Designation: D7280 – 15

Standard Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch by Stainless Steel Crucible Filtration[1]

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*

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D362 Specification for Industrial Grade Toluene (Withdrawn 1989)[3]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

5. Apparatus

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

5.2 *Filter Membrane,* PFTE membrane without backing, 10 µm porosity, 25 mm disk.[5]

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

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

5.5 *Electric Hot Plate or Water Bath,* maintained at 75 °C ± 5 °C.

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[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.

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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.

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

[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.

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

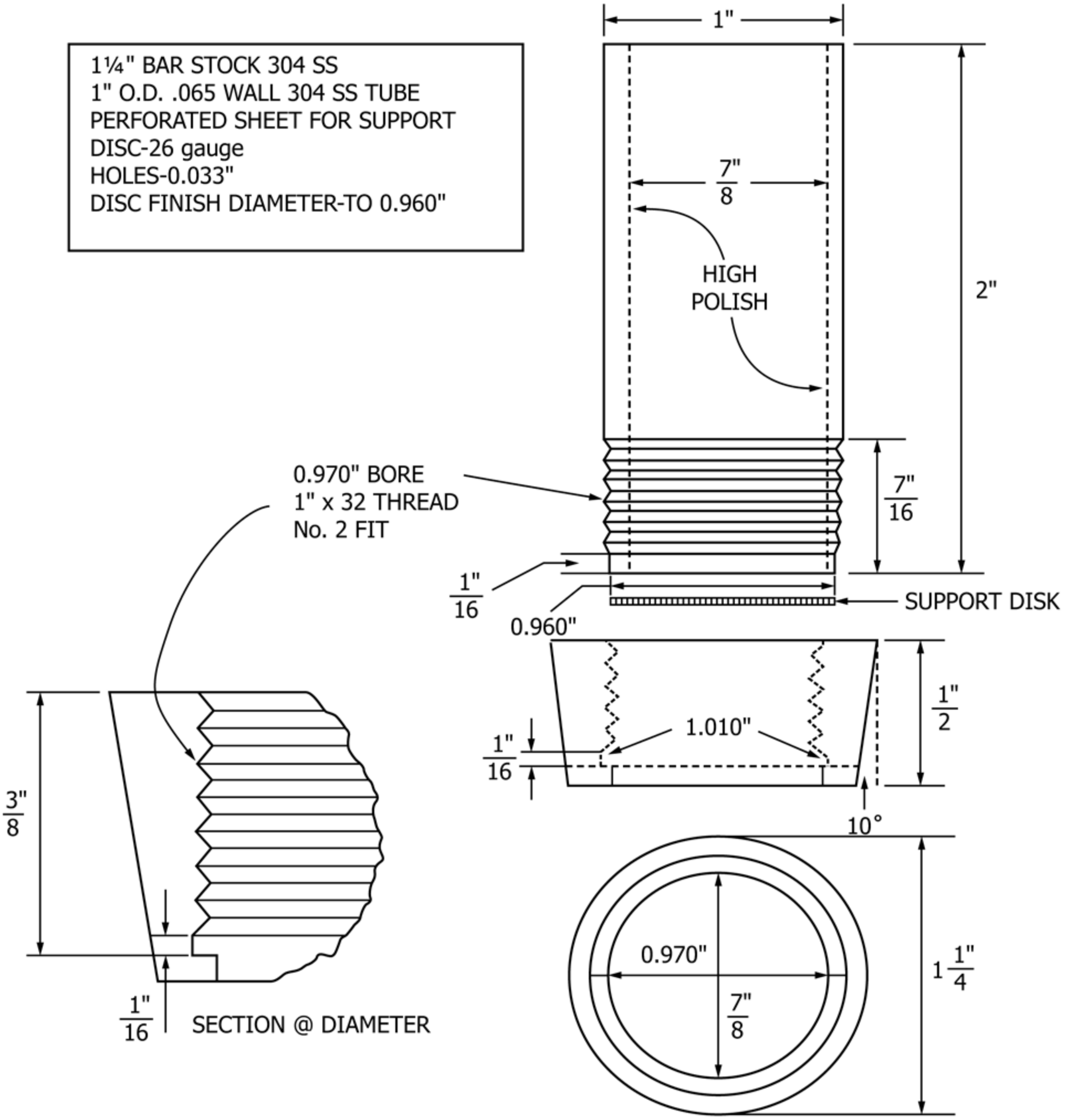

FIG. 1 Stainless Steel Filtering Crucible with Support Disk

6. Reagents and Materials

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

6.1.1 The quinoline shall distill from 5 % to 95 % within a range of 2 °C that shall include the temperature of 237.4 °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 °C to 305 °C, graduated in 0.5 °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 °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 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 °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 °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 °C and 150 °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 °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 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 in any dimension.

