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.

Designation: D7348 – 21

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 *Units*—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
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D3683 Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption (Withdrawn 2018)[3]
D4326 Test Method for Major and Minor Elements in Coal 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 Absorption Spectro
D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
D8146 Guide for Evaluating Test Method Capability and Fitness for Use
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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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

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[1] These test methods are under the jurisdiction of ASTM Committee D05 on Coal and Coke and are 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 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.

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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 residues.[4] 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 mass fractions 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 mass fractions 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 macro thermogravimetric 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 °C 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, the mass of a crucible is determined automatically at specified intervals, and the analysis is complete whenever three successive mass determinations agree

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[4] 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.

within a plateau deviation specified for the instrument. Constant mass is defined as a point where the mass change is ≤ 0.05 % of a 9 min period, either by using three successive mass determinations (for some TGAs) or a fixed 9 min 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.

11.2 When using a muffle furnace for LOI determinations, always start the test with the muffle furnace at ambient temperature.

12. Conditioning

12.1 Heat new crucibles for use in these test methods under the conditions of the test and cool before use.

13. Procedure

13.1 In these procedures Method A refers to LOI determinations at 750 °C whereas Method B refers to LOI determinations at 950 °C.

13.2 For LOI determinations using a macro TGA, the analyses are normally complete when the sample reaches a constant mass as defined in the instrumental operating parameters. (See 11.1.)

13.3 For LOI determinations using a single-step procedure, add approximately 1 g of solid combustion residue to each successive crucible and determine the mass. Select oxygen or air as the furnace atmosphere and gradually raise the temperature of the furnace at a rate such that the furnace temperature reaches 500 °C ± 10 °C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 950 °C ± 20 °C at the end of 1 h. Maintain the higher temperature until the combustion residue test specimens reach a constant mass or for an additional 2 h.

13.4 For LOI determinations using a two-step procedure, add approximately 1 g of solid combustion residue to each successive crucible and determine the mass. For moisture determinations with the macro TGA, turn on the drying gas (see 8.1) and heat the test specimens of known mass in crucibles without covers at 104 °C to 110 °C. A recommended flow rate is one furnace volume change per minute, but higher flow rates (that is, two furnace volumes per minute, as in other standard test methods for coal and coke) are acceptable. Ash determinations on the residues (dried test specimens) from the moisture determination are made by changing the macro TGA furnace atmosphere to oxidizing gas (see 8.1), and gradually raising the temperature of the furnace at a rate such that the furnace temperature reaches 500 °C ± 10 °C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 950 °C ± 20 °C at the end of 1 h. Maintain the higher temperature until the combustion residue test specimens reach a constant mass or for an additional 2 h.

13.5 LOI can be determined in a single-step procedure using a muffle furnace (see 7.1). Place approximately 1 g of combustion residue into a crucible of known mass and determine the mass of the test specimen to the nearest 0.1 mg. Place the crucible with the test specimen, without a cover, into the cold furnace. Turn on the oxidizing gas (see 8.2) and adjust the flow to approximately one furnace volume change per minute. Gradually raise the temperature of the furnace at a rate such that the furnace temperature reaches 500 °C ± 10 °C at the end of 1 h. For Method A, continue the gradual heating of the samples until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 950 °C ± 20 °C at the end of 1 h. Maintain the higher temperature until the combustion residue test specimens reach a constant mass or for an additional 2 h.

13.6 LOI can be determined in a two-step procedure using a muffle furnace and a drying oven (see 7.2). To determine moisture, place approximately 1 g of combustion residue into a crucible of known mass and determine the mass of the test specimen to the nearest 0.1 mg. Place the crucible with the test specimen, without a cover, into the preheated drying oven (104 °C to 110 °C) through which passes a current of preheated drying gas (see 8.1). Close the oven and heat for 1 h. Remove the test specimen crucibles, cover immediately, allow to cool to ambient temperature in a desiccator, and determine the mass. (Additional heatings and mass determinations may be necessary for some solid combustion residues if the 1 h time period is insufficient to bring the test specimen to a constant mass.) For ash determination, place the crucible, without covers, with the dry test specimen in a cold furnace. Gradually raise the temperature of the furnace at a rate such that it reaches 500 °C ± 10 °C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 950 °C ± 20 °C at the end of 1 h. Maintain the higher temperature until the combustion residue test specimens reach a constant mass or for an additional 2 h.

