

Designation: D6349 - 09

# Standard 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<sup>1</sup>

This standard is issued under the fixed designation D6349; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This test method covers a procedure for the analysis of the commonly determined major and minor elements in coal, coke, and solid residues from combustion of coal and coke. These residues may be laboratory ash, bottom ash, fly ash, flue gas desulfurization sludge, and other combustion process residues.

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:<sup>2</sup>

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

- D1193 Specification for Reagent Water
- D2013 Practice for Preparing Coal Samples for Analysis
- D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
- D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
- D5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures
- **E691** Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

# 2.2 ISO Standard:<sup>3</sup>

ISO/IEC Guide 99:2007 International vocabulary of metrology -- Basic and general concepts and associated terms (VIM)

# 3. Summary of Test Method

3.1 The sample to be analyzed is ashed under standard conditions and ignited to constant weight. The ash is fused with a fluxing agent followed by dissolution of the melt in dilute acid solution. Alternatively, the ash is digested in a mixture of hydrofluoric, nitric, and hydrochloric acids. The solution is analyzed by inductively coupled plasma-atomic emission spectrometry (ICP) for the elements. The basis of the method is the measurement of atomic emissions. Aqueous solutions of the samples are nebulized, and a portion of the aerosol that is produced is transported to the plasma torch where excitation and emission occurs. Characteristic line emission spectra are produced by a radio-frequency inductively coupled plasma. A grating monochromator system is used to separate the emission lines, and the intensities of the lines are monitored by photomutilplier tube or photodiode array detection. The photocurrents from the detector are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of elements. Background must be measured adjacent to analyte lines of samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured.

## 4. Significance and Use

4.1 A compositional analysis of coal and coke and their associated combustion residues are often useful in assessing

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<sup>&</sup>lt;sup>1</sup> This test method is 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

TABLE 1	Recommended	Wavelengths	for Elements	Determined
		by ICP		

	-,
Element	Wavelengths, nm
Aluminum	396.152, 256.80, 308.215, 309.271
Barium	455.403, 493.41, 233.53
Calcium	317.93, 315.887, 364.44, 422.67
Iron	259.940, 271.44, 238.204
Magnesium	279.553, 279.08, 285.21, 277.983
Manganese	257.610, 294.92, 293.31, 293.93
Phosphorous	178.287, 214.900
Potassium	766.491, 769.896
Silicon	212.412, 288.16, 251. 611
Sodium	588.995, 589.592
Strontium	421.55
Sulfur	182.04
Titanium	337.280, 350.50, 334.941

their quality. Knowledge of the elemental composition of the associated residues is also useful in predicting the elemental enrichment/depletion compositional behavior of ashes and slags in comparison to the concentration levels in the parent coal. Utilization of the ash by-products and hazardous potential may also depend on the chemical composition and leachability of the inorganic constituents of the coal ash.

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

# 5. Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of major and minor elements. The analyst should follow the manufacturer's operating guide to develop and apply correction factors to compensate for the interferences. The interferences can be classified as spectral, physical, and chemical.

5.1.1 Spectral interferences can be categorized as overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array.

5.1.2 Table 1 lists the elements determined by this method and the recommended wavelengths using conventional nebulization. Sulfur may only be determined if the sample is dissolved by the mixed acid dissolution described in 10.3.2.

5.1.3 Table  $2^4$  lists some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear

relations between concentration and intensity for the analytes and the interferents can be assumed. The analyst should follow the manufacturer's operating guide to develop and apply correction factors to compensate for the interferences.

5.1.4 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids or acid concentrations, or both. The use of a peristaltic pump is recommended to lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample or utilization of standard addition techniques, or both. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon before nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate, particularly nebulizer flow, improves instrument precision. This is accomplished with the use of mass flow controllers.

5.1.5 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique. However, if such effects are observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, matrix matching, and standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

# 6. Apparatus

6.1 *Ashing Furnace*, with an adequate air circulation (two to four volume changes per minute) and capable of having its temperature regulated between 700 and 750°C.

6.2 *Fusion Furnace*, with an operating temperature of 1000 to 1200°C.

