



Designation: ~~D4072 – 98 (Reapproved 2013)~~ D4072 – 98 (Reapproved 2018)

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

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 7.
- 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[D362 Specification for Industrial Grade Toluene \(Withdrawn 1989\)³](#)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](#)

[D4296 Practice for Sampling Pitch](#)

[E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves](#)

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~~ 30 mm in diameter by ~~80-mm~~ 80 mm in height with flat bottom.
- 5.3 *Thimble Cover*, Paper cone, made by wetting with water a ~~70-mm~~ 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- μ m~~ Standard 600 μ m (No. 30) and ~~250- μ m~~ 250 μ m (No. 60), conforming to Specification [E11](#).
- 5.5 *Heater*, having a minimum capacity of ~~300-W~~ 300 W per unit.

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.

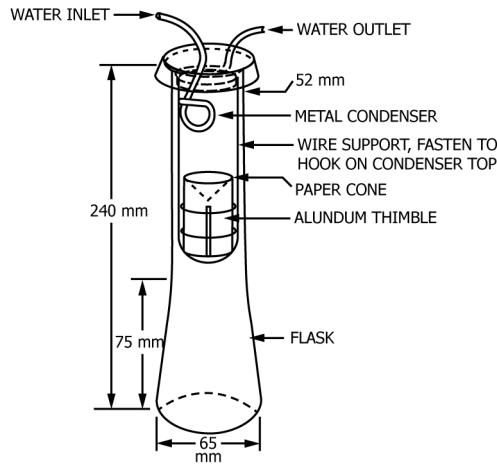


FIG. 1 Extraction Apparatus

6. Reagents

6.1 *Toluene, Industrial Pure*, meeting Specification **D362**. (**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 **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 50°C – 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°C – 125°C and 150°C – 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.

9.3 *Tar*—Dehydrate a representative portion of the bulk sample at atmospheric pressure using a simple sidarm distillation apparatus similar to the one in Test Method **D850** and stop the distillation when the temperature reaches 170°C – 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 **D95** and, if the water content is less than ~~10 mass percent~~, 10 % by mass, 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~~–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~~–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~~–5 mm in any dimension. Crush this sample so that all of it will pass the ~~250- μm~~ –250 μm (No. 60) sieve. Store the sieved working sample in a tightly closed container and use within ~~24 h~~–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- μm~~ –600 μm (No. 30) sieve. Do not exceed ~~10 min~~–10 min for this melting period. Pass the heated sample through the ~~600- μm~~ –600 μm sieve to remove foreign matter.