



Designation: D 3174 – 002

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

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¹ This test method is under the jurisdiction of ASTM Committee ~~D-5~~ D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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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.*

~~1.3 The values stated in SI units are to be regarded as the standard.~~

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 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

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

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

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.

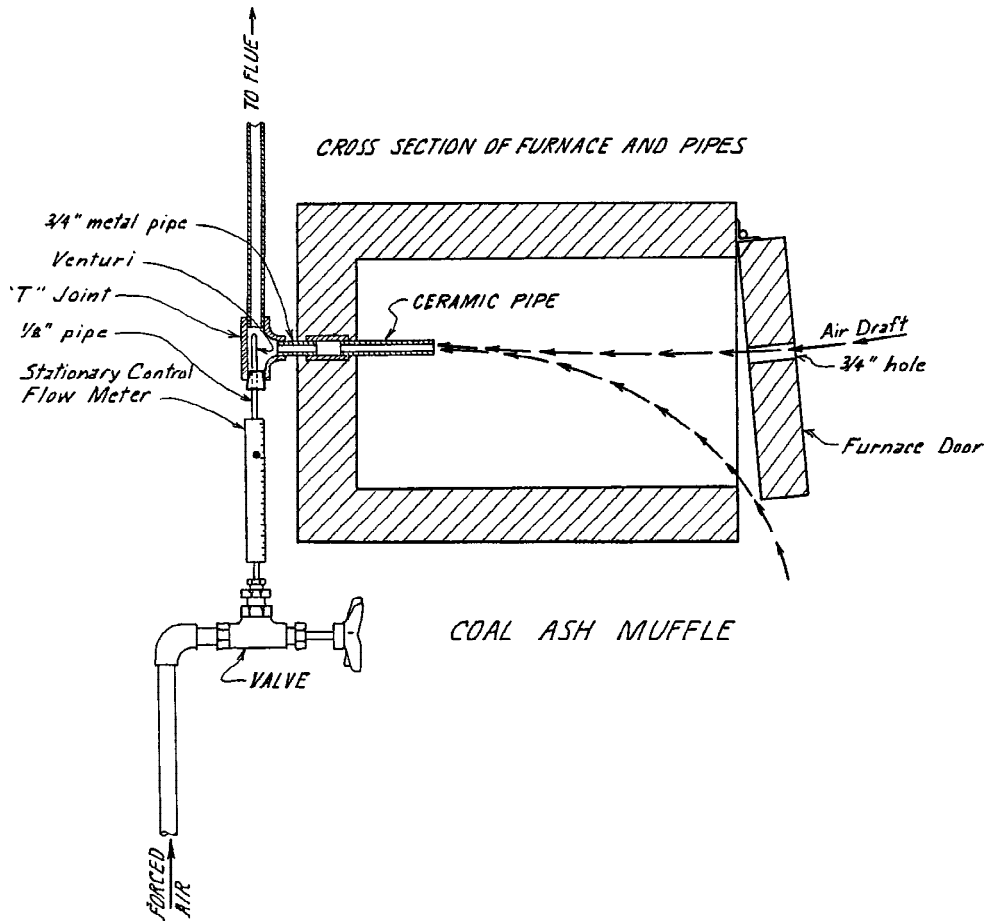
5.4 *Crucible Cover*, aluminum, porcelain, or similar covers.

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 ± 10°C and then adjust or read the temperature on the indicating pyrometer. Use this reading as the proper setting for controlling the furnace.