6.3 *Meeker-Type Burner*, with inlets for fuel gas (propane or natural gas) and compressed air, capable of flame temperatures of 1000 to 1200°C.

6.4 *Platinum Dishes or Crucibles*, 35- to 85-mL capacity. Graphite crucibles with 10- to 15-mL capacity may also be used.

6.5 *Stirring Hotplate and Bars*, with operating temperature up to 200°C.

6.6 *Polycarbonate Bottles*, 250-mL capacity with an O-ring seal and screw cap, capable of withstanding temperatures of 100 to 130°C, the pressure that is developed during the digestion, and resistant to oxidation. Other types of bottles or vials may be used provided they are capable of withstanding the temperatures and pressures developed duing the digestion.

6.7 Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP), either a sequential or simultaneous spectrometer is suitable. Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects

 $<sup>^4</sup>$  Methods for Chemical Analysis of Water and Wastes , (EPA-600/4-79-020), Metals-4, Method 200.7 CLP-M.

TABLE 2 Examples of Analyte Concentration Equivalents Arising from Interference at the 100-ppm (mg/L) Level<sup>4</sup>

Note 1—Dashes indicate that no interference was observed even when interferents were introduced at the following levels: Al, Ca, and Fe = 1000 ppm, Mn = 200 ppm, and Mg = 100 ppm.

				Inte	rferents		
Analyte Elements	Wavelengths, nm	AI	Ca	Fe	Mg	Mn	Ti
Aluminum	308.215					0.21	
Barium	455.103						
Calcium	317.933			0.01	0.01	0.04	0.03
Iron	259.940					0.12	
Magnesium	279.079		0.02	0.13		0.25	0.07
Manganese	257.610	0.005		0.002	0.002		
Silicon	288.148						
Sodium	588.995						0.08

must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument's linear range in which correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements of this method and to maintain quality control data confirming instrument performance and analytical results.

#### 7. Reagents

7.1 *Purity of Reagents*—Reagents grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society in which such specifications are available.<sup>5</sup> 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water as defined by Specification D1193.

7.3 *Standard Stock Solutions*—Stock solutions of 1000 ppm (mg/L) for each element are needed for preparation of dilute standards in the range from <0.1 to 100 ppm. Prepare standard stock solutions from 99.999 % purity metals or salts. Alternatively, one can use commercially available stock solutions specifically prepared for ICP-AES spectroscopy.

7.4 Internal Standard Solution—Stock solution of 1000 ppm (mg/L) of yttrium (Y), scandium (Sc), indium (In), or other suitable element not found in significant concentrations in the test samples.

7.5 Acids:

7.5.1 Hydrochloric Acid—Concentrated HCl. sp gr 1.19.

7.5.2 Hydrofluoric Acid—Concentrated HF, sp gr 1.17.

7.5.3 Nitric Acid— Concentrated HNO<sub>3</sub>, sp gr 1.42.

7.5.4 *Nitric Acid* (5 + 95)—Dilute 50 mL of concentrated nitric acid to 1000 mL.

7.5.5 *Mixed Acid Solution, 70/30 HCl/HF*—Mix seven parts concentrated hydrochloric acid and three parts concentrated hydrofluoric acid.

7.6 *Fluxing Agents*— Lithium tetraborate,  $Li_2B_4O_7$ , or mixtures of lithium tetraborate ( $Li_2B_4O_7$ ) and anhydrous lithium metaborate (LiBO<sub>3</sub>).

7.7 Boric Acids Solution-1.5 %.

7.8 Hydrogen Peroxide—30%

7.9 *Wetting Agents*—Approximately 0.1 g of reagent grade lithium iodide (LiI) or other suitable wetting agent may be added to the flux to facilitate pooling of the melt and removal of the melt of cooled pellet.

7.10 *Standard Solution Diluent*—Use either 7.10.1 or 7.10.2.

