



Designation: ~~D7280 – 06 (Reapproved 2011)~~ D7280 – 15

Standard Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch by Stainless Steel Crucible Filtration¹

This standard is issued under the fixed designation D7280; 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~~ Scope*

1.1 This test method covers the determination of the quinoline-insoluble matter (QI) in tar and pitch using a stainless steel filtering crucible and a filtration membrane.

1.2 This test method was evaluated in round robin testing using materials in the range of zero to twenty quinoline insoluble weight percent (wt%).

1.3 Since this test method is empirical, strict adherence to all details of the procedure is necessary.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*²

[D71 Test Method for Relative Density of Solid Pitch and Asphalt \(Displacement Method\)](#)

[D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation](#)

[D329 Specification for Acetone](#)

[D362 Specification for Industrial Grade Toluene \(Withdrawn 1989\)](#)³

[D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials](#)

[D2318 Test Method for Quinoline-Insoluble \(QI\) Content of Tar and Pitch](#)

[D4296 Practice for Sampling Pitch](#)

[E1 Specification for ASTM Liquid-in-Glass Thermometers](#)

[E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

3. Summary of Test Method

3.1 The sample is digested in hot quinoline and filtered. The insoluble material is washed, dried, and weighed.

4. Significance and Use

4.1 This test method is useful in evaluating and characterizing tar and pitch. It is also useful as one element in establishing the uniformity of shipments and sources of supply.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

Current edition approved May 1, 2014 Dec. 1, 2015. Published May 2014 December 2015. Originally approved in 2006. Last previous edition approved in 2006 2011 as D7280 – 06 (2011).^{2,1} DOI: 10.1520/D7280-06R11-10.1520/D7280-15.

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

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

5. Apparatus

5.1 *Filtering Crucibles*, stainless steel, with ~~fine-screen~~fine-screen bottom, ~~25~~25 mL to ~~40~~40 mL capacity, high-form.⁴ See Fig. 1.

5.2 *Filter Membrane*, PTFE membrane without backing, ~~10- μ m~~10 μ m porosity, ~~25-mm~~25 mm disk.⁵

5.3 *Filter Apparatus*, ~~500-mL~~500 mL filter flask and tube with crucible adapter.

5.4 *Sieves*, U.S. Standard ~~600- μ m~~600 μ m (No. 30) and ~~250- μ m~~250 μ m (No. 60), conforming to Specification E11.

⁴ The sole source of supply of the crucibles known to the committee at this time is Drabik Tool & Die, 15601 Commerce Park Dr., Cleveland, OH 44142. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ The sole source of supply of the filter membranes known to the committee at this time is Mitex brand, Part No. LCWP 02500, available from Millipore Corporation, 290 Concord Road Billerica, MA 01821. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

