Designation: D2764 – 99 (Reapproved 2009)

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.

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 to 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 (No. 30) and 250-µm (No. 60), conforming to Specification E11.

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

6. Reagents and Materials

6.1 *Dimethylformamide*, reagent grade, boiling range 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 at 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.

[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.

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 257 and 302°F (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.

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). 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 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-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 (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 (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 *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 at about 1472°F (800°C). Cool the crucible slowly by placing it in a drying oven for 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.

12. Procedure

12.1 Make and record all weighings to the nearest 0.5 mg.

12.2 Transfer 0.45 to 0.55 g of Celite to a clean, filtering crucible. Distribute the Celite evenly over the bottom. Dry in an oven at 22 to 230°F (105 to 110°C) for 30 min. Cool in a desiccator and weigh. Record the weight of crucible plus Celite.

12.3 Transfer 0.45 to 0.55 g of previously dried Celite to a clean, dry, and tared 100-mL beaker and weigh. Record the weight of Celite. Place 0.5 g of working sample in the same beaker and reweigh. Calculate and record the weight of the sample.

12.4 Add 25 mL of *dry* dimethylformamide (DMF) to the beaker while stirring the mixture with a stirring rod or thermometer to break up lumps, then cover the beaker with a small watch glass. Place the beaker and a wash bottle containing DMF in a water bath maintained at 203 to 212°F (95 to 100°C). (A suitable weight can be used to keep the beaker from being upset in the water bath.) Digest for at least 30 min. Occasionally stir the contents of the beaker to promote digestion. Check for completeness of digestion by inspecting the bottom of the beaker for undigested pitch or tar.

12.5 Insert the filter tube with adapter into the filter flask and place the previously prepared and tared crucible in the adapter. Carefully add sufficient DMF to the crucible to wet the Celite thoroughly. Apply suction, and form a mat of evenly distributed Celite. Maintain suction until filtration and subsequent washing with DMF are completed.

12.6 Pour the hot DMF-pitch mixture into the crucible, while the Celite in the crucible is still wet without disturbing the mat. Stir the mixture in the beaker immediately before pouring successive portions into the crucible. Allow the contents to drain completely, but not to the extent that the insoluble material on the filter appears substantially dry.

12.7 Wash the beaker, thermometer or stirring rod, and crucible with small portions (2 to 3 mL each) of DMF at 203 to 212°F (95 to 100°C) from the wash bottle. Pass all the washes through the filter. Allow each wash to pass almost completely through the filter before the next is added. Use a suitable policeman to sweep the insoluble particles into the crucible. Repeat the DMF washes until the filtrate is the same color as the DMF used for washing. Twelve washings are usually enough.

12.8 Discontinue the suction. Fill the crucible with acetone; reapply suction until the solvent has passed through the filter, then discontinue suction and repeat the operation with fresh acetone three more times. Maintain full suction for a minimum of 5 min after the last acetone wash. Remove the crucible and wipe the outside with a clean, soft cloth or tissue moistened with acetone.

12.9 Place the filtering crucible in the drying oven at 222 to 230°F (105 to 110°C) and dry to constant weight (±0.5 mg). When the hot crucible is removed from the drying oven, it should have no odor of DMF (Note 1). Transfer the crucible to the desiccator and cool for 25 min, then weigh and record the weight of the filtering crucible and its contents.

NOTE 2—Insoluble matter on the filter, after washing with acetone, should have no odor of DMF, which is evidence of insufficient washing. If odor of DMF is detectable, repeat the entire determination.

13. Calculation

13.1 Calculate the DMF-insoluble (DMF-I) content as follows:

$$\text{DMF}-\text{I}, \% = [(A - B - C)/D] \times 100$$

where:

A = total weight of the filtering crucible, Celite (added to the crucible and to the sample), plus DMF insoluble matter.
B = initial weight of the filtering crucible containing dried Celite,
C = weight of dry Celite added to the sample, and
D = weight of working sample used for the determination.

14. Report

14.1 Report the percentage of matter insoluble in dimethylformamide (DMF-I) to the nearest 0.1 %. Duplicate runs that agree within 1.0 % absolute are acceptable for averaging (95 % confidence level).

15. Precision and Bias

15.1 The following criteria should be used for judging the acceptability of results.

15.1.1 *Repeatability*—The average difference between two results (each the average of duplicate determinations), obtained by the same analyst on different days, will approximate 0.4 %. Two such values should be considered suspect (95 % confidence level) if they differ by more than 1.0 % absolute.

15.1.2 *Reproducibility*—The average difference between two results (each the average of duplicate determinations), obtained by analysts in different laboratories, will approximate 0.8 %. Two such values should be considered suspect (95 % confidence level) if they differ by more than 2.5 % absolute.

15.2 *Bias*—This test method has no bias because the value of DMF-1 is defined in terms of this test method.

16. Keywords

16.1 dimethylformamide; insoluble; insoluble matter; pitch; tar