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An American National Standard

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

This standard is issued under the fixed designation D 4422; 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 the standard. The values in parentheses are for information only.

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:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 2013 Test Method for Preparing Coal Samples for Analysis[2]

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 (1292 to 1383°F). 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 ¼ in. (6.3 mm) sieve. If the quantity exceeds 2.3 kg (5 lb), divide the sample to obtain about 2.3 kg (5 lb) 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 Practice D 346 and Test Method D 2013.

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.

9. Procedure

9.1 Ignite a coded crucible to constant weight at 750°C. Weigh 10 g ± 0.1 mg of the dried analysis sample into the coded crucible.

9.2 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.

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.05on Properties of Fuels, Petroleum Coke, and Oil Shale.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.