7.10.1 Weigh 4 g, to the nearest 0.0001 g, of fluxing agent (see 7.6) into a clean 1000-mL beaker containing a magnetic stirring bar. Add 500 mL of 5 + 95 nitric acid (see 7.5.4) to the beaker and place on a stirring hot plate. Heat the mixture to just below boiling and maintain this temperature with constant stirring until the fluxing agent dissolves. This dissolution process should take about 30 min or less (see Note 1). Quantitatively transfer the warm solution to a 1000-mL volumetric flask. After the solution cools to room temperature, dilute to 1000 mL with reagent grade water.

7.10.2 Weigh 4 g, to the nearest 0.0001 g, of fluxing agent (see 7.6) into a platinum dish (or crucible). Heat to 1000°C to form a liquid and cool. Carefully rinse the bottom and outside of the platinum dish to remove possible contamination. Place the cooled platinum dish containing the flux and a magnetic stirring bar into a clean 1000-mL beaker. Add 500 mL of 5 + 95 nitric acid (see 7.5.4) to the beaker and place immediately on the stirring hotplate. Heat the mixture to just below the boiling temperature and maintain this temperature with constant stirring until the melt dissolves. This dissolution process should take about 30 min (see Note 1). After dissolution remove the platinum dish after rinsing with reagent water and collecting the washings in the acid solution. Quantitatively transfer the warm solution to a 1000-mL volumetric flask. After the solution cools to room temperature, dilute to 1000 mL with reagent grade water.

NOTE 1—This time and temperature are sufficient to dissolve the melt completely. If stirring is not maintained constantly, some of the material may not dissolve, and the final solution must be filtered before use.

<sup>&</sup>lt;sup>5</sup> 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.

7.11 *Blank Solutions*—All of the test methods in this standard require two types of blank solutions. A calibration blank that is used to establish the analytical calibration curve and a method blank which is used to evaluate possible contamination and assess spectral background. The calibration blank is also used initially and periodically to verify the baseline of the calibration has not changed significantly.

7.11.1 *Calibration Blank*—The same solution as the Standard Solution Diluent.

7.11.2 *Method Blank*—The method blank shall be processed through the same digestion procedure as the samples and consist of all the reagents in the same volumes as used in preparing the samples.

7.12 Initial calibration verification standard(s) :

7.12.1 Where possible the initial calibration verification standard(s) shall be from alternate producers or different lot numbers from the calibration standard(s).

7.12.2 Where possible the initial calibration verification standard(s) shall be traceable to a primary standard such as a NIST CRM.

7.13 *Periodic calibration verification standard(s)*—The source of these materials can be the same as the calibration materials.

7.14 *Primary Control Sample*—A material that is processed following the same procedure as an analytical sample and is a measurement standard whose quantity value and measurement uncertainty are established without relation to another measurement standard for a quantity of the same kind (see ISO/IEC Guide 99:2007 International Vocabulary of Basic and General Terms in Metrology).

7.15 Secondary Control Sample—A material that is processed following the same procedure as an analytical sample and is a measurement standard whose quantity value and measurement uncertainty are assigned through calibration against, or comparison with, a primary measurement standard for a quantity of the same kind (see ISO/IEC Guide 99:2007 International Vocabulary of Basic and General Terms in Metrology).

#### 8. Sample Preparation

8.1 *Coal and Coke*—Prepare the analysis sample in accordance with Practice D2013 (coal) or Practice D346 (coke) by pulverizing the material to pass a 250- $\mu$ m (No. 60) sieve.

8.1.1 Analyze separate test portions for moisture content in accordance with Test Methods D3173 or D5142 so that calculation to other bases can be made.

8.2 Solid Combustion Residue—Dry a representative portion of the solid residue to constant weight at 110 to 115°C. Determine the moisture loss during this drying step if it is desirable to calculate results to an as-received basis. Crush the dried portion of the sample to pass a No. 200 (75-µm) sieve. Use a mill that minimizes metal contamination.

