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Standard Test Method for *n*-Heptane Insolubles[1]

This standard is issued under the fixed designation D3279; 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.

ε^1 NOTE—Section 8.1 was editorially corrected in March 2016.

1. Scope

1.1 This test method covers determination of the mass percent of asphaltenes as defined by insolubility in normal-heptane solvent. It is applicable to all solid and semi-solid petroleum asphalts containing little or no mineral matter, to gas oils, to heavy fuel oils, and to crude petroleum that has been topped to a cut-point of 343°C or higher.

1.2 The values stated in SI units are to be regarded as the 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.* See Section 7 for a specific hazard statement.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

3. Summary of Test Method

3.1 The sample is dispersed in *n*-heptane and filtered through a glass-fiber pad. The insoluble material is washed, dried, and weighed.

4. Significance and Use

4.1 This test method is useful in quantifying the asphaltene content of petroleum asphalts, gas oils, heavy fuel oils, and crude petroleum. Asphaltene content is defined as those components not soluble in *n*-heptane.

5. Apparatus and Materials

5.1 The assembly of the dispersing apparatus is illustrated in Fig. 1 with details of the component parts as follows:

5.1.1 *Erlenmeyer Flask,* of 250-mL capacity adapted to an Allihn-type reflux condenser.

5.1.2 *Magnetic Stirrer and Magnetic-Stirrer Hot Plate*

5.1.3 *Bitumen Crucible or Gooch Crucible,* glazed inside and outside with the exception of the outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top tapering to 36 mm at the bottom and a depth of 20–30 mm.

5.1.4 *Glass Microfiber Filter Pad,* 32–34 mm in diameter fine porosity, fast flow rate, 1.5υm particle retention.

5.1.5 *Filter Flask,* heavy-wall with side tube, 500-mL capacity.

5.1.6 *Filter Tube,* 40 to 42 mm in inside diameter.

5.1.7 *Rubber Tubing,* or adapter for holding Gooch crucible on the filter tube.

NOTE 1—Other suitable assemblies permitting vacuum filtration with a crucible may be used.

5.1.8 Oven, capable of maintaining a temperature of 110 ± 5°C.

6. Reagent

6.1 *n-Heptane,* 99.0 minimum mol % (Pure Grade).

7. Hazards

7.1 *n*-Heptane has a boiling point of 98°C and a flash point of −1°C, which means that it should be handled with care. It is recommended that both the reflux dispersion and filtration steps be conducted in a ventilated hood and away from flames or other sources of heat.

8. Preparation of Crucible

8.1 Place the Gooch crucible plus one thickness filter pad in an oven at about 110 ± 5°C for 30 min, allow to cool in a desiccator for 30 ± 5 min, and then determine the mass to the nearest 0.1 mg. Designate this mass as A. Store in a desiccator until ready for use.

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[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

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

9. Sample Preparation

9.1 If the sample is not fluid, heat to any convenient temperature, but in any case not more than 100ºC above the softening point.

10. Procedure

10.1 Note Safety precautions in Section 7. Transfer into the tared 250-mL Erlenmeyer flask, the approximate amount of sample to be tested. Use 0.5 to 0.6 g for airblown asphalts, 0.7 to 0.8 g for asphalt paving binders and crude residues, and 1.0 to 1.3 g for gas oils and heavy fuel oils (Note 2). Allow the sample to cool to ambient temperature and determine the mass to the nearest 1mg. Designate this mass as B. Add *n*-heptane in the ratio 100 mL of solvent per 1 g of sample, using proportionally less or more solvent as dependent upon the sample size. Unless the asphalt is in a granular form, heat the flask gently and turn it to cause the sample to be distributed somewhat over the bottom or lower sides of the flask.

