



Standard Test Method for Ash in the Analysis Sample of Coal and Coke from Coal¹

This standard is issued under the fixed designation D 3174; 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 inorganic residue as ash in the analysis sample of coal or coke as prepared in accordance with Method D 2013 or Practice D 346. The results obtained can be applied as the ash in the proximate analysis, Practice D 3172, and in the ultimate analysis, Practice D 3176. For the determination of the constituents in ash, reference is made to Test Methods D 2795 and D 3682. See Terminology D 121 for definition of ash.

1.2 The values stated in SI units are to be regarded as the 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:

- D 121 Terminology of Coal and Coke²
- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²
- D 388 Classification of Coals by Rank²
- D 1756 Test Method for Determination as Carbon Dioxide of Carbonate Carbon in Coal²
- D 1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke²
- D 2013 Method of Preparing Coal Samples for Analysis²
- D 2795 Test Methods for Analysis of Coal and Coke Ash²
- D 3172 Practice for Proximate Analysis of Coal and Coke²
- D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²
- D 3176 Practice for Ultimate Analysis of Coal and Coke²
- D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases²
- D 3682 Test Method for Major and Minor Elements in Coal and Coke Ash by the Atomic Absorption Method²
- E 691 Practice for Conducting an Interlaboratory Study to

¹ This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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² *Annual Book of ASTM Standards*, Vol 05.06.

Determine the Precision of a Test Method³

3. Summary of Test Method

3.1 Ash is determined by weighing the residue remaining after burning the coal or coke under rigidly controlled conditions of sample weight, temperature, time, atmosphere, and equipment specifications.

4. Significance and Use

4.1 Ash, as determined by this test method, is the residue remaining after burning the coal and coke. Ash obtained differs in composition from the inorganic constituents present in the original coal. Incineration causes an expulsion of all water, the loss of carbon dioxide from carbonates, the conversion of iron pyrites into ferric oxide, and other chemical reactions. Ash, as determined by this test method, will differ in amount from ash produced in furnace operations and other firing systems because incineration conditions influence the chemistry and amount of the ash. References for correcting ash results determined by this test method to a mineral-matter-free basis are listed in Classification D 388, Section 8.

5. Apparatus

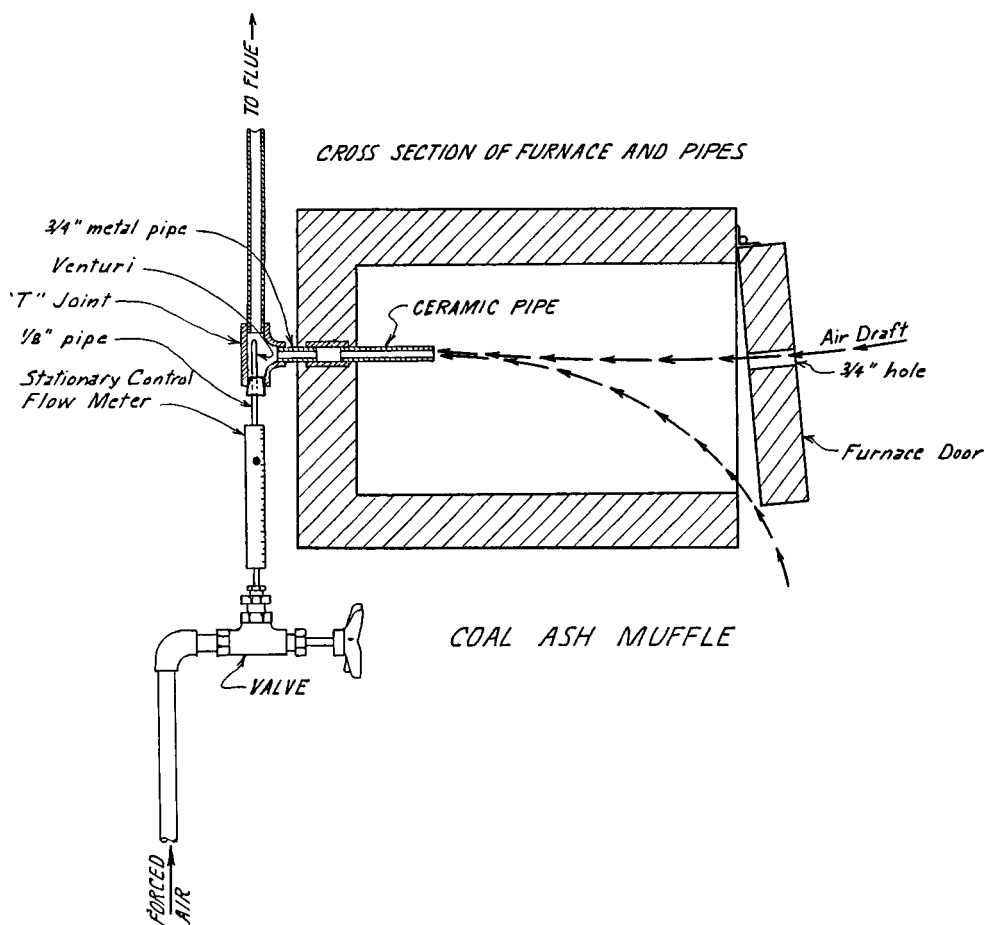
5.1 *Electric Muffle Furnace for Coal or Coke*—For determination of ash of coal, the furnace shall have an adequate air circulation and be capable of having its temperature regulated at 700 to 750°C. The furnace shall be equipped with a temperature indicator and means of controlling the temperature within prescribed limits. Means shall be provided for maintaining air flow at a rate of two to four changes per minute (see Fig. 1 and Fig. 2). Combustion gases shall be vented from laboratory. Inlet and outlet ports shall be located and arranged to distribute the air uniformly throughout the furnace area without the possibility of sweeping solid particles from the capsules. The temperature over the entire working area of the furnace floor shall be maintained within the specified temperature limits.

