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Standard Test Method for Ash in Analysis of Petroleum Coke[1]

This standard is issued under the fixed designation D4422; 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 petroleum coke.

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]

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2013 Practice for Preparing Coal Samples for Analysis

3. Summary of Test Method

3.1 A representative sample of petroleum coke is dried, ground, and ashed in a muffle furnace at 700 to 750°C. The residue or ash is expressed as a percentage of the dry petroleum coke.

4. Significance and Use

4.1 The ash content is one of the properties used to evaluate petroleum coke and indicates the amount of undesirable residue present. Acceptable ash content varies with the intended use.

5. Interferences

5.1 High sulfur content of the furnace gases, regardless of the source of the sulfur, can react with an alkaline ash to produce erratic results. The furnace must be swept with air to achieve oxidation and to decrease the sulfur content of the gases.

5.2 Preparation and testing of the analysis sample must neither remove nor add ash. Improper dividing, sieving, and crushing equipment, and some muffle furnace lining material can contaminate the sample.

6. Apparatus

6.1 *Crucibles*, low wide form glazed porcelain or platinum, 30-mL capacity.

6.2 *Muffle Furnace*, with temperature control between 700 and 750°C and equipped with a means to regulate air circulation.

6.3 *Analytical Balance* capable of weighing to 0.1 mg.

6.4 *Drying Oven* controlled at 110 ± 5°C.

6.5 *Desiccator*.

7. Sample Preparation

7.1 Crush the laboratory sample to pass a 6.3-mm sieve. If the quantity exceeds 2.3 kg, divide the sample to obtain about 2.3 kg and crush this fraction to pass a 850-µm (No. 20) sieve. Further divide the sample to obtain a portion of approximately 200 g and crush to pass a 250-µm (No. 60) sieve. Divide again to obtain approximately 50 g and pulverize this fraction such that 95 % or more passes a 75-µm (No. 200) sieve. This is the analysis sample which is dried to constant weight at 110 ± 5°C.

NOTE 1—If the laboratory sample appears to be wet it must be air-dried prior to crushing to avoid caking.

NOTE 2—Recommended practice for collecting samples and the equipment and procedures for crushing and dividing are described in Practices D346 and D2013.

8. Preparation of Apparatus

8.1 The muffle furnace, when initially set up, must be tested for adequate air circulation. The air flow is adequate if replicate samples do not produce a lower ash at higher air flow rates with the same furnace loading. Maintain air flow at the same level for subsequent analyses to ensure consistency in analytical technique.

[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. Procedure

9.1 Ignite a coded crucible to constant weight at 750°C. Record the mass to 0.1 mg.

9.2 Weigh a 10 g portion of the dried analysis sample into the coded crucible. Record the mass to 0.1 mg.

9.3 Place the crucible into a cold muffle furnace that has been tested for adequate air circulation, and heat directly to above 700°C until constant mass (±0.2 mg) is obtained. Do not exceed 750°C.

9.4 Allow the furnace to cool to 150°C and transfer the crucible to the desiccator for further cooling.

9.5 Weigh the crucible and ash to the nearest 0.1 mg.

10. Calculation

10.1 Calculate the ash percent in the analysis sample as follows:

$$\text{Ash, mass \%} = \frac{A - B}{C} \times 100 \quad (1)$$

where:

A = mass of crucible and ash residue, g,
B = mass of empty crucible, g, and
C = mass of analysis sample used, g.

11. Report

11.1 Report the ash content mass percent to the third decimal (thousandth of a percent) when the average of replicate values permits.

11.2 Report the ash content mass percent to the second decimal (hundredth of a percent) when only single values are determined.

12. Precision and Bias [3]

12.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.02 \quad (2)$$

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.06 \quad (3)$$

NOTE 3—The values in the statements were determined in a cooperative program following D02-1007.[4]

12.2 *Bias*—Bias depends on conformance to the empirical conditions of the test. The ash content must not be understood to be the same as the mineral content of the petroleum coke.

13. Keywords

13.1 ash; petroleum coke

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1190.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1007.