Designation: D 3682 – 00

Standard Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes[1]

This standard is issued under the fixed designation D 3682; 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 This test method covers the analysis of the commonly determined major and minor elements in combustion residues from coal utilization processes.

NOTE 1—Test Methods D 1757 or D 5016 shall be used for determination of sulfur.

1.2 The values stated in SI units (Practice E 380) shall 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke[3]

D 2013 Method of Preparing Coal Samples for Analysis[3]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[3]

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal[3]

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[3]

D 5016 Test Method for Sulfur in Ash from Coal, Coke, and Residues from Coal Combustion Using High-Temperature Tube Furnace Combustion Method with Infrared Absorption[3]

D 5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[4]

3. Summary of Test Method

3.1 The combustion residue to be analyzed is ignited in air at 750°C to a constant weight. The ash is fused within lithium tetraborate ($Li_2B_4O_7$) followed by a final dissolution of the melt in either dilute hydrochloric acid (HCl) or dilute nitric acid (HNO_3). The solution is analyzed by atomic absorption/emission for applicable elements.

4. Significance and Use

4.1 A compositional analysis of the ash in coal is often useful in the total description of the quality of the coal. Knowledge of ash composition is also useful in predicting the behavior of ashes and slags in combustion chambers. Utilization of the ash by-products of coal combustion sometimes depends on the chemical composition of the ash.

4.2 Note that the chemical composition of laboratory-prepared coal ash may not exactly represent the composition of mineral matter in the coal or the composition of fly ash and slag resulting from commercial-scale burning of the coal.

5. Apparatus

5.1 *Ashing Furnace*, with an adequate air circulation and capable of having its temperature regulated between 700 and 750°C.

5.2 *Fusion Furnace*, with an operating temperature of 1000°C.

5.3 *Platinum Dish*, 35- to 85-mL capacity.

5.4 *Stirring Hotplate and Bars*, operating temperature of 200°C.

5.5 *Atomic Absorption Spectrometer*—Any dual-channel instrument using a deuterium (D_2) arc background corrector or other comparable simultaneous background correction system.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. It is intended that all reagents shall conform to

[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D 05.29 on Major Elements in Ash and Trace Elements of Coal.

Current edition approved Oct. 10, 2000. Published January 2001. Originally published as D 3682 – 78. Last previous edition D 3682 – 96.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.06.

[4] *Annual Book of ASTM Standards*, Vol 14.04.

the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The lithium tetraborate and lanthanum chloride reagents in particular should be examined for alkali and alkaline earth contamination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water as defined in Specification D 1193.

6.3 *Aluminum Stock Solution* (1000-ppm aluminum).

6.4 *Calcium Stock Solution* (1000-ppm calcium).

6.5 *Iron Stock Solution* (1000-ppm iron).

6.6 *Lanthanum Chloride Solution* (175-g/L lanthanum chloride ($LaCl_3$) or equivalent 10 % lanthanum).

6.7 *Lithium Tetraborate* —($Li_2B_4O_7$), powder.

6.8 *Magnesium Stock Solution* (1000-ppm magnesium).

6.9 *Manganese Stock Solution* (1000-ppm manganese).

6.10 *Potassium Stock Solution* (1000-ppm potassium).

6.11 *Silicon Stock Solution* (200-ppm silicon) (Note 2).

6.12 *Sodium Stock Solution* (1000-ppm sodium).

6.13 *Solvent Acid*—Dilute 50 mL of concentrated hydrochloric acid (sp gr 1.19) or 50 mL of concentrated nitric acid (sp gr 1.42) to 1000 mL. Either acid solution may be used, but whichever is chosen should be used throughout the subsequent solution preparations.

6.14 *Titanium Stock Solution* (1000-ppm titanium).