NOTE 2—Tests show a small amount of insolubles (±0.3 mass %) remain on walls of the precipitation flask despite repeated washings. When expected level of *n*–C_7 insolubles is 6 % or less, use of a tared 250-mL Erlenmeyer flask is recommended. After all possible precipitate has been washed from the flask to the filtering crucible in 10.3, include the flask with the crucible for the drying, weighing, and calculation procedures in 10.3 and 11.1.

10.2 Place the Erlenmeyer flask, containing the sample plus solvent with magnetic stirrer added, on the magnetic-stirrer hot plate and secure under the reflux condenser. With the magnetic stirrer in operation, adjust the heat for gentle refluxing for a period of 15 to 20 min when testing paving binders, fuel oils, gas oils, or crude residues. For airblown asphalts, a reflux period of 25 to 30 min is recommended. In all cases, allow the dispersed mixture to cool to room temperature for a period of 1 h.

10.3 Set up the filtering crucible plus filter pad in the suction flask and pre-wet with 5 mL of *n*-heptane (see Fig. 2). Warm the flask containing the sample plus solvent to 38 to 49°C on the hot plate and pour its contents (except for the magnetic stirrer) through the filter using a gentle vacuum. Filtration will proceed most rapidly if the supernatant liquid is filtered first with the insolubles transferred to the filter last. Police the beaker or flask while transferring the final precipitate, using either a rubber policeman or stainless steel spatula with a squared end. Wash the precipitate with three portions of *n*-heptane of about 10 mL each, first rinsing out the flask therewith. Place the crucible in the 110 ± 5ºC oven for a period of 30 min, cool in a desiccator, and determine the mass to the nearest 0.1 mg. Designate this mass as C.

11. Calculation and Report

11.1 Calculate the mass percent of normal-heptane insolubles (NHI) as the percentage by weight of the original sample as follows:

where:

A = mass of crucible and filter
B = total mass of sample.
C = mass of crucible, filter, and insoluble material

For percentages of insolubles less than 1.0, report to the nearest 0.01 %; for percentages of insolubles of 1.0 or more, report to the nearest 0.1 %.

12. Precision and Bias

12.1 Precision of the method has been determined as follows:

	Standard Deviation[A]	Acceptable Range of Two Results[A]
Single-operator	0.53 % NHI	1.51 % NHI
Multilaboratory	0.93 % NHI	2.78 % NHI

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C670. The precision is for samples covering a range from 4.0 to 29.0 % HI.

