Designation: ~~D2764 – 99 (Reapproved 2009)~~ D2764 – 99 (Reapproved 2015)$^{\varepsilon 1}$

Standard Test Method for Dimethylformamide-Insoluble (DMF-I) Content of Tar and Pitch[1]

This standard is issued under the fixed designation D2764; 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.

ε[1] NOTE—SI units formatting was corrected editorially in May 2015.

1. Scope

1.1 This test method covers the determination of the dimethylformamide-insoluble matter (DMF-I) 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 inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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 Sections 6 and 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D329 Specification for Acetone
D370 Practice for Dehydration of Oil-Type Preservatives
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 A sample is digested in hot DMF and filtered. Any insoluble matter is washed, dried, and weighed.

4. Significance and Use

4.1 This test method is useful in evaluating and characterizing tars and pitches and as one element in establishing the uniformity of shipments or sources of supply. It is a rapid and reasonably accurate measure of the toluene insoluble (TI) content of tar and pitch Test Method D4072.

5. Apparatus

5.1 *Filtering Crucible,* porcelain, with fine-porosity bottom, ~~30~~30 mL to ~~40 mL~~40 mL capacity, high form, maximum pore diameter 7 µm.

5.2 *Filter Apparatus*—Filter flask and tube with crucible adapter.

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 *Water Bath,* maintained at ~~203~~203 °F to ~~212°F (95~~212 °F (95 °C to ~~100°C).~~100 °C).

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ 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.

6. Reagents and Materials

6.1 *Dimethylformamide,* reagent grade, boiling range ~~4°F (2°C) including 307°F (153°C).~~4 °F (2 °C) including 307 °F (153 °C). Store over a suitable desiccant. Decant immediately before use. If necessary, filter through a plug of glass wool or absorbent cotton until optically clear.

6.2 *Acetone,* meeting Specification D329. (**Warning—**Flammable. Health hazard.)

6.3 *Concentrated Hydrochloric Acid.*

6.4 *Celite Analytical Filter Aid (CAFA)*—Dry to constant ~~weight~~mass at ~~22°F (105°C)~~22 °F (105 °C) and store in tightly stoppered container.

NOTE 1—Do not use any other grade of filtering medium because porosities differ.

7. Hazards

7.1 Fumes of the solvents should be removed by means of proper hoods from all working areas. The working area should be kept free of sparks and flames. DMF fumes should not be inhaled, and prolonged contact of DMF with the skin should be avoided.

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. The sample shall be thoroughly mixed 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 ~~257~~257 °F and ~~302°F (125~~302 °F (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 in accordance with Test Method D370, but stop the distillation when the temperature reaches ~~338°F (170°C).~~338 °F (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. 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 but have a minimum of fine particles. 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, taking care to avoid excessive loss of volatile matter. 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.

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

10.4 *Preservation of Working Samples*—Store samples as large lumps or as solidified melts in closed containers. Discard working samples 24 h after crushing and sieving since changes in composition sometimes occur in pulverized pitch.

11. Crucible Preparation

11.1 If the crucible, after thorough cleaning (11.2), has been used for less than six determinations, clean it as follows. Remove the mat, wash the crucible with distilled water, dry, and ignite in a muffle furnace for ~~1 h~~ 1 h at about ~~1472°F (800°C).~~1472 °F (800 °C). Cool the crucible slowly by placing it in a drying oven for ~~1 h~~ 1 h after removal from the furnace to prevent cracking and place it in a desiccator while still warm.

11.2 After the crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1 + 1 hydrochloric acid. Add equal volume of concentrated hydrochloric acid to distilled water. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as in 11.1.