NOTE 2—Commercial silicon standards prepared from sodium silicate have not proved satisfactory. A standard stock solution can be prepared by fusing 0.1070 g of reignited spectrographic grade silica (SiO_2) with 1 g of lithium tetraborate, dissolving in solvent acid, and diluting to 250 mL as described for sample preparation in 8.3.1 and 8.3.2. This solution is 200-ppm silicon. Preferable standard preparations for silica are made by fusion and dilution of ash sample(s) of known composition in accordance with 8.3.1 and 8.3.2. The standard sample(s) should have a composition(s) similar to the unknown.

7. Sample Preparation

7.1 The combustion residue to be analyzed must first be ignited in air at 750°C to a constant weight before analysis. Allow the ash to cool, transfer to an agate mortar, and grind to pass a 74-μm (No. 200) sieve. Reignite the ash at 750°C for 1 h, cool rapidly, and immediately weigh portions for analysis. If samples are stored, reignite the ash before weighing or determine loss on ignition at 750°C on a separate sample weighed out at the same time as the analysis sample and make the necessary corrections. Thoroughly mix each sample before weighing.

7.2 If the ash is to be prepared from a coal, prepare the coal analysis sample in accordance with Method D 2013 by pulverizing the materials to pass a 250-μm (No. 60) sieve.

7.2.1 Analyze separate test portions of the coal for moisture and ash contents in accordance with Test Methods D 3173, D 3174, or D 5142 so that calculations to other bases can be made.

7.2.2 Prepare the ash from a thoroughly mixed analysis sample of coal. Spread the coal in a layer not over 6 mm (¼ in.) in depth in a fireclay or porcelain roasting dish. Place the dish in a cold muffle furnace and heat gradually so that the temperature reaches 500°C in 1 h and 750°C in 2 h. Ignite at 750°C until all carbonaceous matter is removed. Proceed with the preparation of the ash in accordance with 7.1.

8. Procedure

8.1 The solutions and proportions described below are for typical ash samples as represented by American coals of bituminous rank. Therefore, stronger or weaker dilutions may be required to establish suitable concentrations for those elements of varying percents outside the range of the typical sample. Each analyst must determine the sensitivity and linear range of calibration of their equipment and choose concentration ranges for standards compatible with the samples and instrument specific to their own work.

8.1.1 Calculations used in subsequent sections are developed from the following general formula for percent concentration of element oxide, *E*, in moisture-free coal ash:

$$E = [(C - B)/(A - B)] \times (N/M) \times F \times 100 \quad (1)$$

where:
- A = absorbance of standard;
- B = absorbance of blank;
- C = absorbance of sample solution;
- N = element in standard, ppm;
- M = sample of solution, ppm; and
- F = conversion from element to oxide.

NOTE 3—Concentrations in the ash may be converted to the air-dried coal basis using the following expression:

$$C = (AB/100)$$

where:
- C = oxide in air-dried coal, %;
- A = oxide in ash, %; and
- B = ash as determined in Test Method D 3174 or Test Method D 5142, %.

See Practice D 3180 for procedures to convert values to other bases.

8.2 To minimize the potential of contamination, the platinumware must be prepared by boiling in solvent acid and rinsing thoroughly with reagent-grade water. After this initial cleaning, the platinumware must be handled with *clean* tongs and protected from further contamination from table tops, and so forth. All glassware used in analyses must be equally clean and equally protected.

8.3 *Sample Fusion and Solution*:

8.3.1 Weigh 0.1 ± 0.0002 g of the sample as prepared in 7.3 into a platinum dish (5.3) (Note 4) and add 0.5 g of $Li_2B_4O_7$. Mix the ash and lithium tetraborate well, then add an additional 0.5 g of $Li_2B_4O_7$ to cover the mixture. Place the dish in a clean silica or refractory tray and place in a muffle furnace preheated to 1000°C; 15 min at 1000°C is sufficient to fuse the mixture completely. Remove the tray and dish and cool to room temperature. Carefully rinse the bottom and outside of the

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

platinum dish to remove possible contamination, then place it in a clean 250- or 400-mL beaker. Place a clean TFE-fluorocarbon-coated stirring magnet inside the dish, add 150 mL of solvent acid to the beaker and dish, and place immediately on the stirring hotplate. Heat the solution to just below boiling temperature and maintain for not more than 30 min with constant stirring. This time and temperature are sufficient to completely dissolve the melt. If stirring is not maintained constantly, some of the ash constituents are apt to precipitate and the analysis must be repeated.