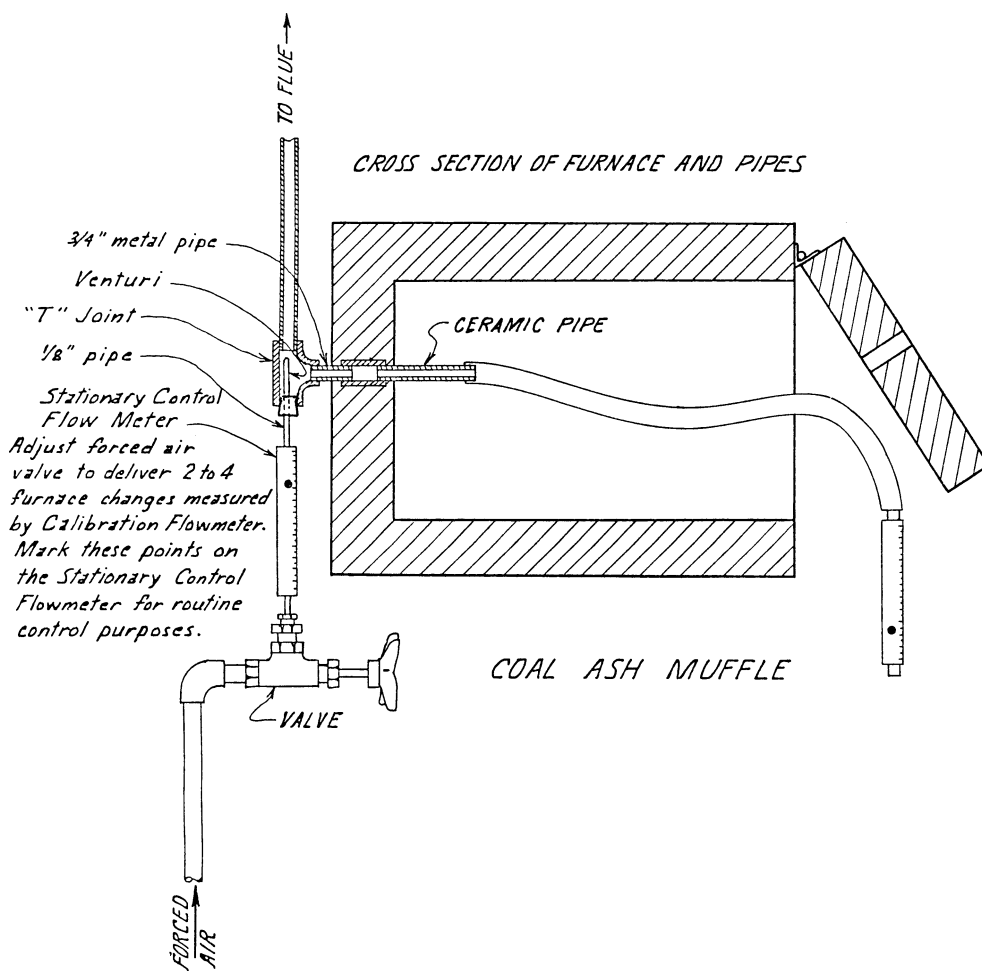
7. Procedure

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.



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

FIG. 1 Air Aspirator



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

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 coals 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 SO₂ 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 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 ; g_1
- B = weight of empty capsule and cover, g ; g_2 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 250- μm (No. 60) Samples*—The relative precision of this test method for the determination of ash covers the range from 2.68 to 17.86 %.

10.1.1 *Repeatability 250 μm (No. 60) Samples*—The difference 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 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 results, 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, should not exceed the repeatability interval (limit) $I(r)$ more than 5 % of such paired values (95 % confidence level). When such apparatus on samples taken at random from a difference is found single quantity of homogeneous material, may be expected to exceed the repeatability interval (limit), there is reason to question one or both 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. The repeatability limit for this test method on 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 0.22 (see Note 3), as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

10.1.2 *Reproducibility 36 μm (No. 8) Samples*—The difference 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 replicate separate and consecutive test determinations, carried out in different laboratories on representative samples 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 after coal in the last stage of reduction, should not exceed same laboratory, by the reproducibility interval (limit) $I(R)$ more than 5 % same operator, using the same apparatus on samples taken at random from a single quantity of such paired values (95 % confidence level). When such difference is found homogeneous material, may be expected to exceed the reproducibility interval (limit), there is reason to question one or both occur with a probability of approximately 95 %.

10.1.2.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results. The reproducibility limit for this method on results calculated to a dry basis is 0.32 (see Note 3).

NOTE 3—These limits apply to the relative spread (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 measurement single quantity of material that is expressed as a percentage homogeneous as derived from possible, may be expected to occur with a statistical evaluation probability of approximately 95 %.

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

10.2 *Precision 2.36-mm (No. 8) Samples*—The relative precision of this test method for the determination of ash covers the concentration range from 5.0 to 15.0 % for bituminous coals and 4.5 to 30.0 % for subbituminous and lignite coals.

10.2.1 *Repeatability*—The difference in absolute value between test results obtained in the same laboratory, by the same operator, using the same apparatus, determined on a single test specimen of two separate 2.36-mm (No. 8) test units of coal reduced entirely to 250- μm (No. 60) and prepared from the same bulk sample using the same apparatus should not exceed the repeatability interval for more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one, or both, of the test results. The repeatability interval for this test method is:

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

TABLE 1

| Range | Repeatability Limit, r | Reproducibility Limit, R |
|-----------------|--------------------------------|----------------------------------|
| 2.69 to 16.77 % | 0.22 | 0.32 |

TABLE 2

| Coal | Range | Repeatability Limit, r |
|-----------------------|---------------|-----------------------------|
| Bituminous | 5.0 to 15.0 % | 0.30 % |
| Subbituminous-Lignite | 4.5 to 30.0 % | 0.33 % |

TABLE 3

| Coal | Range | Reproducibility Limit, R |
|-----------------------|---------------|-------------------------------|
| Bituminous | 5.0 to 15.0 % | 0.49 % |
| Subbituminous-Lignite | 4.5 to 30.0 % | 0.47 % |

— Bituminous coals 0.30 %
 — Subbituminous and lignite coals 0.33 %

10.2.2 Reproducibility—The difference in absolute value between test results obtained in different laboratories, calculated as the average of determinations on single test specimens of two separate 2.36-mm (No. 8) test units of coal reduced entirely to 250- μ m (No. 60) and prepared from the same bulk sample, should not exceed the reproducibility interval for more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one, or both, of the test results. The reproducibility interval for this test method is:

— Bituminous coals 0.49 %
 — Subbituminous and lignite coals 0.47 %

NOTE 4—Supporting data for 2.36-mm (No. 8) coal has been filed at ASTM Headquarters and may be obtained by requisition RR:D05-1015.

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

11. Keywords

11.1 ash; coal; coke

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS 250 μ m (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 | r | R |
|-----------|---------|----------|----------|---------|---------|
| 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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