



Designation: D 3279 – 97 (Reapproved 2001)

## Standard Test Method for *n*-Heptane Insolubles<sup>1</sup>

This standard is issued under the fixed designation D 3279; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 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.

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:*

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

### 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.

5.1.2 *Magnetic Stirrer and Magnetic-Stirrer Hot Plate*, equipped with a voltage regulator.

5.1.3 *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 28 mm.

5.1.4 *Filter Pad*, glass-fiber 32 mm in diameter.<sup>3</sup>

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.

### 6. Solvent

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. Procedure

8.1 Into the 250-mL Erlenmeyer flask, weigh to the nearest 0.1 mg a quantity of the sample to be tested, using 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). 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 ( $\pm 0.3$  mass %) remain on walls of the precipitation flask despite repeated washings. When expected level of *n*-C<sub>7</sub> insolubles is 6 % or less, use of a tared 250-mL Erlenmeyer flask is recommended. After all possible precipitate has been

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> Glass filter pads No. 934-AH (Huribut) may be purchased from Reeve Angel and Company, Clifton, NJ.