Designation: D4715 – 98 (Reapproved 2008)ε1

Standard Test Method for Coking Value of Tar and Pitch (Alcan)[1]

This standard is issued under the fixed designation D4715; 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—Changed to SI-only standard editorially in February 2009.

1. Scope

1.1 This test method covers the determination of the coking value of tar and pitch having an ash content not over 0.5 %, as determined by Test Method D2415.

1.2 Coking values by this test method are higher than those obtained by Test Method D2416. See the bias statement in Section 13.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D2415 Test Method for Ash in Coal Tar and Pitch

D2416 Test Method for Coking Value of Tar and Pitch (Modified Conradson)

D4296 Practice for Sampling Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Method

3.1 A test portion of the tar or pitch is heated for a specified time at 550 ± 10°C in an electric furnace. The percentage of residue is reported as the coking value.

4. Significance and Use

4.1 This test method is useful for indicating the relative coke-forming propensities and for evaluating and characterizing tars and pitches. The test method can also be used as one element in establishing the uniformity of shipments or sources of supply.

5. Apparatus

5.1 *Porcelain Crucible*, tall form, 30 mL capacity, height of 37 mm and diameter 43 mm, with lids of the overlapping type.

5.2 *Nickel Crucible*, 100 to 130 mL capacity, height of 64 mm and diameter of 60 mm, with lids.

5.3 *Wire Support*—Stainless steel wire support, fitting into the nickel crucible, used to keep the porcelain crucible (5.1) in place within the nickel crucible (5.2). This support shall allow a separation of 10 ± 1 mm between the bases of the two crucibles (see Fig. 1).

5.4 *Crucible Tray*—A suitable crucible tray to support the nickel crucibles should be made so that there is at least 7 mm of space between the bottom of the crucible and the base of the furnace.

5.5 *Electric Furnace*, capable of being controlled at 550 ± 10°C.

5.6 *Sieves*—1 mm (U.S. Standard No. 18), 300-µm (U.S. Standard No. 50) and 212–µm (U.S. Standard No. 70), conforming to Specification E11.

6. Reagents and Materials

6.1 *Calcined Petroleum Coke*—Use the fraction which passes a 1-mm mesh sieve (U.S. Standard No. 18) and is retained by a 212–µm mesh sieve (U.S. Standard No. 70).

[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.

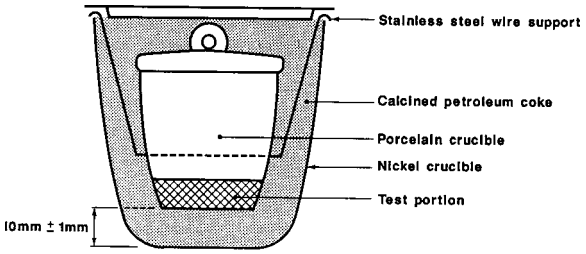

FIG. 1 Assembly of Nickel and Porcelain Crucibles

7. Bulk Sampling

7.1 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.

8. Dehydration of Sample

8.1 *Hard Pitch*—If the solid bulk sample contains free water, air-dry a representative portion.

8.2 *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 and 150°C in an open container until the surface is free from foam. Take care not to overheat, and remove heat source immediately when foam subsides.

8.3 *Tar*—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. Preparation of Working Specimen

9.1 *Hard Pitch*—If the pitch can be crushed at room temperature, prepare a 20 ± 1 g working specimen by suitable crushing, mixing, and quartering of a representative portion of the dry specimen. The crushing can be performed with a small jaw crusher and a mortar and pestle. No particle in the representative specimen should be larger than 5 mm in any dimension. Crush this specimen so that it will pass a 300 µm (U.S. Standard No. 50) mesh sieve. If the ambient temperature is high, the operation can be facilitated by chilling the sample beforehand.

9.2 *Soft Pitch and Tar*—For soft pitch and tar samples, melt the sample and take a sufficient amount of the molten mass for the test. The melting temperature must not exceed 150°C and the melting period not longer than 10 min. It is also possible to transfer the required portion of a soft pitch directly to the porcelain crucible, without preliminary treatment.

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

10. Procedure

10.1 For greater accuracy, carry out the determination in duplicate.

10.2 Heat a clean porcelain crucible with its lid in the electric furnace, controlled at 550 ± 10°C, for 2 h. After cooling to ambient temperature in a desiccator, weigh to the nearest 1 mg.

10.3 Weigh into the crucible, to the nearest 1 mg, a representative portion of 1 ± 0.05 g of the test specimen. Place the wire support in the nickel crucible and make a bed 10 ± 1 mm thick on the base of the crucible with the petroleum coke. Place the porcelain crucible containing the test portion in the wire support so that it sits on the bed of petroleum coke. Cover the porcelain crucible with its lid and completely fill the space between the two crucibles with the petroleum coke so that the porcelain crucible is completely embedded. Take care to avoid contamination of the sample by the coke. Cover the nickel crucible with its lid.

10.4 Place the prepared crucible on the crucible support and place the whole in the electric furnace controlled at 550 ± 10°C, as quickly as possible, in order to avoid heat losses.

10.4.1 There should be a space of not less than 7 mm between the nickel crucible and the floor, walls, and roof of the furnace. The space between the crucible and the door, and the back wall of the furnace should not be less than 5 cm.

10.5 After 2.5 h, remove the nickel crucible from the furnace and allow to cool for 5 to 10 min. Lift the porcelain crucible out of the coke by means of the wire support, brush and wipe free from adhering coke powder, taking care to avoid contamination. Place the crucible and contents, covered, in a desiccator and allow to cool to ambient temperature. Weigh to the nearest 1 mg.

NOTE 1—To clean the porcelain crucibles and lids, discard the residues and burn off carbonaceous matter by igniting at 700 to 1000°C in an electric furnace.

11. Calculation

11.1 Calculate the coking value of the sample as follows:

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

where:

A = mass of residue, g, and
B = mass of sample, g.

11.1.1 If the determination was carried in duplicate, report the mean value.

12. Report

12.1 Report the coking value to the nearest 0.1 mass %.

13. Precision and Bias

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

13.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 1.2 % absolute.

13.1.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determination, shall not be considered suspect unless the reported percentages differ by more than 2.3 % absolute.

13.2 *Bias*—Coking values by this test method are about 2 percentage points higher than those obtained by Test Method D2416.

14. Keywords

14.1 Alcan; coke-forming; coking value; fixed carbon; pitch; residue; tar