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Standard Test Method for Determination of Beta Resins (BR) in Pitch¹

This standard is issued under the fixed designation D 5294; 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 beta resin (BR) in tar and pitches.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety problems, 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 specific precautionary statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation²

D 329 Specification for Acetone³

D 362 Specification for Industrial Grade Toluene³

D 370 Test Method for Dehydration of Oil-Type Preservatives⁴

D 2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch²

D 4296 Practice for Sampling Pitch²

D 4746 Test Method for Determination of Quinoline Insolubles (QI) in Tar and Pitch by Pressure Filtration²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

3. Summary of Test Method

3.1 The BR content of tar or pitch is defined as the toluene-insoluble (TI) content (as determined by the procedure in this test method) minus the quinoline-insoluble (QI) content (as determined by Test Method D 2318 or D 4746).

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 *Toluene-Insoluble (TI) Extraction Apparatus*—A 400-

mL flask with metal cap condenser, as shown in Fig. 1. The flask is available from laboratory suppliers and must be cut to the dimensions shown in Fig. 1.

5.2 *Toluene-Insoluble (TI) Filtration Apparatus*—10- μ m pore size polytetrafluoroethylene filters with a filter holder and 1-L vacuum flask, as shown in Fig. 2.

5.3 *Sieves*, U.S. Standard, 600- μ m (No. 30) and 250- μ m (No. 60), conforming to Specification E 11.

5.4 *Heater*, having a minimum capacity of 300 W per unit. A hot plate or other heaters that maintain the proper reflux rate are acceptable.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Quinoline*, refined, meeting requirements in conjunction with Test Method D 2318 or D 4746.

6.3 *Toluene*, industrial pure, meeting the requirements of Specification D 362.

6.4 *Acetone*, meeting the requirements of Specification D 329.

6.5 *Celite Analytical Filter Aid (CAFA)* (Notes 1 and 2)—Dry to a constant weight at 105°C and store in a tightly sealed container.

NOTE 1—Do not use any other grade of filter aid because filtration characteristics may differ. CAFA is manufactured only by Manville Co.⁷ and is distributed through scientific supply houses.

NOTE 2—QI can also be determined by Test Method D 4746, which does not require filter aid.

7. Safety Precautions

7.1 This test method is to be conducted in a laboratory fume hood due to the use of solvents. The working area must be kept free of sparks and flames. No solvent fumes should

¹ This test method is under the jurisdiction of ASTM Committee D-8 on Roofing, Waterproofing, and Bituminous Materials and is the direct responsibility of Subcommittee D08.17 on Industrial Pitches.

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² *Annual Book of ASTM Standards*, Vol 04.04.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 04.09.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁷ Celite Corporation (formerly Manville Co.), Wayne Interchange, Plaza II, 155 Rt. 46, Wayne, NJ, 07470.

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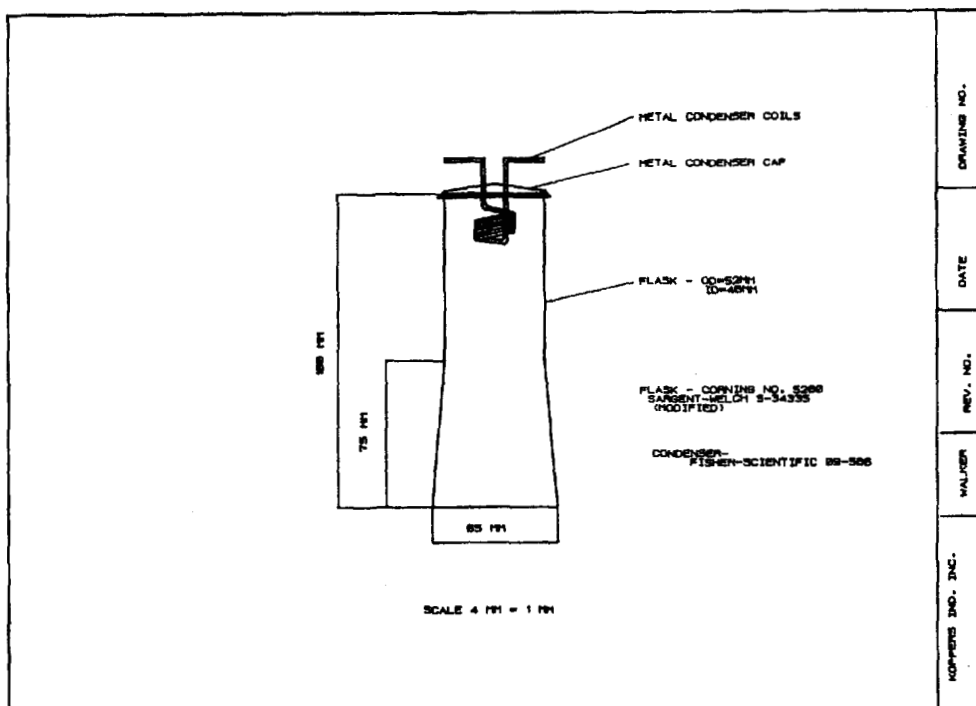


