Designation: D 4055 – 02

An American National Standard

Standard Test Method for Pentane Insolubles by Membrane Filtration[1]

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[2]

D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants[2]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[2]

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 method, the separation is effected by filtration through a membrane.

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-µm 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 µm 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 µm 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),

6.8 *Wash Bottle*[7] equipped with 0.8-µm 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 ± 5°C.

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

[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.

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

6.12 *Oven Thermometer*—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 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 ± 5°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 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 (±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-µm filter.

8.2 *Preparation of Membrane*:

8.2.1 Wash the membrane with 100 mL of filtered pentane (**Warning**—See 7.1) and place the membrane right side up (8.2.1.1) into individual aluminum weighing dish.

8.2.1.1 *Important*—The top side of the membrane as removed from the manufacturer's container shall be similarly orientated for the duration of the procedure. If the filter is reversed, the results are invalid.

8.2.2 Place the dish into the oven and dry for a minimum of 30 min at 90 ± 5°C. At the end of the drying period, remove the dish from the oven and cool for a minimum of 30 min in a desiccator containing calcium chloride or some other material that will assure an atmosphere free of moisture and hydrocarbon vapors.

8.2.3 When the membranes have reached equilibrium (about 30 min), weigh the membrane to the nearest 0.1 mg.

8.2.4 Store the aluminum dish in the desiccator until ready for use.

8.3 *Filtration*:

8.3.1 Remove the membrane filter from the weighing dish with the flat forceps and center in the filtering apparatus. Assemble the funnel and clamp.

8.3.2 Fill the volumetric flask containing the sample to the 100-mL mark with filtered pentane. Stopper and shake well. *Immediately*, filter the mixture using a vacuum of 255 ± 50 mm Hg (8.1.3.1). Rinse the flask and stopper twice with small amounts of filtered pentane, pouring the washings down the sides of the filter funnel. Rinse the walls of the filter funnel with a gentle stream of pentane dispensed from the wash bottle.

8.3.3 Carefully, remove the clamp and funnel. Wash any adhering insolubles from the funnel onto the membrane, using the wash bottle with pentane. Wash the membrane *gently*, particularly the edges, with pentane from the wash bottle. If any sample fails to remain on the membrane the test must be repeated.

8.3.4 Release the vacuum and carefully remove the membrane with the forceps. Place in the same aluminum weighing dish originally used and dry in the oven and cool in the desiccator as in 8.2.2. Weigh the membrane to the nearest 0.1 mg.

NOTE 3—The use of a semimicro balance for particulates weighing less than 0.0010 g is strongly recommended.

9. Calculation

9.1 Calculate the pentane insolubles content of the oil by membrane filtration as follows:

$$\text{Pentane insolubles, \%} = 100(M_2 - M_1)/S \quad (1)$$

where:

S = mass of sample, g,
M_1 = initial mass of membrane, g, and
M_2 = final mass of membrane and insolubles, g.

10. Report

10.1 Report the percentages of pentane insolubles by membrane filtration as:

pentane insolubles (0.8 µm) percent weight

10.2 Some new oil chemistry will require determination of a baseline value for the new oil (that is, the blank determination may not be zero).

11. Precision and Bias[10]

11.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

11.1.1 *Repeatability*—The difference between two 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 values only in one case in twenty:

$$0.177 \times 2 / 3\,x = \text{average of two results} \quad (2)$$

[8] Red Devil 5400 or an equivalent is preferred.

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

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1209.

11.1.2 *Reproducibility*—The interlaboratory precision (reproducibility) of this test is very poor, and this method of measuring pentane insolubles is unsuitable for the purpose of comparison of interlaboratory results. Additional cooperative studies to improve the precision are planned.

$$0.759 \times 2 / 3\,x = \text{average of two results} \quad (3)$$

11.2 *Bias*—A statement of bias is not applicable since a standard reference material for this property is not available.

12. Keywords

12.1 lubricating oils; membrane filtration; pentane insolubles

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Pentane

Warning—Extremely flammable liquid!
Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.

Avoid buildup of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical devices and heaters.

Avoid prolonged breathing of vapor or spray mist.

Avoid prolonged or repeated skin contact.

SUMMARY OF CHANGES

Subcommittee D02.06 has identified the location of selected changes to this standard since the last issue (D 4055–01) that may impact the use of this standard.

(1) Rewrote 8.1.3 for clarity.

(2) Rewrote 8.1.3.1 to clarify a procedural step.