—In this test method, the separation is effected by filtration through a membrane.

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¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

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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* Vol 05.02, volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

3.1.2 *used oil, n*—any oil that has been in a piece of equipment (for example, an engine, gearbox, transformer, or turbine) whether operated or not. **D 4175**

4. Summary of Test Method

4.1 A sample of new or used lubricating oil is mixed with pentane in a volumetric flask. The oil solution is filtered through an 0.8- μ m membrane filter. The flask, funnel, and filter are washed with additional pentane to effect a complete transfer of particulates onto the filter. The filter and its particulates are dried and weighed to give the pentane insolubles.

5. Significance and Use

5.1 Pentane insolubles above 0.8 μ m in size may lead to increased wear. This increased wear can lead to premature equipment failure in critical applications.

6. Apparatus

6.1 In the development of this test method, it was noted that variations, particularly with respect to glassware and filter media, can affect the test result significantly.

6.2 When the user of this test method uses an alternate membrane filter, it is incumbent upon them to establish that the alternate filter will give equal results.

6.3 Precision data were established using the apparatus listed and a 0.8 μ m filter pore size.

6.4 *Membrane Filter*.³

6.5 *Borosilicate Filter Holder*.⁴

6.6 *Borosilicate Filtering Flask*.⁵

6.7 *Forceps*⁶ (~~plain flat tips—not serrated~~), (plain flat tips, not serrated).

6.8 *Wash Bottle*⁷ equipped with 0.8- μ m membranes.

6.9 *Analytical Balance*, capable of weighing with an accuracy of ± 0.1 mg.

6.10 *Vacuum Source*, capable of maintaining a vacuum of 255 ± 50 mm Hg.

6.11 *Oven*, capable of maintaining $90 \pm 5^\circ\text{C}$.

6.12 *Oven Thermometer*—~~ASTM Thermometer 1C~~, ASTM Thermometer 1C.

6.13 *Aluminum Foil Weighing Dish*, about 60 mm in diameter.

6.14 *Desiccator*, with calcium chloride.

6.15 *Mechanical Paint Shaker*, three-dimensional.⁸

6.16 *Volumetric Flask*, 100-mL with ground-glass or TFE-fluorocarbon stopper.

6.17 *Ionizing Unit*⁹—~~Insert, insert~~ under or next to balance pan.

7. Reagent

7.1 Pentane, reagent grade or better. (**Warning**—Extremely flammable. Vapors can cause flash fire. See A1.1.)

8. Procedure

8.1 Using either Practice D 4057 (manual sampling) or Practice D 4177 (automatic sampling), obtain a representative sample of the material to be tested.

8.1.1 *Preparation of Sample and Materials* :

8.1.2 Warm the oil in the original container, if possible, to $60 \pm 5^\circ\text{C}$. Shake for a minimum of 30 min on the mechanical shaker to resuspend the insolubles uniformly.

NOTE 2—Deviation from this sample preparation will normally result in poor precision. Correct sample size is particularly important.

~~8.1.3 Weigh~~ 8.1.3 **Weigh** 1 g of a representative sample of the oil into a 100-mL volumetric flask to the nearest 0.01 g.

8.1.3.1 Certain oils may require smaller size samples to prevent plugging the membrane. When the 100 mL of the mixture has not passed through the membrane after 2 min, discard the test and repeat with a smaller sample size ($\pm 10\%$) in the order listed:

0.80	0.33	0.13	0.05
0.64	0.26	0.11	0.04
0.51	0.21	0.09	0.03
0.41	0.17	0.07	0.02

8.1.4 Filter pentane through an 0.8- μ m filter.

³ Millipore AAWP04700, available from Millipore Corp, Bedford MA, 01273, has been found to be satisfactory; also an equivalent may be used.

⁴ Millipore XX1004700, available from Millipore Corp, Bedford MA, 01273, has been found to be satisfactory; also an equivalent may be used.

⁵ Millipore XX1004705, available from Millipore Corp, Bedford MA, 01273, has been found to be satisfactory; also an equivalent may be used.

⁶ Millipore XX6200006, available from Millipore Corp, Bedford MA, 01273, has been found to be satisfactory; also an equivalent may be used.

⁷ Millipore XX6602500, available from Millipore Corp, Bedford MA, 01273, has been found to be satisfactory; also an equivalent may be used.

⁸ Red Devil 5400 or an equivalent is preferred.

⁹ Staticmaster 24500 has been found to be satisfactory; also an equivalent may be used.