NOTE 4—The use of graphite crucibles and subsequent dissolution of fused beads from them was not investigated; however, their successful use in similar methods has been reported.[6]

8.3.2 Remove the beaker from the hotplate and permit to cool to room temperature. Quantitatively transfer the solution to a 200-mL volumetric flask, wash the platinum dish and beaker with small amounts of solvent acid and dilute to the 200-mL mark with the solvent acid. This solution is 500 ppm with respect to the total sample and contains 5 g/L of $Li_2B_4O_7$ solution.

8.3.3 *Solution 1*—Pipet 20 mL of the sample solution prepared in 8.3.2 into a 50-mL volumetric flask and dilute to volume with solvent acid. This solution is 200 ppm with respect to the total sample and contains 2 g/L of $Li_2B_4O_7$ solution.

8.3.4 *Solution 2*—Pipet 10 mL of the sample solution prepared in 8.3.2 into a 100-mL volumetric flask, add 10 mL of lanthanum solution (see 6.7), and dilute to volume with solvent acid. This solution is 50 ppm with respect to the total sample and contains 0.5 g/L of $Li_2B_4O_7$ solution.

NOTE 5—Lanthanum was included in the solution as a release agent in those determinations that may require it (for example, calcium). Alternative measures, such as the use of nitrous oxide flame, are permissible.

9. Silicon Dioxide (SiO_2)

9.1 *Preparation of Standards*—Prepare a series of standards by combining the following volumes of the silicon stock solution (see 6.11) with 1 mL of the aluminum stock solution (see 6.3) and diluting to 100 mL with the blank solution (see 9.2). Only those standards need be prepared that bracket the expected SiO_2 concentrations in the samples.

Silicon, Stock Solution (6.13), mL	Silicon, ppm	Equivalent SiO_2, %
25	50	53.5
20	40	42.8
15	30	32.1
10	20	21.4

9.2 *Blank Solution*—Prepare a solution of 2 g/L of $Li_2B_4O_7$ solution in solvent acid.

9.3 *Sample Solution*—Use the 200-ppm sample solution as prepared in 8.3.3.

9.4 *Atomic Absorption Operating Conditions*—Use a silicon hollow-cathode lamp and set the monochromator at 251.6 nm. Use a nitrous oxide/acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

9.5 *Determination*—Read out absorbance values for the blank solution, standard solutions, and sample solution. In this and all subsequent determinations, those instruments so equipped may be calibrated to read out "percent concentration" directly. Determine percent SiO_2 as follows:

$$SiO_2, \% = [(C - B)/(A - B)] \times N \times 1.07 \quad (2)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = silicon in standard, ppm.

10. Aluminum Oxide (Al_2O_3)

10.1 *Preparation of Standards*—Prepare standard solutions containing from 10- to 30-ppm aluminum and dilute with blank solution 9.2.

10.2 *Blank Solution*—Use the blank as prepared in 9.2.

10.3 *Sample Solution*—Use Solution 1 as prepared in 8.3.3.

10.4 *Atomic Absorption Operating Conditions*—Use an aluminum hollow-cathode lamp and set the monochromator at 309.2 nm. Use a nitrous oxide-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

10.5 *Determination*—Read out absorbance values for the blank solution, standard solutions, and sample solution. Determine percent Al_2O_3 as follows:

$$Al_2O_3, \% = [(C - B)/(A - B)] \times N \times 0.94 \quad (3)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = aluminum in standard, ppm.

