Designation: D4072 – 23

Standard Test Method for Toluene-Insoluble (TI) Content of Tar and Pitch[1]

This standard is issued under the fixed designation D4072; 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 toluene-insoluble matter (TI) 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard information, see Section 8.

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]

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

D362 Specification for Industrial Grade Toluene (Withdrawn 1989)[3]

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4296 Practice for Sampling Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

4. Summary of Test Method

4.1 The sample is digested, then extracted with hot toluene in an alundum thimble. The insoluble matter is dried and weighed.

5. Significance and Use

5.1 This test method is useful for evaluating and characterizing tars and pitches and as one element in establishing the uniformity of shipments or sources of supply.

6. Apparatus

6.1 *Extraction Apparatus,* Flask with metal cap condenser as shown in Fig. 1.

6.2 *Extraction Thimble,* Alundum AN 485 coarse (formerly RA 98), 30 mm in diameter by 80 mm in height with flat bottom.

6.3 *Thimble Cover,* Paper cone, made by wetting with water a 70 mm filter paper normally folded in a small glass funnel, and drying the funnel in an oven with the paper cone in place.

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

6.5 *Heater,* having a minimum capacity of 300 W per unit.

7. Reagents

7.1 *Toluene, Industrial Pure,* meeting Specification D362. (**Warning—**Flammable.)

7.2 *Concentrated Hydrochloric Acid.* (**Warning—** Corrosive.)

8. Hazards

8.1 Since toluene is a toxic and flammable substance, all working areas should be efficiently hooded and kept free of sparks and flames.

[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.

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

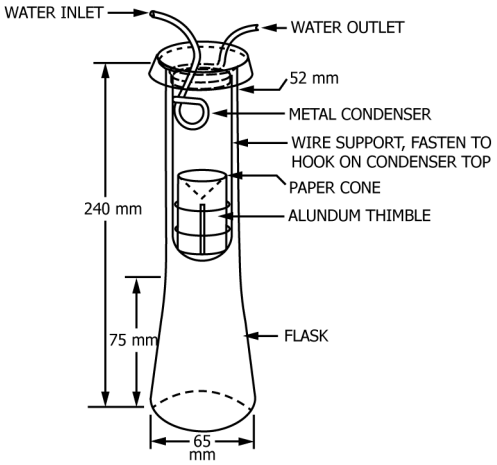

FIG. 1 Extraction Apparatus

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

9. Bulk Sampling

9.1 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.

10. Dehydration of Sample

10.1 *Hard Pitch*—If the solid bulk sample contains free water, air-dry a representative portion in a forced draft oven at 50 °C.

10.2 *Soft Pitch*—If the presence of water is indicated by surface foam on heating, maintain a representative portion of the bulk sample of 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.

10.3 *Tar*—Dehydrate a representative portion of the bulk sample at atmospheric pressure using a simple sidearm 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.

10.3.1 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 % by mass, the TI content is corrected to a dry-tar basis (see 14.2). 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.

11. Preparation of Working Sample

11.1 *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. Store the sieved working sample in a tightly closed container and use within 24 h (see 11.4).

11.2 *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. Do not exceed 10 min for this melting period. Pass the heated sample through the 600 µm sieve to remove foreign matter.

11.3 *Tar*—Heat a representative portion of the 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.

11.4 Discard working samples 24 h after crushing and sieving as changes in composition sometimes occur in pulverized pitch.

12. Preparation of Extraction Thimble

12.1 Dry the clean thimble in an oven at 105 °C ± 5 °C for 45 min, cool in a desiccator, and weigh to the nearest 0.5 mg. Record the mass.

12.1.1 After each use, ignite the thimble at 700 °C to 800 °C for a few hours. Cool the thimble slowly by placing in a drying oven for 1 h after removal from the furnace to prevent cracking. Before reuse, condition the thimble as described in 12.1.

12.1.2 After repeated use, boil the thimble in 1 + 1 hydrochloric acid. (Add equal volume of concentrated hydrochloric acid to distilled water.) to remove residual ash from the pores. Then boil the thimble in distilled water and wash with water. After drying at 105 °C, ignite and condition the thimble as described in 12.1 and 12.1.1.

13. Procedure

13.1 The mass of sample taken for analysis shall yield between 150 mg and 250 mg of matter insoluble in toluene (TI), unless this would require less than the minimum acceptable sample mass of 1 g, in which case 1 g shall be used.

13.2 Place the required amount of sample in a tared 150 mL beaker and weigh to the nearest 0.5 mg. Pour 60 mL of toluene into the beaker, stirring continuously while introducing the toluene, to ensure complete mixture of the sample and toluene and thorough dispersion of the insoluble matter (see 8.1). Place the beaker on an electric hot plate or on a steam or water bath and heat the contents to a temperature of 95 °C ± 5 °C. Maintain this temperature for 25 min. Stir occasionally with a stirring rod during this period to ensure that the sample is completely dispersed in the toluene. Check for completeness of dispersion by inspecting the bottom of the beaker for undigested material.

13.3 Place the tared extraction thimble in a filter tube supported over a beaker or flask. Wet the thimble completely with toluene. After digestion, carefully decant portions of the hot mixture into the wetted thimble (see 8.1). Do not permit the level of the toluene to rise higher than 20 mm from the top of