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.

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 weigh. 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 ± 10°C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 ± 10°C to 750 ± 15°C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 ± 10°C to 950 ± 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 weigh. For moisture determinations with the macro TGA, turn on the drying gas (see 8.1) and heat the weighed test specimens in crucibles without covers at 104 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 ± 10°C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 ± 10°C to 750 ± 15°C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 ± 10°C to 950 ± 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 preweighed crucible and weigh 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 ± 10°C at the end of 1 h. For Method A, continue the gradual heating of the samples until the temperature rises from 500 ± 10°C to 750 ± 15°C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 ± 10°C to 950 ± 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 preweighed crucible and weigh 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 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 weigh. (Additional heatings and weighings may be necessary for some solid combustion residues if the 1-h time period is insufficient to bring the test specimen to a constant weight.) 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 ± 10°C at the end of 1 h. For Method A, continue the gradual heating until the temperature rises from 500 ± 10°C to 750 ± 15°C at the end of 1 h. For Method B, continue the gradual heating until the temperature rises from 500 ± 10°C to 950 ± 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 concentrations of major and minor elements, use the heating procedure described in Test Methods D3682 (7.1), D4326 (7.1), and D6349 (9.2), 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 concentrations of trace elements, use the heating procedure described in Test Methods D3683 (9.1) and D6357 (9.1).

## 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 percentage from the single-step procedure as follows:

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

*Where:*

$W$ = mass of test specimen used, g, and
$B$ = mass of test specimen after heating at 750°C or 950°C, g.

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

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

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

**TABLE 1 Concentration Range and Limits of Repeatability and Reproducibility for LOI (750°C and 950°C) Test Methods**

| | Concentration Range, Percent | Repeatability Limit, r | Reproducibility Limit, R |
|---|---|---|---|
| | 750°C | | |
| **Single-step Procedure** | | | |
| Macro-TGA | 1.1–11.7 | 0.29 | 0.37 |
| Muffle Furnace | 1.0–11.0 | 0.22 | 0.28 |
| **Two-Step Procedure**(dry basis) | | | |
| Macro-TGA | 0.97–11.3 | 0.19 | 0.27 |
| Muffle Furnace | 0.9–11.4 | 0.37 | 0.52 |
| | 950°C | | |
| **Single-step Procedure** | | | |
| Macro-TGA | 1.1-11.7 | 0.21 | 0.28 |
| Muffle Furnace | 1.1-11.8 | 0.47 | 0.75 |
| **Two-Step Procedure** | | | |
| Macro-TGA | 1.0-11.4 | 0.17 | 0.22 |
| Muffle Furnace | 0.9-11.5 | 0.36 | 0.71 |

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

*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 method 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) 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 %.

16.2 *Bias*—A bias statement is not available because a suitable Certified Reference Material with a certified value for the LOI is not currently available. NIST Standard Reference Material 1880a, Portland Cement and NIST Standard Reference Material 2696, Silica Fume provide a reference value for the LOI, was included in the interlaboratory study with the results shown in Table 2.

16.3 An interlaboratory study, designed consistent with Practice E691, was conducted in 2006. Twenty labs participated. The details of the study and supporting data are given in Research Reports RR:D05-1031 and RR:D05-1036.[4]

## 17. Keywords

17.1 ash; combustion residues; loss on ignition; LOI; macro thermogravimetric analysis; macro TGA

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1031.

Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1036.

**TABLE 2 Comparison of the Percent LOI (750°C and 950°C) Value (Reference and Not Certified) for NIST 1880a and 2696 with the Interlaboratory Study Values**

| | 750°C | | |
|---|---|---|---|
| | NIST 2696 Reference Value | Macro TGA Dry | Muffle Furnace Dry |
| % LOI (dry) | 2.12 ± 0.10 | 2.25 | 2.12 |
| Bias | | 0.14 | 0.00 |
| | 950°C | | |
| | NIST 1880a Reference Value | Macro TGA Two-step | Muffle Furnace Two-step |
| % LOI (as-received) | 1.320 | 1.345 | 1.084 |
| Bias | | 0.025 | −0.236 |

# ANNEX

## (Mandatory Information)

## A1. PRECISION STATISTICS

A1.1 The precision of these test methods, characterized by repeatability (Sr, r) and reproducibility ($S_R$, R) has been determined for the following materials as listed in Tables A1.1-A1.4.