11. Iron Oxide (Fe_2O_3)

11.1 *Preparation of Standards*—Prepare standard solutions containing from 2- to 10-ppm iron and 10 mL of lanthanum solution per 100 mL. Dilute with blank solution (11.2).

11.2 *Blank Solution*—Prepare a solution of solvent acid containing 0.5-g/L $Li_2B_4O_7$.

11.3 *Sample Solution*—Use Solution 2 as prepared in 8.3.4.

11.4 *Atomic Absorption Operating Conditions*—Use an iron hollow-cathode lamp and set the monochromator at 248.3 nm. Use an air-acetylene flame of stoichiometric composition. Adjust other instrument parameters to optimum for the particular equipment used.

11.5 *Determination*—Read the absorbance values for the blank solution, the standard solutions, and the sample solution. Determine percent Fe_2O_3 as follows:

$$Fe_2O_3, \% = [(C - B)/(A - B)] \times N \times 2.86 \quad (4)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = iron in standard, ppm.

[6] Muter, R. B. and Nice, L. L., "Major and Minor Constituents in Siliceous Materials by Atomic Absorption Spectroscopy," Advances in Chemistry Series 141, *Trace Elements in Fuels*, American Chemical Society, Washington, DC, 1975, pp. 57–65.

12. Calcium Oxide (CaO)

12.1 *Preparation of Standards*—Prepare standard solutions containing 1- to 10-ppm calcium and 10 mL of lanthanum solution per 100 mL. Dilute with the blank solution as described in 11.2.

12.2 *Blank Solution*—Use blank solution as described in 11.2.

12.3 *Sample Solution*—Use Solution 2 as described in 8.3.4.

12.4 *Atomic Absorption Operating Conditions*—Use a calcium hallow-cathode lamp and set the monochromator at 422.7 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

12.5 *Determination*—Read the absorbance values for the blank solution, the standard solutions, and the sample solution. Determine percent CaO as follows:

$$\text{CaO, \%} = [(C - B)/(A - B)] \times N \times 2.80 \quad (5)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = calcium in standard, ppm.

13. Magnesium Oxide (MgO)

13.1 *Preparation of Standards*—Prepare standard solutions containing 0.5- to 2-ppm magnesium and 10 mL of lanthanum solution per 100 mL. Dilute with blank solution as described in 11.2.

13.2 *Blank Solution*—Use blank solution as described in 11.2.

13.3 *Sample Solution*—Use Solution 2 as described in 8.3.4.

13.4 *Atomic Absorption Operating Conditions*—Use a magnesium hollow-cathode lamp and set the monochromator at 285.1 nm. Use an air-acetylene flame of stoichiometric composition. Adjust other instrument parameters to optimum for the particular equipment used.

13.5 *Determination*—Read the absorbance values for the blank solution, the standard solutions, and the sample solution. Determine percent MgO as follows:

$$MgO\text{, \%} = [(C - B)/(A - B)] \times N \times 3.32 \quad (6)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = magnesium in standard, ppm.

14. Sodium Oxide (Na_2O)

14.1 *Preparation of Standards*—Prepare standard solutions containing 0.5- to 2-ppm sodium and 10 mL of lanthanum[7] solution per 100 mL. Dilute with blank solution as described in 11.2.

14.2 *Blank Solution*—Use blank solution as described in 11.2.

14.3 *Sample Solution*—Use Solution 2 as described in 8.3.4.

14.4 *Atomic Absorption Operating Conditions*—Use a sodium hollow-cathode lamp and set the monochromator at 589.0 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

14.5 *Determination*—Read the absorbance values for the blank solution, the standard solutions, and the sample solution. Determine percent Na_2O as follows:

$$Na_2O\text{, \%} = [(C - B)/(A - B)] \times N \times 2.70 \quad (7)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = sodium in standard, ppm.

NOTE 6—Alternatively, sodium may be determined by flame emission.

