Designation: D4746 – 20

Standard Test Method for Determination of Quinoline Insolubles (QI) in Tar and Pitch by Pressure Filtration[1]

This standard is issued under the fixed designation D4746; 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 by pressure filtration and gives results comparable to those obtained by Test Method D2318.

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

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards are given in Section 7, 6.2, and 6.3.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D70 Test Method for Density of Semi-Solid Asphalt Binder (Pycnometer Method)

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

D329 Specification for Acetone

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

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 vessel[3] (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 psig 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

[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 sole source of supply of the pressure filtration vessel known to the committee at this time is Koppers Inc., 436 Seventh Ave., Pittsburgh, PA 15219-1800. 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.

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

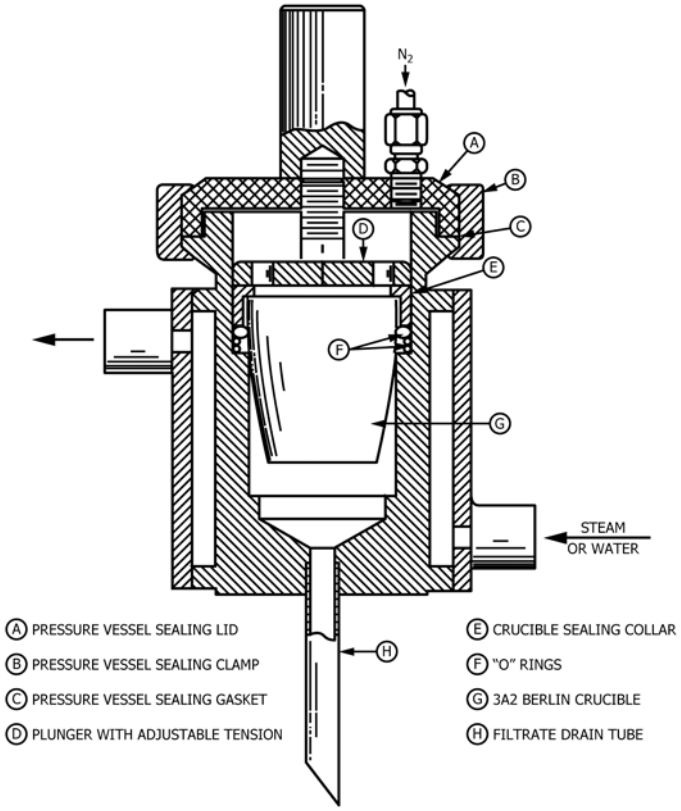

FIG. 1 Pressure Filter

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 mL.

5.4 *Filtering Crucible,* porcelain, with a medium-porosity bottom, top outer diameter 1.75 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 °F to 300 °F (150 °C).

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

5.9 *Mortar and Pestle.*

5.10 *Two Wash Bottles,* 500 mL.

5.11 *Steam Bath.*

5.12 *Sieves*—U.S. Standard 600 µm (No. 30) and 250 µm (No. 60).

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 D850 using a total-immersion thermometer with a range from 383 °F to 581°F (195 °C to 305 °C), graduated in 1 °F (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 range may 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 D70, 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 percent of water as determined by Test Method D95. 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 D329. (**Warning—** Flammable (Health Hazard).)

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 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 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 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 °F and 302 °F (125 °C 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 D95 and, if the water