



Standard Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch¹

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1. Scope

1.1 This test method covers the determination of the quinoline-insoluble matter (QI) in tar and pitch.

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

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.* For hazards information, see Section 7.

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 Toluene³

D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials

D4296 Practice for Sampling Pitch

E1 Specification for ASTM Liquid-in-Glass Thermometers

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Method

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

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

4. Significance and Use

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

5. Apparatus

5.1 *Filtering Crucibles*, porcelain, with fine-porosity bottom, 25 to 40-mL capacity, high-form, maximum pore diameter, 7 μ m.

5.2 *Filter Apparatus*—Filter flask and tube with crucible adapter.

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

5.4 *Electric Hot Plate or Water Bath*, maintained at $75 \pm 5^\circ\text{C}$.

6. Reagents and Materials

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

6.1.1 The quinoline shall distill from 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 to 305°C , graduated in 0.5°C , and conforming to the requirements for Thermometer 69C as described in Specification E1.

6.1.2 The quinoline shall have a specific gravity at $15.5/15.5^\circ\text{C}$ of 1.092 to 1.098, as determined by Test Method D71, or other 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 Celite Filter Aid.

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 *Concentrated Hydrochloric Acid*.

6.5 *Celite Analytical Filter Aid (CAFA)*—Dry to constant weight at 105°C , and store in tightly stoppered container.

6.5.1 Do not use any other grade of filter aid because filtration characteristics may differ. CAFA is manufactured only by Manville Co. and distributed through scientific supply houses.

7. Hazards

7.1 Fumes of the solvents should be removed by means of proper hoods from all working areas. The working area should be kept free of sparks and flames. Quinoline fumes should not be inhaled, and prolonged contact with the skin should be avoided. Toluene is toxic and flammable.

7.2 Observe proper laboratory procedures for handling hydrochloric and diluting acid.

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 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 [9.3.2](#) 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. 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.

9.3.2 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. Crush this sample so that *all of it* will pass the 250-µm (No. 60) sieve but will have a minimum of fine

particles. 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. 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. Crucible Preparations

11.1 If the crucible, after thorough cleaning (see [11.2](#)), has been used for less than six determinations, clean it as follows: Remove the mat, wash the crucible with distilled water, dry, and ignite in a muffle furnace for 1 h at about 800°C. Cool the crucible slowly by placing it in a drying oven for 1 h after removal from the furnace to prevent cracking and place it in a desiccator while still warm.

11.2 After the crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1 + 1 hydrochloric acid. Add equal volume of concentrated hydrochloric acid to distilled water. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as in [11.1](#).

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 and 150 mg of matter insoluble in quinoline (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.

12.3 Transfer 1 ± 0.1 g of dry CAFA to a clean, dry, filtering crucible. Weigh immediately, and record the mass of crucible plus CAFA.

12.4 Place the required amount of sample in a tared beaker and weigh. Calculate and record the mass of the sample. Transfer about half of the CAFA from the crucible to the beaker with sample. Distribute the remaining CAFA evenly over the bottom of the crucible.

12.5 Add 25 mL of quinoline to the beaker while stirring the mixture with a stirring rod or thermometer to break up lumps, then cover the beaker with a small watch glass. Place the beaker and a wash bottle containing quinoline on an electric hot plate or in a water bath maintained at $75 \pm 5^\circ\text{C}$. (A suitable weight can be used to keep the beaker from being upset in the water bath.) Digest for at least 20 min. Occasionally stir the contents of the beaker to promote digestion. Check for completeness of digestion by inspecting the bottom of the beaker for undigested pitch.

12.6 Insert the filter tube with adapter in the filter flask, and place the previously prepared and tared crucible in the adapter.

Carefully add sufficient quinoline to the crucible to wet the Celite thoroughly. Apply suction and form a mat of evenly distributed Celite. Maintain suction until filtration and subsequent washing with quinoline are completed.

12.7 Pour the hot quinoline-pitch mixture into the crucible, while the Celite in the crucible is still wet, without disturbing the mat. Stir the mixture in the beaker immediately before pouring successive portions into the crucible. Allow the contents to drain completely, but not to the extent that the insoluble material on the filter appears substantially dry.

12.8 Wash the beaker, thermometer, or stirring rod, and crucible with small portions (2 to 3 mL each) of quinoline at $75 \pm 5^\circ\text{C}$ from the wash bottle. Pass all the washes through the filter. Allow each wash to pass almost completely through the filter before the next is added. Use a suitable policeman to sweep the insoluble particles into the crucible. Repeat the quinoline washes until the filtrate is the same color as the quinoline used for washing. Twelve washings are usually enough.

12.9 After the final quinoline wash has passed through the filter, fill the crucible with toluene and wash with a total of 75 to 100 mL of toluene using suction. After all the toluene has passed through the filter, wash the contents of the crucible in the same manner with acetone. Maintain full suction for a minimum of 5 min after the acetone wash. Remove the crucible, and wipe the outside with a clean, soft cloth or tissue moistened with acetone.

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

12.10 Place the filtering crucible in the drying oven at 105 to 110°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. Transfer the crucible to the desiccator and cool for 25 min, then weigh and record the weight of the filtering crucible and its contents (see 12.1).

12.10.1 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.

12.11 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

13.1 Calculate the QI content as follows:

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

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 9.3.2), correct the QI value determined in 13.1 to a dry-tar basis as follows:

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

14. Report

14.1 Report the QI content to the nearest 0.1 %.

15. Precision and Bias

15.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

15.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 0.2 when the QI content is 4 % or less, or by more than 0.3 when the QI content is more than 4 %.

15.1.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless the reported percentages differ by more than 0.3 when the QI content is 4 % or less, or by more than 0.6 when the QI content is more than 4 %.

15.2 *Bias*—This test method has no bias because the value of quinoline-insoluble is defined in terms of this test method.

16. Keywords

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

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