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Standard Test Method for *n*-Heptane Insolubles¹

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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, each with a 35/25 ball joint.condenser.
- 5.1.2 Magnetic Stirrer and Magnetic-Stirrer Hot Plate; Plate equipped with a voltage regulator.
- 5.1.3 <u>Bitumen Crucible or Gooch Crucible</u>, 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 20–30mm.
- 5.1.4 <u>Glass Microfiber Filter Pad</u>, glass-fiber 32 <u>32</u> <u>32</u> mm in diameter.diameter fine porosity, fast flow rate, 1.5 m 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 Gooch-crucible may be used.
 - 5.1.8 Oven, capable of maintaining a temperature of 110 ± 5 °C.

6. SolventReagent

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

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.



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FIG. 1 Dispersing Apparatus

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 $110 \pm 5^{\circ}$ C for 30 min, allow to cool in a desiccator for 30 ± 5 , and then determine the mass to the nearest 0.1 mg. Designate this mass as A. Store in a desicator until ready for use.

9. Sample Preparation

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

10. Procedure

10.1 Into the Note Safety precautions in Section 7. Transfer into the tared 250-mL Erlenmeyer flask, weigh to the nearest 0.1 mg a quantity of the the approximate amount of sample to be tested, using tested. Use 0.5 to 0.6 g for airblown asphalts, 0.7 to