10.1.1 Crush this sample so that all of it will pass the 250 m (No. 60) sieve but will have a minimum of fine particles.

10.1.2 Store the sieved working sample in a tightly closed container, and use within 24 h (see 10.4).

10.2 *Soft Pitch*—If the pitch is too soft to grind and too sticky to mix, heat a representative portion of the dry sample to the lowest temperature that will permit passage through the 600 µm (No. 30) sieve, taking care to avoid excessive loss of volatile matter. Do not exceed 10 min for this melting period.

10.2.1 Pass the heated sample through the 600 µm sieve to remove foreign matter.

10.3 *Tar*—Heat a representative portion of the dry tar to the lowest temperature that will permit passage through the 600 µm (No. 30) sieve, then filter through this sieve to remove foreign matter.

10.4 *Preservation of Samples*—Store samples as large lumps or as solidified melts in closed containers. Discard working samples 24 h after crushing and sieving as changes in composition sometimes occur in pulverized pitch.

11. Filter Crucible Preparation

11.1 Unscrew the bottom ring of the filtering crucible and inspect the support disk and the ring surfaces to be certain that they are free of any foreign particles.

11.2 Place the supporting disk back into the ring and seat the disk on the inside supporting shoulder of the ring.

11.3 Using forceps, carefully place a single membrane over the threaded part of the ring onto the support disk. The membrane must be centered exactly within the threaded hole.

11.4 Screw the barrel into the ring assembly until it is finger tight. Do not overtighten the barrel and ring.

12. Procedure

12.1 Make and record all weighings to the nearest 1 mg.

12.2 Select a sample mass that will yield between 75 mg and 150 mg of matter insoluble in QI, unless this would require less than the minimum acceptable sample mass of 0.5 g, in which case 0.5 g shall be used.

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[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.

12.3 Transfer 1 g ± 0.1 g of dry certified analytical filter aid (CAFA) to a clean, dry, stainless steel filtering crucible prepared in 11.1. Weigh immediately, and record the mass of crucible plus CAFA.

12.4 After the crucible containing the diatomaceous earth filter aid has been weighed, it should be placed in a desiccator until ready to be used.

12.5 Place the required amount of sample in a tarred 100 mL or 150 mL beaker and weigh.

12.5.1 Calculate and record the mass of the sample.

12.5.2 Remove the crucible from the desiccator and transfer about half of the CAFA from the crucible to the beaker with sample.

12.5.3 Distribute the remaining CAFA evenly over the bottom of the crucible.

12.5.4 Place the crucible with the diatomaceous earth filter aid and the weighed beaker with the diatomaceous earth filter aid and sample back into the desiccator until it is ready to be used.

12.6 Heat a beaker containing quinoline on a hot plate at 75 °C ± 5 °C or place a wash bottle containing quinoline in a water bath at 75 °C ± 5 °C, and allow the quinoline to heat up to the set temperature of 75 °C ± 5 °C.

12.7 Add 25 mL of hot quinoline to the beaker while stirring the mixture with a stirring rod to break up lumps, and then cover the beaker with a small watch glass.

12.7.1 Place the beaker and a wash beaker or bottle containing quinoline on an electric hot plate or in a water bath maintained at 75 °C ± 5 °C. (A suitable weight can be used to keep the beaker from being upset in the water bath.)

12.7.2 Digest for at least 20 min.

12.7.3 Occasionally stir the contents of the beaker to promote digestion.

12.7.4 Check for completeness of digestion by inspecting the bottom of the beaker for undigested pitch.

12.8 Insert the filter tube with adapter in the filter flask, and place the previously prepared and tarred crucible in the adapter.

12.8.1 Carefully add sufficient quinoline to the crucible to wet the diatomaceous earth filter aid thoroughly.

12.8.2 Apply suction and form a mat of evenly distributed diatomaceous earth filter aid.

12.8.3 Maintain suction until filtration and subsequent washing with quinoline, toluene and acetone are completed.

12.9 Pour the hot quinoline-pitch mixture into the crucible, while the diatomaceous earth filter aid in the crucible is still wet, without disturbing the mat.

12.9.1 Stir the mixture in the beaker immediately before pouring successive portions into the crucible.

12.9.2 Allow the contents to drain completely, but not to the extent that the insoluble material on the filter appears substantially dry.

