



Standard Test Method for Determination of Quinoline Insolubles (QI) in Tar and Pitch by Pressure Filtration¹

This standard is issued under the fixed designation D 4746; 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.

^{e1} NOTE—Warning notes were editorially moved into the standard text in November 2003.

1. Scope

1.1 This test method covers the determination of the quinoline-insoluble matter (QI) in tar and pitch by pressure filtration and gives results comparable to those obtained by Test Method D 2318.

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

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.* Specific precautionary statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

- D 70 Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)
- D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
- D 329 Specification for Acetone
- D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials
- D 2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch
- D 4296 Practice for Sampling Pitch
- E 1 Specification for ASTM Thermometers

3. Summary of Test Method

3.1 The sample is digested in hot quinoline and filtered through a heated pressure filter. The insoluble material is washed with hot, fresh quinoline and with cold acetone, dried, and weighed.

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 *Pressure Filtration Vessel*—The pressure filtration vessel³ (see Fig. 1) is a stainless steel (304) jacketed block heated by steam or cooled with water, sealed by a lid, flat gasket, and clamp. The interior of the block is designed to accept a 3A2 Berlin porcelain filtration crucible (see 5.4). The crucible is sealed within the block by the use of three O-rings, a crucible sealing collar, and an adjustable plunger.

5.1.1 The seal is accomplished when the sealing lid is placed on top of the block and clamped. The adjustable plunger applies a force to the crucible sealing collar, which in turn pushes downward on the crucible and simultaneously compresses O-ring gaskets, forming a seal between surfaces of the crucible and the wall of the block. The filtration of material is accomplished by nitrogen (10 to 30 psig) introduced through the lid of the pressure filter. The filtrate exits from the drain tube at the bottom of the block. The filtrate is disposed into a Buchner flask, attached to drain tube by a No. 7 rubber stopper. A vacuum hose is attached to the Buchner flask and placed in a 100-mL beaker containing water to indicate the exit of nitrogen when the filtration is completed.

5.2 *Beaker*, 50 mL.

5.3 *Buchner Flask*, 500.

5.4 *Filtering Crucible*, porcelain, with a medium-porosity bottom, top outer diameter 1 $\frac{3}{4}$ in. (45 mm), bottom outer diameter 1.2 in. (30 mm), height 2 in. (50 mm).

5.5 *Analytical Balance*.

5.6 *Desiccator*.

5.7 *Thermometer*, 0 to 300°F (150°C).

5.8 *Drying Oven*, maintained at 221°F (105°C).

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 The sole source of supply of the pressure filtration vessel known to the committee at this time is Koppers Co., Inc., 1005 Wm. Pitt Way, Pittsburgh, PA 15238. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

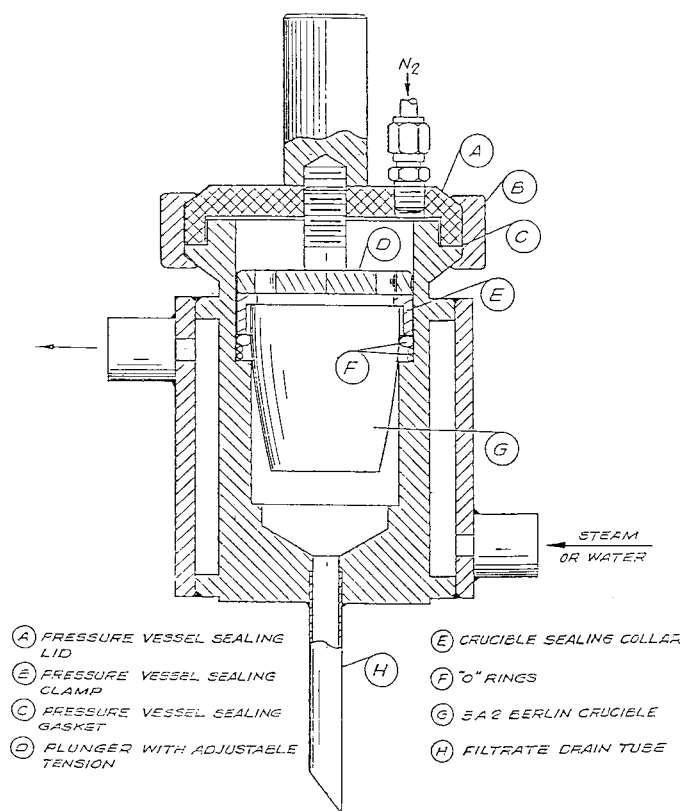


FIG. 1 Pressure Filter

- (A) PRESSURE VESSEL SEALING LID
- (E) PRESSURE VESSEL SEALING CLAMP
- (C) PRESSURE VESSEL SEALING GASKET
- (D) PLUNGER WITH ADJUSTABLE TENSION
- (E) CRUCIBLE SEALING COLLAR
- (F) O" RINGS
- (G) SA 2 EERLIN CRUCIBLE
- (H) FILTRATE DRAIN TUBE

5.9 Mortar and Pestle.

5.10 Two Wash Bottles, 500 mL.

5.11 Steam Bath.

6. Reagents

6.1 Quinoline—Refined, meeting the following requirements:

6.1.1 The quinoline shall be distilled from 5 to 95 % within a range of 2°F (1°C) that shall include the temperature of 459.3°F (237.4°C) after corrections for barometric pressure and emergent steam have been applied. The distillation shall be carried out in accordance with Method D 850 using a total-immersion thermometer with a range from 383 to 581°F (195 to 305°C), graduated in 1°F (0.5°C) and conforming to the requirements for thermometer 69C as described in Specification E 1.

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 D 70, 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 D 95. If not, redistill the quinoline in an 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 1 + 1 Hydrochloric Acid Solution—Add equal volume concentrated hydrochloric acid to distilled water. (Warning—Corrosive.)

6.3 Acetone, meeting Specification D 329. (Warning—Flammable (Health Hazard).)

7. Safety Precautions

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 skin should be avoided.

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

8. Bulk Sampling

8.1 Samples from shipments shall be taken in accordance with Practice D 4296 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 122°F (50°C).

9.2 Soft Pitch (Softening Point <140°F (60°C))—If the presence of water is indicated by surface foam on heating, maintain a representative portion of the bulk sample at a temperature between 257 and 302°F (125 and 150°C) in an open container until the surface is free of foam. Take care not to overheat, and remove the heat source immediately when the foam subsides.

9.3 Tar—A wet tar sample can 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.

9.3.2 As an alternative to dehydration, the water content of the tar is determined by Test Method D 95 and, if the water content is less than 10 weight %, the QI content is corrected to a dry-tar basis (see 13.2). This alternative 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 Pitch that is sufficiently hard at room temperature shall be finely pulverized in a mortar. Crush this sample so that all of it will pass the 250-µm (No. 60) sieve. Soft pitches and tars shall be warmed and well agitated before sampling.

10.2 The amount of sample to be taken for the test shall be such that the final quantity of quinoline insoluble (QI) shall be not less than 0.1 g. As a guide, the following sample sizes are recommended:

	Sample Size, g
Crude coke oven tar	5.0
Horizontal retort tars	2.5
Fiber pitch	5.0
Other pitches	2.5
Distilled tars	2.5
Vertical retort tars	10.0 to 25.0

10.2.1 When the sample contains 15 % QI or if the softening point is greater than 248°F (120°C), the amount used for testing should be reduced, but to not less than 0.5 g.