15. Potassium Oxide (K_2O)

15.1 *Standard Preparation*—Prepare standard solutions containing 0.5- to 2-ppm potassium and 10 mL of lanthanum[7] solution per 100 mL. Dilute with blank solution as described in 11.2.

15.2 *Blank Solution*—Use blank solution as described in 11.2.

15.3 *Sample Solution*—Use Solution 2 as described in 8.3.4.

15.4 *Atomic Absorption Operating Conditions*—Use a potassium hollow-cathode lamp and set the monochromator at 766.5 nm. Use an air-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

15.5 *Determination*—Read the absorbance values for the blank solution, the standard solution, and the sample solution. Determine percent K_2O as follows:

$$K_2O\text{, \%} = [(C - B)/(A - B)] \times N \times 2.41 \quad (8)$$

where:
A = absorbance of standard nearest C,
B = absorbance of blank,
C = absorbance of sample, and
N = potassium in standard, ppm.

NOTE 7—Alternatively, potassium may be determined by flame emission.

NOTE 8—It may be found convenient to combine the standards for iron, calcium, magnesium, sodium, and potassium in one solution.

16. Titanium Dioxide (TiO_2)

16.1 *Standard Preparation*—Prepare a standard solution containing 3-ppm titanium and 5 g/L of $Li_2B_4O_7$ solution dissolved in solvent acid. If required, successive dilutions may be made with the blank solution (see 16.2).

16.2 *Blank Solution*—Prepare a solution containing 5 g/L of $Li_2B_4O_7$ solution dissolved in solvent acid.

16.3 *Sample Solution*—Use the original sample solution as prepared in 8.3.2.

16.4 *Atomic Absorption Operating Conditions*—Use a titanium hollow-cathode lamp and set the monochromator at 364.3 nm. Use a nitrous oxide-acetylene flame. Adjust other instrument parameters to optimum for the particular equipment used.

16.5 *Determination*—Read the absorbance for the blank solution, the standard solution and the sample solution. Determine percent TiO_2 as follows:

[7] Supporting data are available from ASTM Headquarters. Request RR:D05-1000.

$$TiO_2, \% = [(C - B)/(A - B)] \times 1.00 \quad (9)$$

where:
A = absorbance of standard,
B = absorbance of blank, and
C = absorbance of sample.

17. Report

17.1 Report the percentages for the elemental oxides in the ash as specified in 9.5, 10.5, 11.5, 12.5, 13.5, 14.5, 15.5, and 16.5.

17.2 Report the method used for concurrent moisture and ash determination, if applicable.

17.3 Use Practice D 3180 for procedures to convert values to other bases.

18. Precision and Bias

18.1 *Precision*—The precision of this test method is in accordance with Table 1. The precision characterized by the repeatability (S_r, r) and reproducibility (S_R, R) is in accordance with Table A1.1.

18.1.1 *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 material, may be expected to occur with a probability of approximately 95 %.

18.1.2 *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 material that is as nearly homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

18.2 *Bias*—The NIST Standard Reference Material 1633b was included in the interlaboratory study to ascertain possible bias between reference material values and those determined by this test method. A comparison of the NIST values and those obtained in the interlaboratory study are given in Table 2.

18.3 An interlaboratory study, designed in accordance with Practice E 691, was conducted in 1999. Six laboratories participated.[8]

19. Keywords

19.1 aluminum oxide ($A1_2O_3$); ash analysis; atomic absorption spectroscopy; calcium oxide (CaO); elemental ash composition; ferric oxide (Fe_2O_3); magnesium oxide (MgO); potassium oxide (K_2O); silicon dioxide (SiO_2); sodium oxide (Na_2O); titanium dioxide (TiO_2)

[8] Supporting data are available from ASTM Headquarters. Request RR:D05-1000.