12.10 Wash the beaker, stirring rod, and crucible with small portions (3 mL to 5 mL each) of quinoline at 75 °C ± 5 °C from the wash bottle or beaker containing the heated quinoline.

12.10.1 Pass all the washes through the filter.

12.10.2 Allow each wash to pass almost completely through the filter before the next is added.

12.10.3 Use a suitable policeman to sweep the insoluble particles into the crucible.

12.10.4 Repeat the quinoline washes until the beaker is free of any insoluble material and the filtrate is the same color as the quinoline used for washing. Twelve washings are usually enough.

12.11 After the final quinoline wash has passed through the filter, fill the crucible with toluene and wash with a total of 75 mL to 100 mL of toluene using suction.

12.11.1 After all the toluene has passed through the filter, wash the contents of the crucible in the same manner with acetone.

12.11.2 Maintain full suction for a minimum of 5 min after the acetone wash.

12.11.3 Remove the crucible, and wipe the outside with a clean, soft cloth or tissue moistened with acetone.

12.11.4 During the quinoline and toluene washing, do not allow the CAFA mat to get completely dry as the filtration rate may decrease considerably.

12.12 Place the filtering crucible in the drying oven at 110 °C ± 5 °C and dry to constant weight (±1 mg). When the hot crucible is removed from the drying oven, it shall have no odor of quinoline.

12.12.1 Transfer the crucible to the desiccator and cool for 25 min, and then weigh and record the weight of the filtering crucible and its contents (see 12.1).

12.12.2 Insoluble matter on the filter, after washing with acetone, should have no odor of quinoline, which is evidence of insufficient washing. If odor of quinoline is detectable, repeat the entire determination.

12.13 If the mass of matter insoluble in quinoline is less than 75 mg or more than 150 mg, repeat the determination with an adjusted sample mass of not less than 0.5 g (see 12.2).

13. Calculation or Interpretation of Results

13.1 Calculate the QI content as follows:

$$\text{QI, mass \%} = \left[(A - B)/C \right] \times 100 \tag{1}$$

where:

A = total mass of the filtering crucible and CAFA added to the crucible, and matter insoluble in quinoline,

B = initial mass of the filtering crucible containing dried CAFA, and

C = mass of sample taken for the determination.

13.2 If the QI was determined on a wet tar sample (see 9.3.3), correct the QI value determined in 13.1 to a dry-tar basis as follows:

$$\text{QI, mass \% (dry basis)} = \frac{\text{QI, mass \% (wet basis)}}{100 - \text{water content of tar, mass \%}} \times 100 \tag{2}$$

14. Report

14.1 Report the QI content to the nearest 0.1 %.

15. Precision and Bias[7]

15.1 The precision was determined by an interlaboratory study conducted in accordance with Practice E691. In this study, eight laboratories tested four materials, and the QI content of the materials ranged from about 2 to 19 wt%. Based on this study, the criteria in 15.1.1 and 15.1.2 shall be used for judging the acceptability of results (95% probability) obtained in the QI content range of about 2 to 19 wt%.

15.1.1 *Repeatability*—Duplicate values (wt%) by the same operator shall not be considered suspect unless the determined QI values differ by more than the value of *r* given by the following expression:

$$r = 0.0074x + 0.3716$$

x = arithmetic average of measured QI values.

15.1.2 *Reproducibility*—The values (wt%) reported by each of two laboratories, representing the arithmetic average of duplicate QI determinations, shall not be considered suspect unless the reported values differ by more than the value of R given by the following expression:

$$R = 0.0281X + 0.4886$$

X = arithmetic average of duplicate QI determinations.

15.1.3 *Calculated r and R values at Various QI Levels*—The following table gives *r* and *R* values for QI levels of 2 to 20 wt% calculated using the expressions in 15.1.1 and 15.1.2:

QI, wt%	r	R
2	0.4	0.5
4	0.4	0.6
6	0.4	0.7
8	0.4	0.7
10	0.4	0.8
12	0.5	0.8
14	0.5	0.9
16	0.5	0.9
18	0.5	1.0
20	0.5	1.1

15.2 Bias can not be determined because no material having an accepted reference value is available.

15.3 *Relative Bias*—"t-test" performed on the % QI results by this test method and those obtained by Test Method D2318 using a ceramic crucible did not indicate any statistically significant difference at 95% confidence level.

16. Keywords

16.1 coal-tar insolubles; pitch; QI; quinoline insolubles; tar

SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D7280 –) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Revised subsection 6.1.1.

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1648.