5.2 *Porcelain Capsules*, about 22 mm (7/8 in.) in depth, and 44 mm (1 3/4 in.) in diameter, or similar shallow dishes or platinum crucibles.

5.3 *Balance*, sensitive to 0.1 mg.

5.4 *Crucible Cover*, aluminum, porcelain, or similar covers.

³ *Annual Book of ASTM Standards*, Vol 14.02.



(Suggested method for inducing regulated air flow through ashing furnace.)

FIG. 1 Air Aspirator

6. Temperature Calibration

6.1 Place a preignited capsule with 1 g of sand at the center of the working area of the furnace, and by the use of a potentiometer and thermocouple or other suitable temperature measuring device, measure the temperature of the sand in the crucible. The crucible and sand should be at temperature equilibrium with the furnace. There should be two to four air changes per minute moving throughout the furnace (the air flow may be measured by using a wet-test meter or equivalent calibrated at standard conditions for air connected to the ceramic-pipe exhaust). Adjust the furnace temperature until the potentiometer reads $750 \pm 10^\circ\text{C}$ and then adjust or read the temperature on the indicating pyrometer. Use this reading as the proper setting for controlling the furnace.

7. Procedure

7.1 The sample shall be the material pulverized to pass No. 60 (250- μm) sieve in accordance with Method D 2013 or Practice D 346.

