



Standard Test Method for Toluene-Insoluble (TI) Content of Tar and Pitch¹

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^{ε1} NOTE—Warning notes were editorially moved into the standard text in November 2003.

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 the 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 specific hazard information, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

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

D 362 Specification for Industrial Grade Toluene

D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials

D 4296 Practice for Sampling Pitch

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Summary of Test Method

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

4. Significance and Use

4.1 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.

5. Apparatus

5.1 *Extraction Apparatus*—Flask with metal cap condenser as shown in Fig. 1.

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

5.3 *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.

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

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

6. Reagents

6.1 *Toluene, Industrial Pure*, meeting Specification D 362. (**Warning**—Flammable.)

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

7. Hazards

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

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 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.

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.

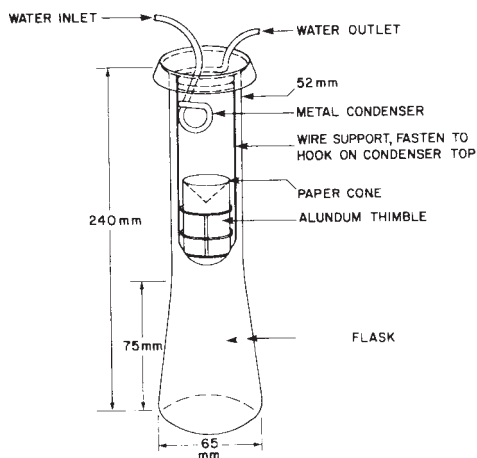


FIG. 1 Extraction Apparatus

9.3 *Tar*—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 D 850 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.

9.3.1 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 mass percent, the TI 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 *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- μ m (No. 60) sieve. 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- μ m (No. 30) sieve. Do not exceed 10 min for this melting period. Pass the heated sample through the 600- μ m sieve to remove foreign matter.

10.3 *Tar*—Heat a representative portion of the tar to the lowest temperature that will permit passage through the 600- μ m (No. 30) sieve; then filter through this sieve to remove foreign matter.

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

11. Preparation of Extraction Thimble

11.1 Dry the clean thimble in an oven at $105 \pm 5^\circ\text{C}$ for 45 min, cool in a desiccator, and weigh to the nearest 0.5 mg. Record the mass.

11.1.1 After each use, ignite the thimble at 700 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 11.1.

11.1.2 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 11.1 and 11.1.1.

12. Procedure

12.1 The mass of sample taken for analysis shall yield between 150 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.

12.2 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 7.1). Place the beaker on an electric hot plate or on a steam or water bath and heat the contents to a temperature of $95 \pm 5^\circ\text{C}$. 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.

12.3 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 7.1). Do not permit the level of the toluene to rise higher than 20 mm from the top of the thimble. It is advisable while filtering to retain the major part of the insoluble matter in the beaker. Finally transfer the insoluble matter from the beaker to the thimble with small portions of toluene. Wash the beaker with toluene until it is clean, using a suitable brush or policeman to detach any particles adhering to the beaker. Transfer all washings to the thimble.

12.4 When almost all toluene has drained from the thimble, wash the thimble and its contents once with a small quantity of toluene. Allow the toluene to drain from the thimble.

12.5 While the extraction thimble is still moist with toluene, remove it from the filter tube and place it in the wire support of the extraction apparatus. Cover the thimble with the paper cone, point down. Assemble the extraction apparatus on a hot plate, with a felt gasket between the top of the extraction flask and condenser cover, and with fresh toluene in the bottom of the extraction flask. Add three to six small lumps of pumice stone to the flask to promote smooth boiling, thereby preventing bumping and spattering during extraction. The surface of the toluene in the flask shall be about 25 mm below the bottom of the thimble (see 7.1).

12.6 Conduct the extraction at such a rate of heating that condensed toluene falls continuously from the condenser at a rate of not less than 1 nor more than 2 drops/s. Continue the extraction at this rate for a period of 18 h (Note 1).

NOTE 1—The reflux rate can be affected by the ambient temperature, especially when the apparatus is operating overnight unattended (for example, the temperature of the laboratory is reduced at night and the reflux rate slows or ceases). One way to alleviate this condition is to place a metal shield around the heater and extraction apparatus which would prevent the colder air entering the hood from excessively cooling the extraction apparatus. The shield can be as simple as a can with the ends cut out.

12.7 At the end of this period, remove the heat source and allow the toluene to drain from the thimble, then remove the thimble from the extraction apparatus. Remove the paper cone and gently lay the thimble on its side, with the open end tilted up, on a watch glass in the fume hood so that the toluene can evaporate. Leave it for 30 min. Place the air-dried thimble in a drying oven at $105 \pm 5^\circ\text{C}$ for 60 min. Remove the dry thimble from the oven and place it immediately in a desiccator. When cool, weigh the thimble and contents to the nearest 0.5 mg, and record the mass (see 12.8).

12.8 If the mass of matter insoluble in toluene is less than 150 mg or more than 250 mg, repeat the determination with an adjusted sample weight of not less than 1 g (see 11.1).

13. Calculation

13.1 Calculate the toluene-insoluble (TI) content as follows:

$$TI, \% = [(B - A)/C] \times 100 \quad (1)$$

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where:

- A = mass of alundum thimble without cone,
- B = mass of alundum thimble plus toluene-insoluble material, and
- C = mass of sample.

13.2 If the TI was determined on a wet tar sample (see 9.3.1), correct the TI value determined in 13.1 to a dry-tar basis as follows:

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

14. Report

14.1 Report the toluene-insoluble (TI) 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 0.9.

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 2.0.

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

16. Keywords

16.1 extraction; pitch; tar; toluene-insolubles