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An American National Standard

Designation: IP 143/01

Standard Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products[1, 2]

This standard is issued under the fixed designation D 6560; 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 a procedure for the determination of the heptane insoluble asphaltene content of gas oil, diesel fuel, residual fuel oils, lubricating oil, bitumen, and crude petroleum that has been topped to an oil temperature of 260°C (see A1.2.1.1).

1.2 The precision is applicable to values between 0.50 and 30.0 % m/m. Values outside this range may still be valid but may not give the same precision values.

1.3 Oils containing additives may give erroneous results.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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 86 Test Method for Distillation of Petroleum Products[3]

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[3]

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[4]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[4]

2.2 *IP Standard:*[5]

Specifications for IP Standard Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *asphaltenes*, *n*—wax-free organic material insoluble in heptane, but soluble in hot toluene (benzene).

NOTE 1—Benzene is included in this definition solely on the basis of its classical references in the definition of asphaltenes. The precision of this test method when using toluene has been found to be the same as when using benzene.

4. Summary of Test Method

4.1 A test portion of the sample is mixed with heptane and the mixture heated under reflux, and the precipitated asphaltenes, waxy substances, and inorganic material are collected on a filter paper. The waxy substances are removed by washing with hot heptane in an extractor.

4.2 After removal of the waxy substances, the asphaltenes are separated from the inorganic material by dissolution in hot toluene, the extraction solvent is evaporated, and the asphaltenes weighed.

5. Significance and Use

5.1 Asphaltenes are the organic molecules of highest molecular mass and carbon-hydrogen ratio normally occurring in crude petroleum and petroleum products containing residual material. They may give problems during storage and handling if the suspension of asphaltene molecules is disturbed through excess stress or incompatibility. They are also the last molecules in a product to combust completely, and thus may be one indicator of black smoke propensity. Their composition normally includes a disproportionately high quantity of the sulfur, nitrogen, and metals present in the crude petroleum or petroleum product.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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[2] This standard is based on material published in the IP *Standard methods for Analysis and Testing of Petroleum and Related Products and British Standard 2000 Parts*, copyright The Institute of Petroleum, 61 New Cavendish Street, London, W1M 8AR. Adapted with permission of The Institute of Petroleum.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

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

[5] Available from Institute of Petroleum, 61 New Cavendish St., London, WIM 8AR, UK.

6. Apparatus

6.1 *General*—Ground-glass joints from different sources may have one of two diameter to length ratios. For the purposes of this test method, either is suitable, and for some applications, the diameter itself can be one of two . However, it is critical that the male and female parts of each joint are from the same series to avoid recession or protuberance.

6.2 *Condenser*, with a coil or double surface, fitted with a 34/45 or 34/35 ground-glass joint at the bottom to fit the top of the extractor. Minimum length is 300 mm.

6.3 *Reflux Extractor*, conforming to the dimensions given in Fig. 1. Tolerances are ± 1 mm on the height and outer diameter (OD) of the extractor body and ± 0.5 mm on all other dimensions. The female ground-glass joint at the top shall match the male at the bottom of the condenser, and the male ground-glass joint at the bottom shall match the female of the conical flask.

6.4 *Conical Flasks*, of borosilicate glass of appropriate capacity (see 11.2 and Table 1), with ground-glass joints to fit the bottom of the extractor.

NOTE 2—Sizes 24/39, 24/29, 29/43 or 29/32 are suitable.

6.5 *Stopper*, of borosilicate glass of a size to fit the conical flask.

6.6 *Evaporating Vessel*, of borosilicate glass. Either a hemispherical dish of approximately 90 mm diameter, or another suitable vessel used in conjunction with a rotovapor.

NOTE 3—A rotovapor in conjunction with a nitrogen atmosphere reduces the hazard of toluene evaporation (see 11.7).

6.7 *Filter Funnel*, of borosilicate glass, approximately 100 mm diameter.

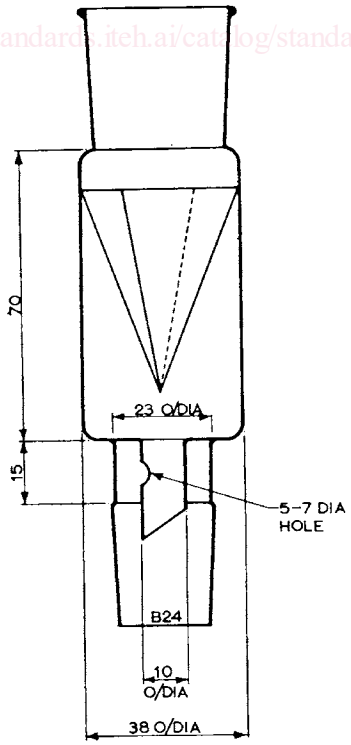

FIG. 1 Extractor

TABLE 1 Test Portion Size, Flask, and Heptane Volumes

Estimated Asphaltene Content % m/m	Test Portion Size g	Flask Volume mL	Heptane Volume mL
Less than 0.5	10 ± 2	1000	300 ± 60
0.5 to 2.0	8 ± 2	500	240 ± 60
Over 2.0 to 5.0	4 ± 1	250	120 ± 30
Over 5.0 to 10.0	2 ± 1	150	60 ± 15
Over 10.0 to 25.0	0.8 ± 0.2	100	25 to 30
Over 25.0	0.5 ± 0.2	100	25 ± 1

6.8 *Filter Papers*, Whatman[6] Grade 42, 110 or 125-mm diameter.

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

6.10 *Forceps*, of stainless steel, spade ended.

6.11 *Timing Device*, electronic or manual, accurate to 1.0 s.

6.12 *Oven*, capable of maintaining a temperature from 100 to 110°C.

6.13 *Graduated Cylinders*, of 50 and 100 mL capacity.

6.14 *Stirring Rods*, of glass or polytetrafluoroethylene (PTFE), 150 by 3-mm diameter.

6.15 *Cooling Vessel*, consisting of either a dessicator without desiccant, or another suitable tightly-stoppered vessel.

6.16 *Mixer*, high-speed, nonaerating.

7. Reagents

7.1 *Toluene (methylbenzene)* [$C_6H_5CH_3$], analytical reagent or nitration grade.

7.2 *Heptane* [C_7H_{16}], analytical reagent grade.

8. Sampling

8.1 Unless otherwise specified, take samples by the procedures described in Practice D 4057 or D 4177.

9. Test Portion Preparation

9.1 Test portions from the laboratory samples shall be drawn after thorough mixing and subdivision. Heat viscous samples of residual fuels to a temperature that renders the sample liquid, but not above 80°C, and homogenize, using the mixer (see 6.16) as necessary.

9.2 Heat samples of penetration grade bitumens to a temperature not exceeding 120°C, and stir well before taking an aliquot.

9.3 Samples of hard bitumens shall be ground to a powder before an aliquot is taken.

9.4 Samples of crude petroleum shall be prepared in accordance with the procedure described in the Annex A1, unless it is known that the crude petroleum contains negligible quantities of material boiling below 80°C.

10. Apparatus Preparation

10.1 Clean all glass flasks (see 6.4) and dishes (see 6.6) by a means that matches the cleanliness obtained by the use of a strongly oxidizing agent, such as chromosulfuric acid, ammonium peroxydisulfate in concentrated sulfuric acid at approximately 8 g/L, or sulfuric acid itself, soaking for at least 12 h,

[6] Available from Whatman Int. Ltd., Maidstone, UK.