13.7 If the solid combustion residue is heated to prepare an ash for the determination of the mass fractions of major and minor elements, use the heating procedure described in Test Methods D3682, D4326, and D6349, or the 750 °C procedure (Method A) described previously.

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

14. Calculation of Results

14.1 With a computer-controlled macro TGA, the computer is normally programmed to perform calculations automatically. The equations used in the calculations are as listed in the following sections.

14.2 Calculate the LOI expressed as a percentage from the single-step procedure as follows:

$$LOI = [(W - B)/W] \times 100 \quad (1)$$

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TABLE 1 Mass Fraction Range and Limits of Repeatability (r), Reproducibility (R), and Precision Ratio (PR) for LOI (750°C and 950°C) Test Methods

	Mass Fraction Range, Percent	Repeatability Limit, r	Reproducibility Limit, R	Precision Ratio, (PR)
		750 °C		
Single-step Procedure				
Macro-TGA	1.1 to 11.7	0.16	0.23	1.43
Muffle Furnace	1.0 to 11.0	0.19	0.26	1.35
Two-Step Procedure (dry basis)				
Macro-TGA	2.2 to 11.3	0.18	0.26	1.55
Muffle Furnace	0.9 to 11.4	0.26	0.45	1.82
		950 °C		
Single-step Procedure				
Macro-TGA	1.1 to 11.7	0.21	0.30	1.42
Muffle Furnace	2.6 to 11.8	0.39	0.58	1.62
Two-Step Procedure				
Macro-TGA	1.0 to 11.4	0.16	0.24	1.45
Muffle Furnace	2.4 to 11.4	0.27	0.50	1.84

Where:

W = mass of test specimen used, g,
B = mass of test specimen after heating at 750 °C or 950 °C, g, and
100 = conversion factor from dimensionless mass fraction to percent, %.

14.3 Calculate the LOI expressed as a percentage from the two-step procedure as follows:

$$M = [(W - C)/W] \times 100 \quad (2)$$

$$Ash = [D/W] \times 100 \quad (3)$$

$$LOI = [(C - D)/W] \times 100 \quad (4)$$

Where:

M = percent moisture as determined in the test specimen;
W = mass of test specimen used, g;
C = mass of test specimen after drying in moisture test, g;
D = mass of ash residue after heating at 750 °C or 950 °C, g; and
LOI = percent loss on ignition as determined in the test specimen.

15. Report

15.1 Report the temperature or method used, or both, to determine the LOI vales. For reporting analyses to other than the as-determined basis, refer to Practice D3180.

16. Precision and Bias

16.1 *Precision*—The precision data of these test methods for the determination of LOI for combustion residues are shown in Table 1. The precision characterized by the repeatability (S_r, r) and reproducibility (S_R, R), analytical performance value (APV_r), and precision ratio (PR) is described in Tables A1.1-A1.4 in Annex A1.

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

16.1.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results, carried out in different laboratories using samples taken at random from a single quantity of 250 µm (No. 60 USA Standard sieve) material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

16.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

16.2 *Scope Limits for a Test Method*—Guide D8146 offers techniques for evaluating the statistical capability and fitness for use in standard test methods. Guide D8146 defines the analytical performance value (APV_r) as a metric that assesses the precision of a test method relative to applicable specification limits. APV_r is the ratio of the repeatability (r) of a test method to specific limits, expressed as a percent. Guide D8146, subsection 5.3.2, states that it is generally expected that the analytical performance value, or APV_r, should be less than 28 % for a capable test method at its lowest limit. Since the repeatability, r, is equal to 2.8 x S_r, this is equivalent to saying that the ratio of the within laboratory standard deviation (S_r) to a sample test level, expressed as a percentage, should be less than 10 %.

16.2.1 The lower scope limit of a test method is the larger of [lowest sample mean tested in the Interlaboratory Study (ILS)] or [the test level where the ratio of the repeatability, r, of a test sample to the test sample value is 28 %.]. The lower scope limits for all of the LOI test samples reported in Tables A1.1-A1.4 had APV_r values less than 28 %.

16.2.2 Additionally, the upper scope limit of a test method is set to a value that does not exceed the parameter value of the test material with the highest value used in the ILS. The upper

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