TABLE 1 Concentrations Ranges and Limits for Repeatability and Reproducibility for Major and Minor Elemental Oxides in Ash from Combustion Residues

Elemental Oxide	Concentration Range, %	Repeatability Limit r	Reproducibility Limit R
SiO_2	33.14 – 57.73	$0.33 + 0.04\ \bar{x}$[A]	$-1.21 + 0.14\ \bar{x}$
Al_2O_3	11.32 – 31.83	0.80	$-0.24 + 0.09\ \bar{x}$
Fe_2O_3	2.93 – 41.11	$0.01 + 0.06\ \bar{x}$	$0.36 + 0.06\ \bar{x}$
MgO	0.45 – 7.12	$0.04 + 0.03\ \bar{x}$	$0.04 + 0.09\ \bar{x}$
CaO	1.46 – 22.19	$0.21 + 0.04\ \bar{x}$	$0.15 + 0.11\ \bar{x}$
TiO_2	0.57 – 1.44	0.13	0.19
K_2O	0.45 – 2.99	$0.03 + 0.03\ \bar{x}$	$0.05 + 0.05\ \bar{x}$
Na_2O	0.14 – 7.15	$0.06 + 0.03\ \bar{x}$	$0.04 + 0.18\ \bar{x}$

[A] Where $\bar{x}$ is the average of two single test results.

TABLE 2 Comparison of Certified Values for Standard Reference Material 1633b with Interlaboratory Study Values for Major and Minor Elemental Oxides in Ash from Combustion Residues

Elemental Oxide	AA-RR Value	NIST Value	Bias, %	Significant (95 % Confidence Level)
SiO_2	49.57 ± 0.96	49.25 ± 0.17	0.65	no
Al_2O_3	28.16 ± 0.31	28.44 ± 0.51	–0.98	no
Fe_2O_3	10.96 ± 0.25	11.12 ± 0.33	–1.44	no
MgO	0.79 ± 0.02	0.80 ± 0.01	-1.25	no
CaO	2.01 ± 0.12	2.11 ± 0.08	–4.74	no
TiO_2	1.30 ± 0.04	1.32 ± 0.02	–1.52	no
K_2O	2.25 ± 0.04	2.35 ± 0.04	–4.25	yes
Na_2O	0.26 ± 0.02	0.27 ± 0.004	–3.70	no

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS

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

SiO_2					
Materials	Average	S_r	S_R	r	R
CAN	33.1437	0.6687	1.3581	1.8725	3.8026
FPA	57.7325	0.8709	2.7464	2.4386	7.69
IL	48.7917	0.604	2.1085	1.6912	5.9037
JO	40.1846	0.5457	1.1898	1.5279	3.3316
WDK	34.6421	0.5795	0.9385	1.6227	2.6279
95-1	29.8767	0.4486	1.4572	1.256	4.0801
95-6	29.7379	0.5258	1.2832	1.4724	3.593
NIST 1633B	49.5717	0.9622	1.856	2.6941	5.1969

CaO					
Materials	Average	S_r	S_R	r	R
CAN	4.4988	0.1446	0.169	0.4049	0.4732
FPA	1.4646	0.103	0.1536	0.2883	0.4301
IL	4.4004	0.1224	0.1993	0.3427	0.5581
JO	7.4804	0.1686	0.3083	0.472	0.8633
WDK	17.5079	0.5356	0.7637	1.4998	2.1384
95-1	22.1937	0.3805	0.8789	1.0653	2.4609
95-6	19.6621	0.307	0.7844	0.8597	2.1962
NIST 1633B	2.0154	0.1196	0.1579	0.335	0.4422

Al_2O_3					
Materials	Average	S_r	S_R	r	R
CAN	11.3254	0.185	0.3371	0.518	0.9438
FPA	29.0638	0.2896	1.1124	0.8109	3.1146
IL	19.2546	0.3629	0.597	1.0162	1.6715
JO	31.8275	0.2288	0.8521	0.6406	2.386
WDK	15.825	0.2908	0.4738	0.8142	1.3267
95-1	18.9821	0.2141	0.5738	0.5994	1.6066
95-6	19.7283	0.3554	0.3554	0.9951	0.9951
NIST 1633b	28.1637	0.3148	0.794	0.8815	2.2231