**TABLE A1.1 Repeatability ($S_r$, r) and Reproducibility ($S_R$, R) Parameters Used for Calculation of Precision Statement for Macro-TGA, One-Step Procedure for LOI (750°C and 950°C)**

| Material | Average % LOI | $S_r$ | $S_R$ | r | R |
|---|---|---|---|---|---|
| | | 750°C | | | |
| EBIT | 11.7025 | 0.1054 | 0.1186 | 0.2952 | 0.3321 |
| NDL | 2.4303 | 0.0958 | 0.1082 | 0.2682 | 0.3029 |
| BB | 2.495 | 0.0467 | 0.0624 | 0.1306 | 0.1748 |
| CB | 9.8156 | 0.0706 | 0.093 | 0.1977 | 0.2605 |
| TXL | 4.0441 | 0.1604 | 0.1708 | 0.4491 | 0.4782 |
| BDPS | 1.0722 | 0.038 | 0.0645 | 0.1064 | 0.1807 |
| SHND | 4.8909 | 0.0852 | 0.096 | 0.2384 | 0.2688 |
| 7FA | 4.4325 | 0.1477 | 0.1874 | 0.4135 | 0.5248 |
| 8FA | 11.0131 | 0.118 | 0.2103 | 0.3303 | 0.5887 |
| | | 950°C | | | |
| EBIT | 11.6509 | 0.0940 | 0.1549 | 0.2632 | 0.4336 |
| NDL | 2.4157 | 0.1359 | 0.1415 | 0.3806 | 0.3961 |
| BB | 2.5407 | 0.0822 | 0.1119 | 0.2302 | 0.3132 |
| CB | 9.8302 | 0.0851 | 0.1420 | 0.2382 | 0.3976 |
| TXL | 4.0582 | 0.1005 | 0.1160 | 0.2814 | 0.3248 |
| BDPS | 1.0714 | 0.0604 | 0.0604 | 0.1691 | 0.1691 |
| SHND | 4.9136 | 0.0639 | 0.0680 | 0.1789 | 0.1905 |
| 7FA | 4.4198 | 0.1091 | 0.1116 | 0.3054 | 0.3126 |
| 8FA | 10.8991 | 0.0850 | 0.1613 | 0.2380 | 0.4516 |

**TABLE A1.2 Repeatability ($S_r$, r) and Reproducibility ($S_R$, R) Parameters Used for Calculation of Precision Statement for Macro-TGA, Two-Step Procedure for Dry Basis LOI (750°C and 950°C)**

| Material | Average % LOI | $S_r$ | $S_R$ | r | R |
|---|---|---|---|---|---|
| | | 750°C | | | |
| EBIT | 11.3225 | 0.0494 | 0.1057 | 0.1383 | 0.296 |
| NDL | 2.153 | 0.0532 | 0.0684 | 0.1488 | 0.1915 |
| BB | 2.3175 | 0.0529 | 0.054 | 0.1483 | 0.1512 |
| CB | 9.625 | 0.0392 | 0.0915 | 0.1097 | 0.2563 |
| TXL | 3.8755 | 0.0939 | 0.1297 | 0.2628 | 0.3631 |
| BDPS | 0.967 | 0.0637 | 0.0706 | 0.1785 | 0.1978 |
| SHND | 4.6285 | 0.0524 | 0.0661 | 0.1467 | 0.185 |
| 7FA | 4.273 | 0.0561 | 0.058 | 0.157 | 0.1623 |
| 8FA | 10.6325 | 0.0759 | 0.1105 | 0.2125 | 0.3094 |
| NIST 2696 | 2.2687 | 0.1154 | 0.1471 | 0.323 | 0.4119 |
| | | 950°C | | | |
| EBIT | 11.3795 | 0.0827 | 0.1276 | 0.2315 | 0.3572 |
| NDL | 2.1886 | 0.0445 | 0.0539 | 0.1245 | 0.1508 |
| BB | 2.3932 | 0.0535 | 0.0624 | 0.1498 | 0.1748 |
| CB | 9.6830 | 0.0491 | 0.1078 | 0.1375 | 0.3017 |
| TXL | 3.9339 | 0.0669 | 0.0763 | 0.1874 | 0.2137 |
| BDPS | 0.9564 | 0.0335 | 0.0465 | 0.0938 | 0.1303 |
| SHND | 4.6620 | 0.0444 | 0.0565 | 0.1242 | 0.1582 |
| 7FA | 4.2350 | 0.0861 | 0.0916 | 0.2412 | 0.2564 |
| 8FA | 10.5536 | 0.1251 | 0.1487 | 0.3502 | 0.4164 |
| NIST 1880a | 1.0484 | 0.0689 | 0.0915 | 0.1930 | 0.2561 |