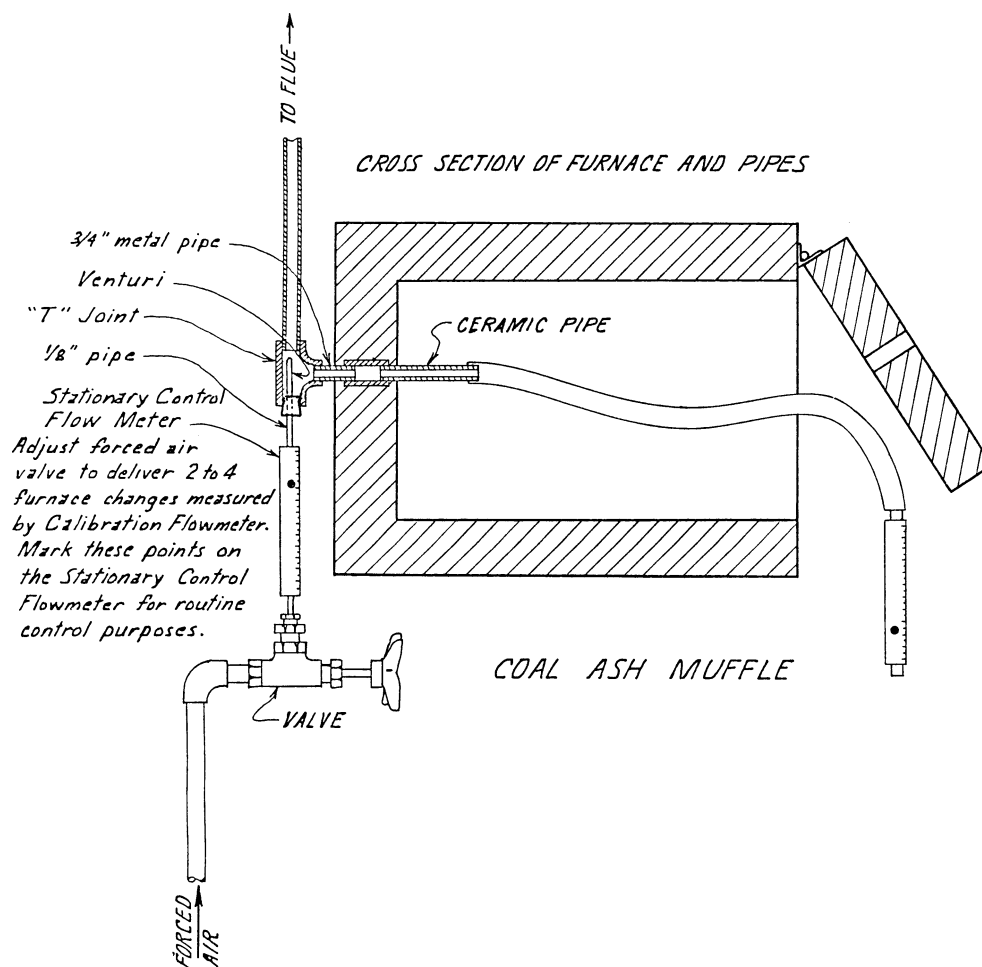
7.2 Transfer approximately 1 g (weighed to the nearest 0.1 mg) of the thoroughly mixed sample to a weighed capsule and cover quickly. An alternative way is to use the dried coal from the moisture determination in Test Method D 3173. After removing the covers, place the capsule containing the sample

in a cold furnace and heat gradually at such a rate that the temperature reaches 450 to 500°C in 1 h.

7.3 Heat coal samples so that a final temperature of 700 to 750°C is reached by the end of the second hour. Heat coke samples so that a final temperature of 950°C is reached by the end of the second hour. Continue to heat at the final temperature for additional 2 h (see Note 1). Remove the capsule from the muffle, place the cover on the capsule, cool under conditions to minimize moisture pickup, and weigh.

NOTE 1—While the 4-h incineration interval described is sufficient with most coals to reach a condition of complete burn off, certain cokes and nonreactive coals may require additional time. If unburned carbon particles are observed, or if duplicate results are suspect, the samples should be returned to the furnace for sufficient time to reach a constant weight (± 0.001 g). By this means, pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle, “two to four changes per minute,” must be assured at all times to ensure complete oxidation of the pyritic sulfur and to remove the SO_2 formed. The 4-h time limit may be reduced if the sample reaches a constant weight at 700 to 750°C in less than 4 h.

NOTE 2—Some samples may be encountered that contain a high amount of carbonates (calcite) or pyrites or both. In such cases, sulfur retained as sulfates may be both unduly high and nonuniform between duplicate samples. In such cases, sulfate sulfur in the ash can be determined in accordance with Test Methods D 1757 and the value properly corrected. If such is done, the ash value should be reported and



Calibration Flowmeter with Tubing—Ambient Air—For calibration use only, adjust forced air valve to deliver two to four furnace volume changes per minute (at standard temperature-pressure conditions.)

NOTE 1—Flowmeters are usually calibrated for one atmosphere at 70°F (760-mm Hg at 21.1°C).
 (Suggested layout for calibration.)

