Designation: D3174 – 12 (Reapproved 2018)$^{\varepsilon 1}$

Standard Test Method for
Ash in the Analysis Sample of Coal and Coke from Coal[1]

This standard is issued under the fixed designation D3174; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

ε^1 NOTE—An editorial correction was made to 8.1 in September 2020.

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 Practice D2013 or Practice D346. The results obtained can be applied as the ash in the proximate analysis, Practice D3172, and in the ultimate analysis, Practice D3176. For the determination of the constituents in ash, reference is made to Test Methods D3682, D4326, and D6349. Test Methods D6357 should be used to prepare ash to be used for trace element analysis. See Terminology D121 for definition of ash.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D388 Classification of Coals by Rank
D1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke (Withdrawn 2009)[3]
D2013 Practice for Preparing Coal Samples for Analysis
D2795 Test Methods for Analysis of Coal and Coke Ash (Withdrawn 2001)[3]
D3172 Practice for Proximate Analysis of Coal and Coke
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3176 Practice for Ultimate Analysis of Coal and Coke
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes
D4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence
D5016 Test Method for Total Sulfur in Coal and Coke Combustion Residues Using a High-Temperature Tube Furnace Combustion Method with Infrared Absorption
D6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry
D6357 Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Furnace Atomic Absorption Spectro
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D121.

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[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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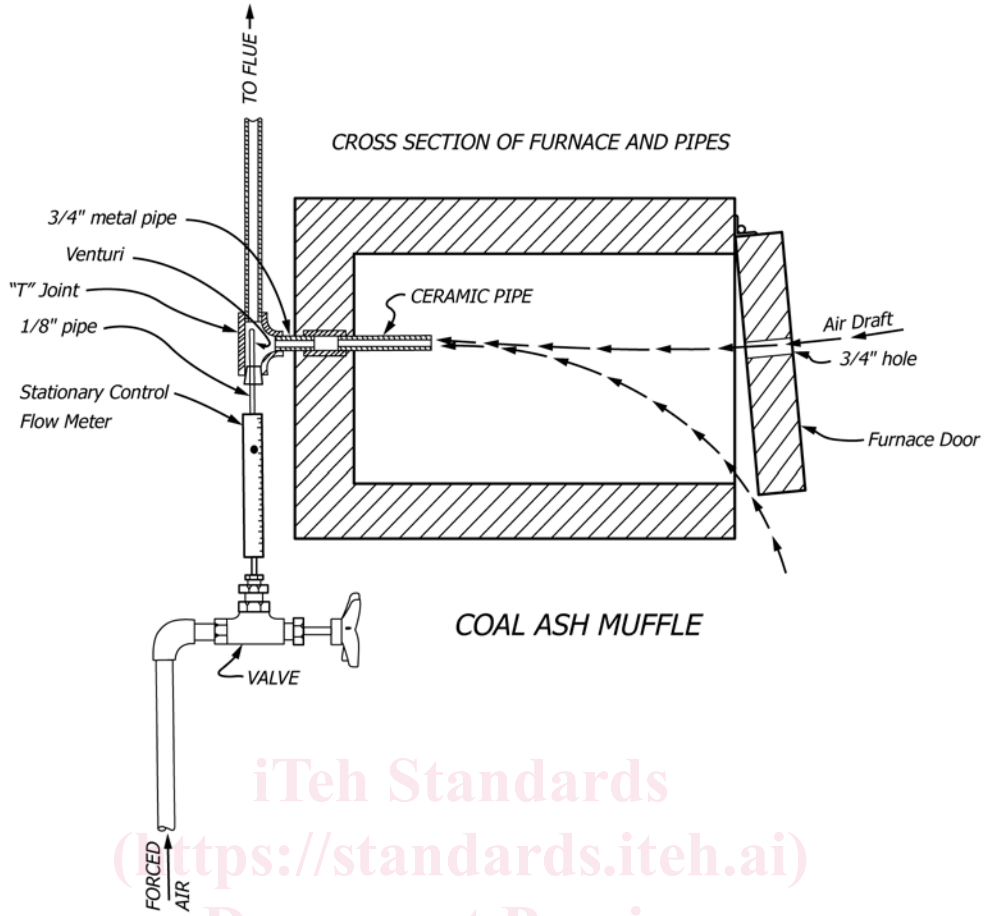
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.



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

FIG. 1 Air Aspirator

4. Summary of Test Method

4.1 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.

5. Significance and Use

5.1 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 D388, Section 9.