**TABLE A1.3 Repeatability ($S_r$, r) and Reproducibility ($S_R$, R) Parameters Used for Calculation of Precision Statement for Muffle Furnace, One-Step Procedure for LOI (750°C and 950°C)**

| Material | Average % LOI | $S_r$ | $S_R$ | r | R |
|---|---|---|---|---|---|
| | | 750°C | | | |
| EBIT | 11.64 | 0.0709 | 0.0924 | 0.1986 | 0.2587 |
| NDL | 2.2763 | 0.123 | 0.1306 | 0.3443 | 0.3657 |
| BB | 2.4925 | 0.0608 | 0.0612 | 0.1702 | 0.1714 |
| CB | 9.8129 | 0.0395 | 0.0588 | 0.1105 | 0.1647 |
| TXL | 3.5796 | 0.0713 | 0.1111 | 0.1998 | 0.3111 |
| BDPS | 1.0208 | 0.0571 | 0.0664 | 0.1598 | 0.186 |
| SHND | 4.6871 | 0.081 | 0.0986 | 0.2267 | 0.2762 |
| 7FA | 4.4367 | 0.1041 | 0.1047 | 0.2914 | 0.2932 |
| 8FA | 10.9746 | 0.0843 | 0.1343 | 0.236 | 0.3759 |
| | | 950°C | | | |
| EBIT | 11.7936 | 0.1086 | 0.2246 | 0.3041 | 0.6289 |
| NDL | 2.6050 | 0.1494 | 0.1684 | 0.4184 | 0.4716 |
| CB | 9.9729 | 0.2084 | 0.2572 | 0.5835 | 0.7202 |
| TXL | 3.7696 | 0.1714 | 0.4017 | 0.4798 | 1.1248 |
| BDPS | 1.0718 | 0.1665 | 0.2397 | 0.4663 | 0.6711 |
| SHND | 4.8404 | 0.2258 | 0.3418 | 0.6323 | 0.9569 |
| 7FA | 4.4568 | 0.1221 | 0.1625 | 0.3418 | 0.4551 |

**TABLE A1.4 Repeatability ($S_r$, r) and Reproducibility ($S_R$, R) Parameters Used for Calculation of Precision Statement for Muffle Furnace, Two-Step Procedure for Dry Basis LOI (750°C and 950°C)**

| Material | Average % LOI | $S_r$ | $S_R$ | r | R |
|---|---|---|---|---|---|
| | | 750°C | | | |
| EBIT | 11.4039 | 0.1071 | 0.1762 | 0.2998 | 0.4933 |
| NDL | 2.0889 | 0.081 | 0.1854 | 0.2268 | 0.5191 |
| BB | 2.3739 | 0.0526 | 0.1055 | 0.1472 | 0.2954 |
| CB | 9.705 | 0.0757 | 0.1164 | 0.212 | 0.3259 |
| TXL | 3.5261 | 0.0788 | 0.1588 | 0.2206 | 0.4446 |
| BDPS | 0.9068 | 0.0554 | 0.1019 | 0.1551 | 0.2852 |
| SHND | 4.5211 | 0.1126 | 0.1701 | 0.3154 | 0.4764 |
| 7FA | 4.2629 | 0.0965 | 0.1435 | 0.2703 | 0.4019 |
| 8FA | 10.7336 | 0.1301 | 0.2452 | 0.3643 | 0.6865 |
| NIST 2696 | 2.1164 | 0.3207 | 0.3351 | 0.898 | 0.9384 |
| | | 950°C | | | |
| EBIT | 11.4738 | 0.0989 | 0.2104 | 0.2769 | 0.5891 |
| NDL | 2.4234 | 0.3403 | 0.6485 | 0.9529 | 1.8159 |
| BB | 2.4406 | 0.1309 | 0.1605 | 0.3665 | 0.4493 |
| CB | 9.8116 | 0.1055 | 0.1958 | 0.2955 | 0.5483 |
| TXL | 3.5737 | 0.1866 | 0.3659 | 0.5225 | 1.0246 |
| BDPS | 0.8663 | 0.1153 | 0.1914 | 0.3227 | 0.5359 |
| SHND | 4.7912 | 0.2520 | 0.4970 | 0.7057 | 1.3916 |
| 7FA | 4.3513 | 0.1151 | 0.2462 | 0.3223 | 0.6894 |
| 8FA | 10.6187 | 0.1165 | 0.2250 | 0.3263 | 0.6299 |
| NIST 1880a | 0.7931 | 0.1864 | 0.3862 | 0.5218 | 1.0813 |