Designation: D4312 – 95a (Reapproved 2005)

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

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.

1.4 *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[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 (No. 30) and 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.

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 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.

Current edition approved May 1, 2005. Published May 2005. Originally approved in 1983. Last previous edition approved in 2000 as D4312–95a (2000)$^{\varepsilon 1}$. DOI: 10.1520/D4312-95AR05.

[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] Withdrawn.

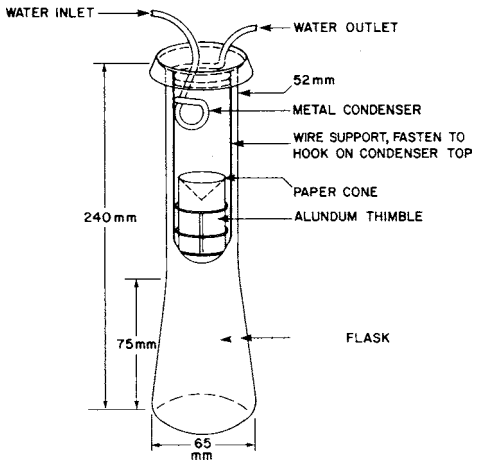

FIG. 1 Extraction Apparatus

9.3 *Tar*—Dehydrate a representative portion of the bulk sample in accordance with Test Method D370, but 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). 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.

9.3.2 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 %, 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*—Crush samples to pass a 250-µm (60-mesh) screen but so they are retained on a 150 µm (100-mesh) screen. Remove pitch from 150-µm (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 in any dimension. Store sieved working sample in a tightly closed container and use within 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-µm (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-µm sieve to remove foreign matter.

10.3 *Tar*—Heat a representative portion of the dry 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.

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

11. Preparation of Alundum Thimble

11.1 Dry the clean thimble in an oven at 105 ± 5°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 hydrochloric acid (1 + 1) 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 Weigh 0.50 g sample to the nearest 0.5 mg in a 150 mL tared beaker.

12.2 While stirring sample with a glass rod, add 75 mL of hot toluene (95 ± 5°C) and continue stirring for a few minutes until the sample is dispersed. Digest sample containing toluene on an appropriate heat source (preferably a hot water or steam bath) at 95 ± 5°C for 25 min with occasional stirring.

12.3 While sample is digesting, weigh the alundum thimble, AN 485 (formerly RA 98, 20-µm porosity, 30 by 80-mm size) to nearest 0.5 mg. Place the thimble in a filtering tube, which is supported on a funnel rack or equivalent apparatus to drain soluble portion of sample.

12.4 Pour the digested sample into the thimble. Avoid filling the thimble higher than 20 mm from the top. Use a glass rod, a brush, or a rubber policeman, if necessary, and cold toluene from a wash bottle to completely transfer the insoluble particles into the thimble.

12.5 When most of the solution has drained from the thimble, transfer the thimble to the extraction apparatus as described in Test Method D4072. With a beaker, add hot toluene to extraction flask. Fill to a level approximately 25 mm below the bottom of thimble.

12.6 Adjust the hot plate, and set the drip (reflux) rate of toluene at 120 to 150 drops/min from the condenser to maintain the level of liquid in the thimble. Continue extraction for 3 h. The liquid level is easily observed if the paper cone is replaced with one made from coarse stainless steel or brass screen. However, it is not necessary to use a cone.

NOTE 1—One could fill extraction flask to required level, turn heat on before thimble is placed into basket.

12.7 Cut heat off, let drain, remove with tongs, and place thimble in filtering tube. Wash the inside and outside of thimble with acetone several times from a wash bottle, starting from top of thimble (several small washes are more efficient than

filling the thimble). Place thimble in an oven at 110 ± 5°C for 30 min. Cool in a desiccator and weigh.

13. Calculation

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

$$\text{TI, mass \%} = 100(B - A)/C \quad (1)$$

where:

A = mass of the alundum thimble,
B = mass of alundum thimble plus toluene-insolubles, and
C = mass of sample.

13.2 If the TI was determined on a wet tar sample (see 9.3.2), correct the TI value determined in 13.1 to a dry-tar basis as follows:

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

14. Report

14.1 Report the toluene-insoluble (TI) content to the nearest 0.1 %.

15. Precision and Bias

15.1 Based upon present available data, 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 1.0.

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 3.0.

15.2 *Bias*—During the interlaboratory study to determine the precision of this test method, the TI was also determined by Test Method D4072 (long method). It was found that the TI determined by Test Method D4312 was higher than that determined by Test Method D4072 by an average value of about 1 percentage point.

16. Keywords

16.1 beta resin; pitch; soft pitch; tar; toluene insolubles