Designation: D2415 – 15

Standard Test Method for Ash in Coal Tar and Pitch[1]

This standard is issued under the fixed designation D2415; 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 ash content of tar and pitch.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 carefully volatilized and burned in a muffle furnace or by other suitable means, after which the carbonaceous residue is completely oxidized and the remaining ash stabilized at 900 °C in the muffle furnace.

4. Significance and Use

4.1 This test method determines the amount of inorganic matter in the sample.

5. Apparatus

5.1 *Muffle Furnace*—A muffle furnace with good air circulation and capable of having its temperature regulated at 900 °C ± 10 °C.

5.2 *Dish or Crucible,* porcelain, silica, or platinum, having a capacity of 35 mL to 45 mL and a diameter at the top of 55 mm to 60 mm.

5.3 *Sieve,* U.S. Standard 600 µm (No. 30), conforming to Specification E11.

6. Bulk Sampling

6.1 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.

7. Dehydration of Sample

7.1 *Hard Pitch*—If the solid bulk sample contains free water, air-dry a representative portion in a forced draft oven at 50 °C.

7.2 *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 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.

7.3 *Tar*—Dehydrate a representative portion of the bulk sample in accordance with Test Method D850, but stop the distillation when the temperature reaches 170 °C. Separate any oil from the water which 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.

8. Preparation of Working Sample

8.1 *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 600 µm (No. 30) sieve.

8.2 *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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

Current edition approved Dec. 1, 2015. Published December 2015. Originally approved in 1965. Last previous edition approved in 2012 as D2415 – 98 (2012). DOI: 10.1520/D2415-15.

[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.

***A Summary of Changes section appears at the end of this standard**

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.

8.3 *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.

8.4 *Preservation of Samples*—Store samples as large lumps or as solidified melts in closed containers. Do not save crushed samples for future analyses since changes in composition sometimes occur in pulverized pitch.

9. Procedure

9.1 Ignite a clean dish or crucible for 1 h in the muffle furnace at 900 °C ± 10 °C. Cool slowly to about 100 °C, then place the dish or crucible in a desiccator. When at room temperature, weigh to the nearest 0.1 mg.

9.2 Transfer a 10 g portion of the representative, dehydrated sample to the tared dish or crucible and weigh to the nearest 0.1 mg. Place the container and the sample in the cold muffle furnace and gradually heat to redness at a rate that avoids mechanical loss from boil-over or spattering, due to too rapid an expulsion of volatile matter. Instead of the muffle furnace, a hot plate or gas flame may be used to remove volatiles, as long as the same precautions against mechanical loss are taken. After the volatile matter has been driven off and a semi-coke remains, complete the ignition in the muffle furnace at 900 °C ± 10 °C. When all carbon appears to have burned off, cool the dish or crucible to about 100 °C before placing it in a desiccator. When at room temperature weigh to the nearest 0.1 mg. Repeat the ignition at 900 °C ± 10 °C for 30 min intervals until constant weight is obtained.

10. Calculation

10.1 Calculate the ash content of the sample as follows:

$$\text{Ash, \%} = 100A/B \quad (1)$$

where:

A = weight of ash, and
B = weight of sample.

11. Report

11.1 Report the weight percent of ash to the nearest 0.01 %.

12. Precision and Bias

12.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

12.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 0.01.

12.1.2 *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 0.03.

12.2 *Bias*—This procedure has no bias because the value of ash is defined in terms of this test method.

13. Keywords

13.1 ash; inorganic matter; pitch; tar

SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D2415 – 98 (2012)) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Replaced Practice D370 with Test Method D850.