



Designation: **D4312 – 95a (Reapproved 2010) D4312 – 15**

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

This standard is issued under the fixed designation D4312; 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—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.
- 1.3.1 *Exception*—Other non-SI units are provided for information only.

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.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards*:²

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

D329 Specification for Acetone

D362 Specification for Industrial Grade Toluene (Withdrawn 1989)³

~~D370 Practice for Dehydration of Oil-Type Preservatives~~

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

D4072 Test Method for Toluene-Insoluble (TI) Content of Tar and Pitch

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 is one element in establishing the uniformity of shipment or sources of supply.

5. Apparatus

5.1 *Extraction Apparatus*, flask with metal cap condenser as shown in Fig. 1.

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

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

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.

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

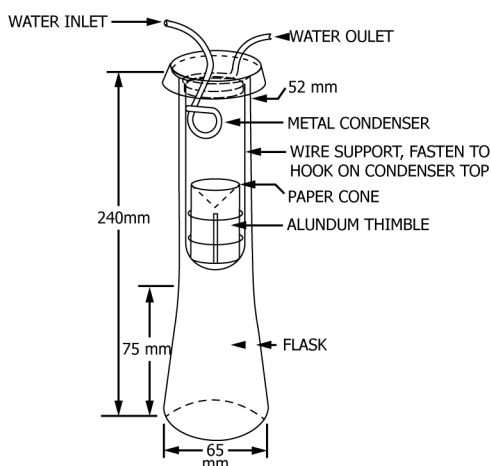


FIG. 1 Extraction Apparatus

6. Reagents

6.1 *Toluene, Industrial Pure*, meeting Specification D362.

6.2 *Acetone*, meeting the requirements of Specification D329.

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.

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.

9.2 *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 $\pm 25 \pm 125^\circ\text{C}$ and $\pm 50^\circ\text{C} \pm 150^\circ\text{C}$ 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 in accordance with at atmospheric pressure using a simple side-arm distillation apparatus similar to the one in Test Method D370D850, but and stop the distillation when the temperature reaches $\pm 70^\circ\text{C} \pm 170^\circ\text{C}$. Separate any oil from the water that has distilled over. (If crystals are present, warm sufficiently to ensure their solution). Thoroughly 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 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 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.

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-%, 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*—Crush samples to pass a $250\text{-}\mu\text{m}$ (60-mesh) $250\text{-}\mu\text{m}$ (60 mesh) screen but so they are retained on a $150\text{-}\mu\text{m}$ (100-mesh) $150\text{-}\mu\text{m}$ (100 mesh) screen. Remove pitch from $150\text{-}\mu\text{m}$ (100-mesh) $150\text{-}\mu\text{m}$ (100 mesh) screen by a brush. Crushing can be done with a small jaw crusher or a mortar and pestle, or both. No particle in the representative sample should be larger than 5 mm-5 mm in any dimension. Store sieved working sample in a tightly closed container and use within 24 h-24 h.

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 $600\text{-}\mu\text{m}$ $600\text{-}\mu\text{m}$ (No. 30) sieve, taking care to avoid excessive loss of volatile matter. Do not exceed 10 min for this melting period. Pass the heated sample through the $600\text{-}\mu\text{m}$ $600\text{-}\mu\text{m}$ sieve to remove foreign matter.