FIG. 1 Extraction Apparatus

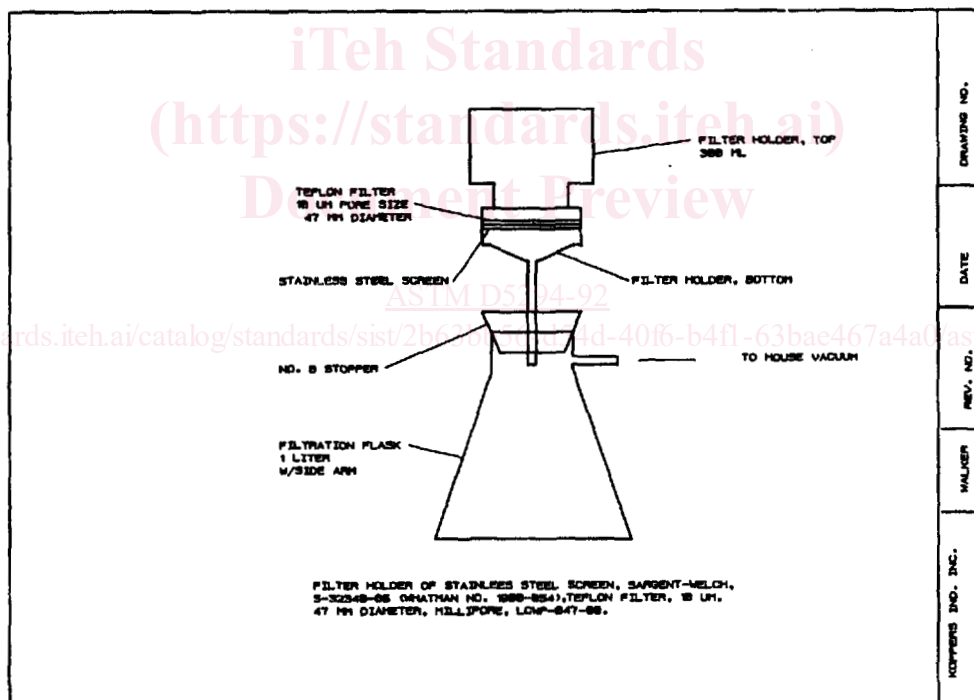


FIG. 2 Filtration Apparatus

be inhaled, and prolonged contact with skin should be avoided.

8. Bulk Sampling

8.1 Specimens from shipments shall be taken in accordance with Practice D 4296 and shall be free of foreign substances. Thoroughly mix the specimen immediately be-

fore removing a representative portion for the determination or for dehydration.

9. Dehydration of Specimen

9.1 *Hard Pitch*—If the solid bulk specimen contains free water, air-dry a representative portion by spreading on a tray for 4 h.

9.2 *Soft Pitch*—If the presence of water is indicated by