

This document is not an ASTM standard and is intended only to provide the user of an ASTM standard an indication of what changes have been made to the previous version. Because it may not be technically possible to adequately depict all changes accurately, ASTM recommends that users consult prior editions as appropriate. In all cases only the current version of the standard as published by ASTM is to be considered the official document.



---

**Designation: D 4422 – 94 (Reapproved 1998)**



## Standard Test Method for Ash in Analysis of Petroleum Coke<sup>1</sup>

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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke, and Oil Shale—Carbon Material.

Current edition approved Sept. 15, 1994; Nov. 1, 2003. Published November 1994; December 2003. Originally published as D 4422 – 84; approved in 1984. Last previous edition approved in 1998 as D 4422 – 894(1998).

### 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 given 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:<sup>2</sup>

- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
- ~~D-2013 Test Method for~~ 2013 Practice of 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 (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.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards*, Vol 05.05, volume information, refer to the standard's Document Summary page on the ASTM website.

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 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. Record the mass to 0.1 mg.

9.2 Weigh a 10 g ± 0.1 mg portion of the dried analysis sample into the coded ~~crucible~~.

9.2 ~~Place~~ 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.

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

9.45 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 <sup>3</sup>

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 RR:D02-1007.<sup>4</sup>

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

<sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1190.

<sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1007.

## SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D 4422–94(1998)) that may impact the use of this standard.

- (1) Updated 9.1.
- (2) Inserted new 9.2.
- (3) Revised 10.1.

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or [service@astm.org](mailto:service@astm.org) (e-mail); or through the ASTM website ([www.astm.org](http://www.astm.org)).*