TiO_2					
Materials	Average	S_r	S_R	r	R
CAN	0.574 58	0.054 28	0.091 09	0.151 97	0.255 06
FPA	1.439 58	0.051 76	0.089 63	0.144 93	0.250 95
IL	0.925 00	0.036 55	0.054 40	0.102 35	0.152 33
JO	1.172 5	0.042 26	0.042 26	0.118 33	0.118 33
WDK	0.913 33	0.029 63	0.032 79	0.082 96	0.091 8
95-1	1.140 83	0.038 73	0.069 46	0.108 44	0.194 48
95-6	1.303 75	0.051 87	0.065	0.145 23	0.181 99
NIST 1633b	1.279 17	0.048 13	0.085 68	0.134 77	0.239 91

K_2O					
Materials	Average	S_r	S_R	r	R
CAN	1.367 92	0.026 33	0.054 73	0.073 71	0.153 26
FPA	2.991 67	0.050 94	0.080 55	0.142 62	0.225 53
IL	2.175	0.029 3	0.030 19	0.082 03	0.084 52
JO	0.523 75	0.017 52	0.029 19	0.049 06	0.081 73
WDK	0.54	0.019 44	0.026 14	0.054 42	0.073 19
95-1	0.451 67	0.022 67	0.027 74	0.063 47	0.077 68
95-6	0.648 75	0.017 52	0.032 97	0.049 06	0.092 32
NIST 1633b	2.251 25	0.044 33	0.071 96	0.124 13	0.201 49

Na_2O					
Materials	Average	S_r	S_R	r	R
CAN	0.140 83	0.028 63	0.031 08	0.080 15	0.087 04
FPA	0.254 58	0.016 71	0.035 18	0.046 78	0.098 52
IL	1.366 25	0.028 16	0.075 69	0.078 85	0.211 94
JO	0.251 67	0.024 49	0.033 86	0.068 59	0.094 81
WDK	1.067 08	0.026 54	0.059 91	0.074 3	0.167 75
95-1	3.488 33	0.109 19	0.278 28	0.305 73	0.779 18
95-6	7.149 58	0.086 87	0.459 96	0.243 23	1.287 89
NIST 1633b	0.258 75	0.024 41	0.034 7	0.068 35	0.097 15

Fe_2O_3					
Materials	Average	S_r	S_R	r	R
CAN	41.1154	0.8321	1.0852	2.3299	3.0385
FPA	2.9279	0.0505	0.1685	0.1414	0.4719
IL	15.86	0.2502	0.4939	0.7005	1.383
JO	4.6396	0.1449	0.2774	0.4058	0.7768
WDK	6.5746	0.1445	0.3407	0.4045	0.9539
95-1	6.535	0.1482	0.1995	0.415	0.5587
95-6	5.3317	0.0833	0.2662	0.2332	0.7454
NIST 1633b	10.9625	0.2547	0.3576	0.7131	1.0012

MgO					
Materials	Average	S_r	S_R	r	R
CAN	0.448 33	0.019 79	0.037 79	0.055 41	0.105 81
FPA	1.049 17	0.011 9	0.050 77	0.033 33	0.142 16
IL	0.951 25	0.036 8	0.065 73	0.103 04	0.184 03
JO	1.315 83	0.034 8	0.065 25	0.097 44	0.182 7
WDK	4.313 75	0.060 79	0.175 91	0.170 22	0.492 54
95-1	7.122 5	0.084 93	0.224 3	0.237 82	0.628 04
95-6	6.197 08	0.114 17	0.272 78	0.319 68	0.763 79
NIST 1633b	0.787 08	0.024 12	0.052 03	0.067 55	0.145 69