6. Apparatus

6.1 *Electric Muffle Furnace for Coal or Coke*—For determination of ash of coal and coke, the furnace shall have an adequate air circulation and be capable of having its temperature regulated at 500 °C, 750 °C, and 950 °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.

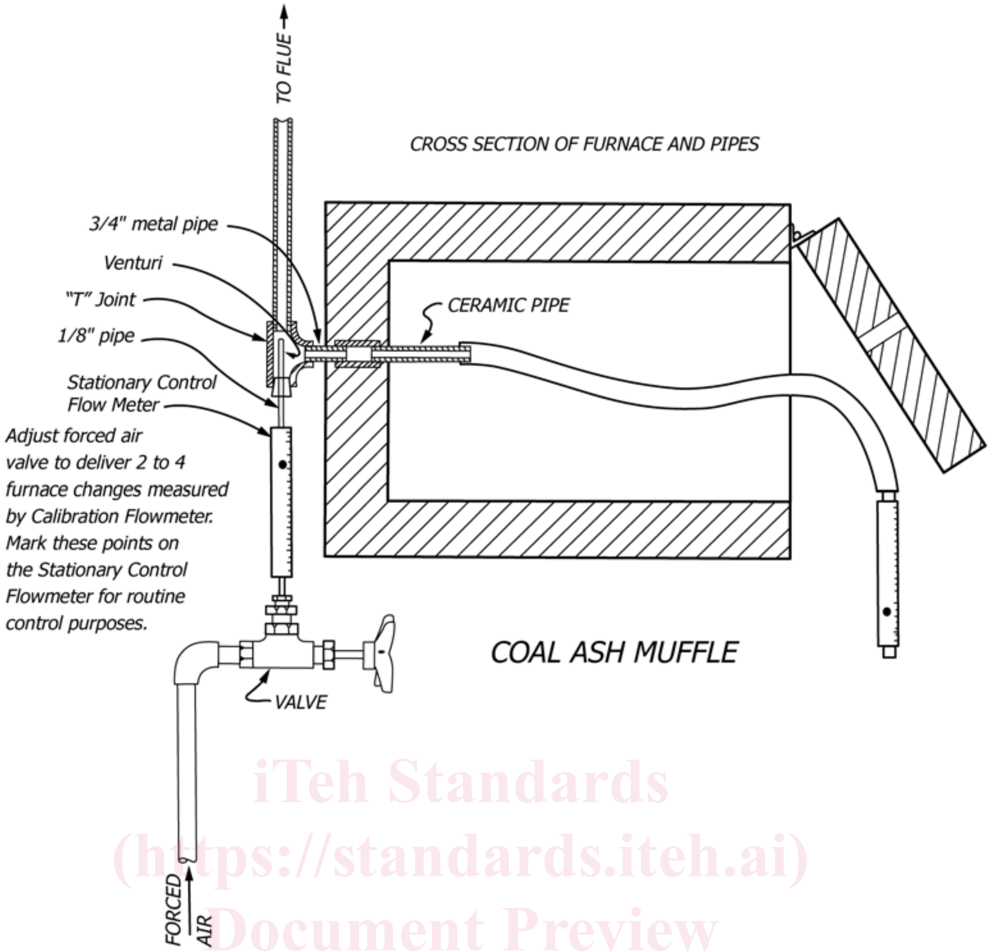
6.2 *Porcelain Capsules,* about 22 mm (⅞ in.) in depth, and 44 mm (1¾ in.) in diameter, or similar shallow dishes or platinum crucibles.

6.3 *Balance,* sensitive to 0.1 mg.

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

7. Temperature Calibration

7.1 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



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

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

8. Procedure

8.1 The sample shall be the material pulverized to pass No. 60 (250 µm) sieve in accordance with Practice D2013 or Practice D346.

8.2 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 D3173. 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 500 °C ± 10 °C at the end of 1 h.

8.3 For coals continue heating the sample until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. For cokes continue heating the sample until the temperature rises from 500 °C ± 10 °C to 950 °C ± 20 °C at the end of 1 h. Continue to heat at the final temperature (750 °C or 950 °C) for an additional 2 h. Remove the capsule from the muffle furnace, place the cover on the capsule, cool under conditions to minimize moisture pickup, and weigh.

8.4 The two-stage ashing procedure allows pyritic sulfur to be oxidized and expelled before most metal carbonates are decomposed. An ample supply of air in the muffle furnace, "two to four volume changes per minute," must be assured at all times to ensure complete oxidation of the pyritic sulfur and to remove the SO_2 formed.

8.5 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