Designation: D7348 – 13

Standard Test Methods for Loss on Ignition (LOI) of Solid Combustion Residues[1]

This standard is issued under the fixed designation D7348; 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 These test methods cover the determination of the mass loss from solid combustion residues upon heating in an air or oxygen atmosphere to a prescribed temperature. The mass loss can be due to the loss of moisture, carbon, sulfur, and so forth, from the decomposition or combustion of the residue.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*[2]

D121 Terminology of Coal and Coke

D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal

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

D3683 Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption

D4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence

D6316 Test Method for Determination of Total, Combustible and Carbonate Carbon in Solid Residues from Coal and Coke

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 Ab

D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D121.

4. Summary of Test Method

4.1 Loss on ignition (LOI) is determined by measuring the loss in mass of the test specimen when heated under controlled conditions of temperature, time, atmosphere, specimen mass, and equipment specifications. The LOI can be determined by measuring the mass loss in a single procedure or in a two-step procedure in which mass losses, equivalent to the moisture and ash values of the test specimen, are determined.

5. Significance and Use

5.1 LOI refers to the mass loss of a combustion residue whenever it is heated in an air or oxygen atmosphere to high temperatures. In the cement industry, use of the term LOI normally refers to a mass loss in a sample heated to 950°C. To combustion engineers, the term LOI normally refers to mass losses in samples heated to temperatures normally less than 950°C. These test methods establish a procedure for determining LOI values for combustion residues heated to 750°C or 950°C. LOI values from these test methods can be used by industries that utilize combustion residues in various processes and products.

5.2 If the solid combustion residue is heated to estimate the combustible or unburned carbon in the sample, it has been shown that LOI and estimation of unburned carbon do not necessarily agree well with each other and that LOI should not be used as an estimate of unburned carbon in all combustion

[1] These test methods are under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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

residues.[3]. Direct determination of unburned (combustible) carbon can be carried out using Test Method D6316.

5.3 If the solid combustion residue is heated to prepare an ash for the determination of the concentrations of major and minor elements, use the heating procedure described in Test Methods D3682, D4326, and D6349, or the procedures for the 750°C LOI determination described in these test methods (Method A).

5.4 If the solid combustion residue is heated to prepare an ash for the determination of the concentrations of trace elements, use the heating procedure described in Test Methods D3683 and D6357.

NOTE 1—Combustion residues produced in furnace operations or other combustion systems can differ from the ash yield, as determined in Test Methods D3174 and D7582, because combustion conditions influence the chemistry and amount of ash. Combustion causes an expulsion of all water, the loss of carbon dioxide from carbonates, the conversion of metal sulfides into metal oxides, metal sulfates and sulfur oxides, and other chemical reactions. Likewise, the "ash" obtained after igniting combustion residues can differ in composition and amount from Test Methods D3174 and D7582 ash yields because of different heating procedures, combustion of unburned carbon, and decomposition of materials in the residue.

6. Interferences

6.1 There are no known interferences for these test methods.

7. Apparatus

7.1 *Furnace*—The apparatus shall consist of a furnace with a cavity large enough to accept multiple crucibles. The furnace shall be constructed so the cavity is surrounded by a suitable refractory and insulated so as to develop a uniform temperature in all parts of the cavity but with a minimum free space. The furnace shall be capable of being heated rapidly (10°C/min or faster) from ambient to 950°C. The temperature shall be monitored and maintained at values specific to each of the determinations. Provisions shall be made to introduce drying and oxidizing gases and to remove products of drying, decomposition, and combustion. A recommended flow rate is one furnace volume change per minute, but higher flow rates (that is, two furnace volumes per minute as in some other standard test methods for coal and coke) are acceptable. The furnace can be a stand alone muffle furnace or a computer-controlled macrothermogravimetric analyzer (macro TGA) system. In macro TGA, a sample size of 1 g (or larger) is used. In a typical analysis, the temperature is ramped from ambient to a specific temperature and held at that temperature for a prescribed length of time. In thermogravimetric analysis, the mass of a sample in a controlled atmosphere is recorded repeatedly as a function of temperature or time.

7.2 *Drying Oven*—For determining the moisture in solid combustion residue samples, use a drying oven with openings for drying gas circulation and capable of temperature regulation between the limits of 104 and 110°C. A drying gas flow rate of approximately one volume change per minute is recommended but higher flow rates, that is, two volume changes per minute as in some other standard test methods for coal and coke, are acceptable.

7.3 *Crucibles,* use a crucible of a convenient form that allows extensive contact between the specimen and reactant gas. The crucibles can be made of porcelain, fused silica, or similar materials. The crucibles shall have the dimensions specified by the instrument manufacturer.

7.4 *Balance,* sensitive to 0.1 mg. In the macro TGA, the balance is an integral part of the system. For other systems, the balance is a separate piece of apparatus.

7.5 Operation of the instrumental system in its entirety shall be verified in accordance with the manufacturer's operating instructions.

7.6 *Venting Equipment*—Combustion and decomposition gases evolved during the test procedures shall be vented from the laboratory and suitable venting equipment shall be installed in the vicinity of the apparatus.

8. Reagents and Materials

8.1 *Drying Gases*—Air dried to a moisture content of 1.9 mg/L or less (dew point –10°C or less). Nitrogen (99.5 % purity) is normally used with the macro TGA system. Argon can also be used.

8.2 *Oxidizing Gases*—Oxygen (99.5 % purity) or air.

9. Hazards

9.1 The user shall insure acceptable documented safety procedures are in place for the handling of all reagents and test materials and for the operation of laboratory equipment specified for these test methods.

10. Sampling, Test Specimens, and Test Units

10.1 The sample used for analysis shall be thoroughly mixed and of such fineness to pass through a 250-µm (No. 60) sieve. Pulverizing the sample to this fineness is required.

11. Preparation of Apparatus

11.1 For LOI determinations using a macro TGA, follow the manufacturer's recommended procedure for verifying system stability and for loading and taring the crucibles. Various modes of operation are possible depending on the instrument used and the manner in which the determinations are completed. The instrument can be programmed to terminate the test whenever the test specimens and crucibles have reached a constant mass. Typically, crucibles are weighed automatically at specified intervals, and the analysis is complete whenever three successive weighings agree within a plateau deviation specified for the instrument. Constant mass is defined as a point where the mass change is ≤ 0.05 % of a nine-minute period, either by using three successive weighings (for some TGAs) or a fixed nine-minute period (for some TGAs). This mass change of 0.05% is equivalent to 0.0005 g for a 1.0000 g sample. Alternately, the instrument can be programmed to allow for moisture determination by heating the test specimens for a specified time period (for example, 1 h) at the prescribed temperature limits.

[3] Burris S.C. , Li, D., and Riley J.T, "Comparison of Heating Losses and Macro Thermogravimetric Analysis Procedures for Estimating Unburned Carbon in Combustion Residues," *Energy Fuels* Vol 19 2005, pp. 1493-1502.