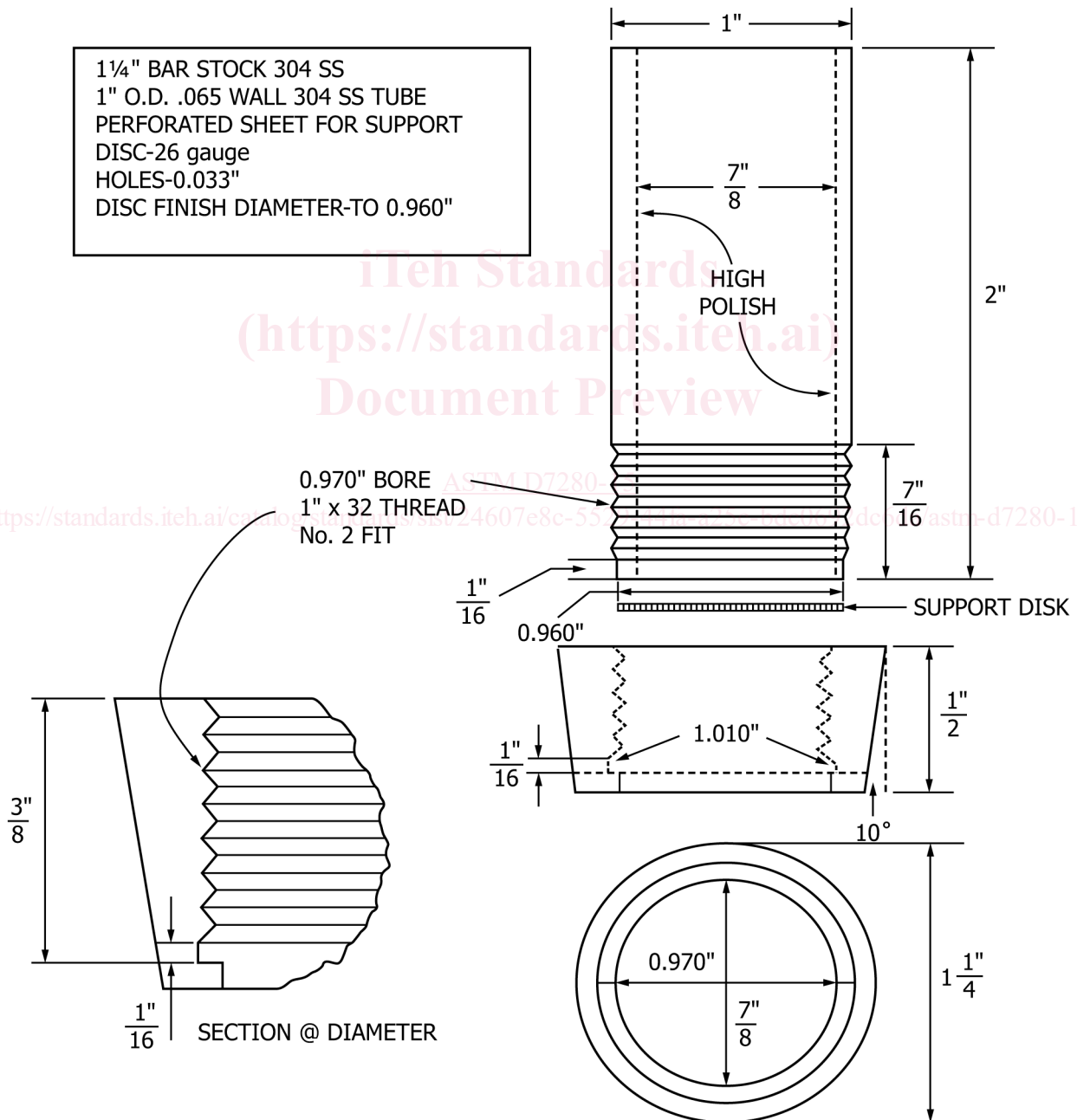


FIG. 1 Stainless Steel Filtering Crucible with Support Disk

5.5 *Electric Hot Plate or Water Bath*, maintained at $75.75\text{ }^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

6. Reagents and Materials

6.1 *Quinoline, Refined*—meeting the following requirements:

6.1.1 The quinoline shall distill from ~~55 %~~ to 95 % within a range of 2°C that shall include the temperature of ~~237.4~~ $237.4\text{ }^{\circ}\text{C}$ after corrections for barometric pressure and emergent stem have been applied. The distillation shall be carried out in accordance with Test Method **D850** using a total immersion thermometer with a range from ~~195~~ $195\text{ }^{\circ}\text{C}$ to ~~305~~ $305\text{ }^{\circ}\text{C}$, graduated in ~~0.5~~ $0.5\text{ }^{\circ}\text{C}$, and conforming to the requirements for Thermometer 69C as described in Specification **E1**. Temperature measuring devices such as precision thermocouples, resistance temperature detectors (RTDs), and liquid-in-glass thermometers with equal or better accuracies in the appropriate temperature ranges can be used.

6.1.2 The quinoline shall have a specific gravity at ~~15.5~~ $15.5\text{ }^{\circ}\text{C}$ of 1.092 to 1.098, as determined by Test Method **D71**, or another method of equivalent accuracy.

6.1.3 The quinoline shall be clear and light in color and shall contain less than 0.5 volume % of water, as determined by Test Method **D95**. If not, redistill the quinoline in all-glass apparatus, discarding the first 5 % and collecting the next 90 %. If the quinoline contains suspended matter but is clear, light in color, and contains less than 0.5 % water, filter the quinoline through a crucible containing ~~5 g~~ 5 g of diatomaceous earth filter aid.^{6,5}

6.1.4 Store the quinoline in a tightly closed, dark bottle.

6.2 *Toluene*, Industrial Pure, meeting Specification **D362**.

6.3 *Acetone*, meeting Specification **D329**.

6.4 *Filter Aid*—Dry to constant weight at ~~105~~ $105\text{ }^{\circ}\text{C}$, and store in tightly stoppered container.

NOTE 1—Manville Certified Analytical Filter Aid (CAFA) or Supelco/Sigma Aldrich (CAFA II) Part #11485U were used in determining precision and bias and were found to give acceptable filtration performance. Any other grade of filter aid may differ in filtration characteristics.

7. Hazards

7.1 Fumes of the solvents should be removed by means of proper hoods from all working areas.

7.2 The working area should be kept free of sparks and flames.

7.3 Quinoline fumes should not be inhaled, and prolonged contact with the skin should be avoided.

7.4 Toluene is toxic and flammable.

8. Bulk Sampling

8.1 Samples from shipments shall be taken in accordance with Practice **D4296** and shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

9. Dehydration of Sample

9.1 *Hard Pitch*—If the solid bulk sample contains free water, air-dry a representative portion in a forced draft oven at ~~50~~ $50\text{ }^{\circ}\text{C}$.

9.2 *Soft Pitch*—If the presence of water is indicated by surface foam on heating, maintain a representative portion of the bulk sample at a temperature between ~~125~~ $125\text{ }^{\circ}\text{C}$ and ~~150~~ $150\text{ }^{\circ}\text{C}$ in an open container until the surface is free of foam. Take care not to overheat, and remove heat source immediately when foam subsides.

9.3 *Tar*—A wet tar sample may either be dehydrated or used as received as long as conditions stated in 9.3.1 and 9.3.3 are met.

9.3.1 Dehydrate a representative portion of the bulk sample at atmospheric pressure using a simple side-arm distillation apparatus similar to the one in Test Method **D850**, and stop the distillation when the temperature reaches ~~170~~ $170\text{ }^{\circ}\text{C}$.

9.3.2 Separate any oil from the water that has distilled over (if crystals are present, warm sufficiently to ensure their solution), and thoroughly mix the oil with the residual tar in the still after the latter has cooled to a moderate temperature.

9.3.3 As an alternative to dehydration, the water content of the tar is determined by Test Method **D95**, and if the water content is less than 10 mass %, the QI content is corrected to a dry-tar basis (see 13.2). This alternative test method applies only to stable emulsions of water in tar, that is, no water separates when the tar sample is left undisturbed for 24 h at room temperature.

10. Preparation of Working Sample

10.1 *Hard Pitch*—If the pitch can be crushed at room temperature, prepare a ~~20 g~~ 20 g working sample by suitable crushing, mixing, and quartering of a representative portion of the dry sample. The crushing can be done with a small jaw crusher and a Mullite mortar and pestle. No particle in the representative sample shall be larger than ~~5 mm~~ 5 mm in any dimension.

⁶ The sole source of supply of the diatomaceous earth filter aid known to the committee at this time is Celite, available from World Minerals, Santa Barbara, CA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.