FIG. 2 Air Aspirator

designated both as determined and corrected.

8. Calculation

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

$$\text{Ash in analysis sample, \%} = [(A - B)/C] \times 100 \quad (1)$$

where:

- A = weight of capsule, cover, and ash residue, g,
- B = weight of empty capsule and cover, g, and
- C = weight of analysis sample used, g.

9. Report

9.1 For reporting analyses to other than as-determined basis, refer to Practice D 3180.

10. Precision and Bias

10.1 Precision

10.1.1 250 μm (No. 60) Samples—The precision of this test method for the determination of ash in the analysis sample of coal and coke is shown in Table 1. The precision characterized

TABLE 1

Range	Repeatability Limit, <i>r</i>	Reproducibility Limit, <i>R</i>
2.69 to 16.77 %	0.22	0.32

by repeatability (S_r , r) and reproducibility (S_R , R) is described in Table A1.1 in Annex A1.

10.1.1.1 Repeatability Limit (r)—The value below which the absolute difference between two test results calculated to a dry basis (Practice D 3180) of separate and consecutive test determinations, carried out on the same sample of 250 μm (No. 60) coal and coke in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

10.1.1.2 Reproducibility Limit (R)—The value below which the absolute difference between two test results calculated to a dry basis (Practice D 3180), carried out in different laboratories, using samples of 250 μm (No. 60) coal and coke taken at

random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

10.1.2 *36 μm (No. 8) Samples*—The precision of this test method for the determination of ash in the analysis sample of coal and coke is shown in Table 2 and Table 3.

10.1.2.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D 3180) of separate and consecutive test determinations, carried out on the same sample, using the same riffle, determined on a single test specimen of two separate 2.36 μm (No. 8) test units of coal reduced entirely to 250 μm (No. 60) and prepared from the same bulk sample coal in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

TABLE 2

Coal	Range	Repeatability Limit, <i>r</i>
Bituminous	5.0 to 15.0 %	0.30 %
Subbituminous-Lignite	4.5 to 30.0 %	0.33 %

TABLE 3

Coal	Range	Reproducibility Limit, <i>R</i>
Bituminous	5.0 to 15.0 %	0.49 %
Subbituminous-Lignite	4.5 to 30.0 %	0.47 %

10.1.2.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D 3180), carried out in different laboratories, using samples of 2.36 μm (No. 8) coal reduced entirely to 250 μm (No. 60), taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

10.1.2.3 An interlaboratory study, designed consistent with Practice E 691, was conducted in 1989. Eight laboratories participated in this study.⁴

10.2 *Bias*—Since this is an empirical test method, the degree of absolute bias cannot be determined.

11. Keywords

11.1 ash; coal; coke

⁴ Supporting data are available from ASTM Headquarters. Request Report RR: D05-1015.

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS 250 μm (No. 60) SAMPLES

A1.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A1.1.

A1.2 *Repeatability Standard Deviation (S_r)*—The standard deviation of test results obtained under repeatability conditions.

A1.3 *Reproducibility Standard Deviation (S_R)*—The standard deviation of test results obtained under reproducibility conditions.

TABLE A1.1 Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters Used for Calculation of Precision Statement

Material	Average	S_r	S_R	<i>r</i>	<i>R</i>
91-2 lvb	6.86364	0.049024	0.072788	0.13717	0.20366
91-1 hvAb	2.68068	0.047123	0.055633	0.13185	0.15566
91-5 hvAb	10.0873	0.065132	0.080675	0.18224	0.22573
89-4 hvCb	10.475	0.075743	0.121383	0.21193	0.33963
91-4 hvCb	12.515	0.071347	0.082831	0.19963	0.23176
90-1 subB	16.7593	0.07752	0.1306	0.2169	0.36542
89-7 subA	8.81705	0.06302	0.10659	0.17633	0.29824
91-6 subA	11.0723	0.061279	0.083981	0.17146	0.23498
89-6 subC	13.5182	0.127066	0.194514	0.35553	0.54425
Lignite	17.865	0.103367	0.163095	0.28922	0.45634

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