13. Keywords

13.1 asphaltenes; heptane insolubles

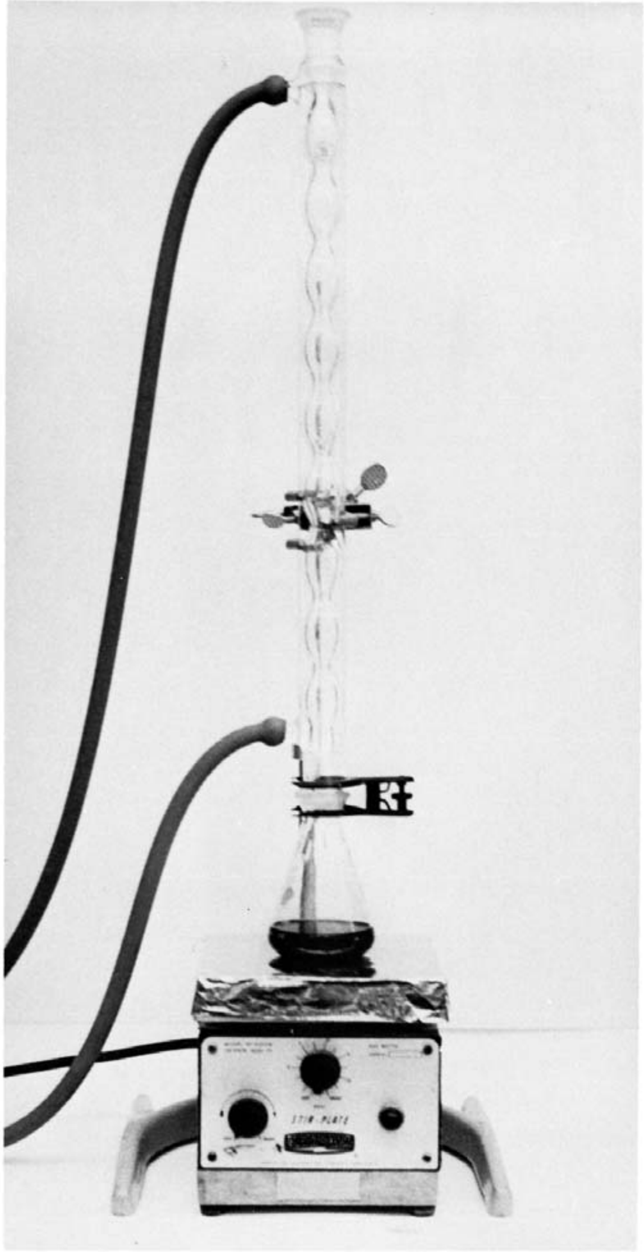

FIG. 1 Dispersing Apparatus

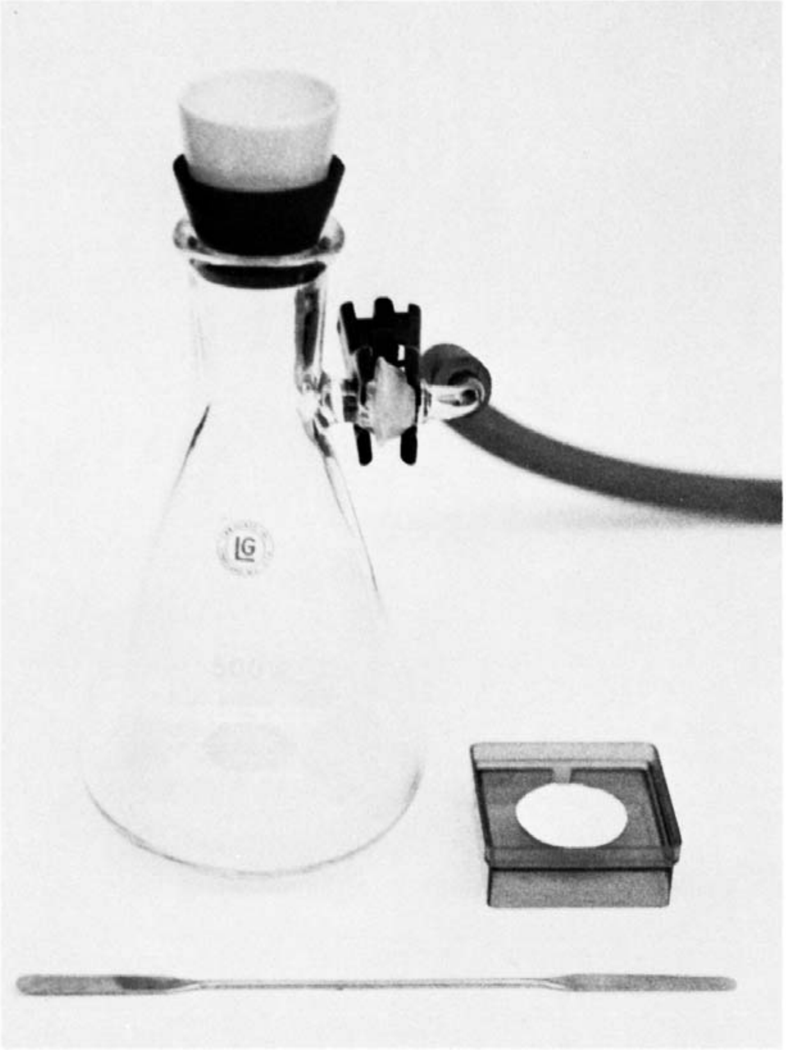

FIG. 2 Filtration Apparatus

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