# 9. Preparation of Ash

9.1 Ashing of Coal and Coke Analysis Sample—Prepare the ash from a thoroughly mixed analysis sample of coal or coke (see 8.1) that has been ground to pass a 250- $\mu$ m (No. 60) U.S.A. standard sieve. Spread the coal and coke in a layer not over 6 mm (<sup>1</sup>/<sub>4</sub> in.) in depth in a porcelain, quartz, or fused

silica 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 or for an additional 2 h. Allow the dish to cool, transfer to an agate mortar, and grind to pass a 75- $\mu$ m (No. 200) sieve. Reignite the ash at 750°C for 1 h, cool rapidly, and weigh portions for analysis. If samples are stored and the absorption of moisture or CO<sub>2</sub>, or both, is in question, reignite the ash using the 500-750°C staged combustion before use. Alternatively, determine loss on ignition using the 500-750°C staged combustion on a separate sample weighed out at the same time as the test portion and make the necessary corrections. Thoroughly mix each sample before weighing.

9.2 Ashing of Solid Combustion Residues-Spread an appropriate amount of the prepared sample from 8.2 in a layer not over 2 mm in a porcelain, quartz, or fused silica 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 the sample at 750°C until all carbonaceous matter is removed. Allow the ash to cool, transfer to an agate mortar, and grind to pass a 75-µm (No. 200) sieve. Reignite the ash for 1 h at 750°C, cool rapidly, and weigh portions for analysis. If samples are stored and the absorption of moisture or CO<sub>2</sub>, or both, is in question, reignite the ash using the 500-750°C staged combustion before use. Alternatively, determine loss on ignition using the 500-750°C staged combustion on a separate sample weighed out at the same time as the test portion and make the necessary corrections. Thoroughly mix each sample before weighing.

#### **10. Procedure**

10.1 The solutions and proportions described below are the typical ash samples as represented by American coals. 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. Analysts must determine the sensitivity and linear range of calibration of their own equipment and choose concentration ranges for standards compatible with the samples and instrument specific to their own work.

10.2 To minimize the potential of contamination, platinum ware must be prepared by boiling in dilute  $HNO_3$  (5 + 95) and rinsing thoroughly with reagent-grade water. After this initial cleaning, the platinum ware must be handled with clean tongs and protected from contamination from table tops, and so forth. All glassware used in analyses must be equally clean and protected.

10.3 Ash Dissolution—Two methods of dissolving the ash samples are offered for this test method. The analyst may choose the method most appropriate for their laboratory and instrumentation. Laboratories using the fusion method (see 10.3.1) for dissolving the ash should be aware that a considerable amount of sulfur may be lost from the ash during the fusion process. A blank test solution containing the same concentration of reagents used for the ash samples shall be prepared and analyzed with the ash sample solutions.

10.3.1 *Sample Fusion and Dissolution*—Weigh 0.1g (to the nearest 0.1mg) of the ash sample as prepared in 9.1 or 9.2 into a platinum dish (or crucible) (see Note 2). Weigh 0.4g (to

nearest 0.5 mg) of the fluxing agent and add to the ash sample. Mix the ash and fluxing agent thoroughly and heat to melting at 1000 to  $1200^{\circ}$ C with stirring, according to 10.3.1.1 or 10.3.1.2, until a clear melt is obtained.

10.3.1.1 If a muffle furnace is used for heating, place the platinum dish in a clean silica or refractory tray and place in a muffle furnace preheated to 1000°C; 7 min at this temperature is sufficient to fuse most mixtures completely, but heating should be continued until a clear pellet is obtained. Use platinum-tipped tongs to swirl the melt gently to dissolve the ash. Remove the tray with the dish and cool to room temperature. Carefully rinse the bottom and outside of the platinum dish to remove possible contamination; then place is in a clean 250- or 400-mL beaker. Place a clean TFE-fluorocarbon coated magnetic stirring bar in the platinum dish and add 50 mL of 5 + 95 HNO<sub>3</sub> (see 7.5.4) to the melt in the platinum dish. Immediately place the beaker with the dish on the stirring hotplate. Stir and heat the solution to just below boiling and maintain this near boiling condition until the melt is dissolved or for not more than 30 min (see Note 3). Remove the platinum dish from the beaker, rinse the dish with small amounts of reagent water, and quantitatively transfer the solution to a 100-mL volumetric flask. Add 1 mL of internal standard to the flask and dilute to the 100-mL mark with water. This solution is 1000 ppm with respect to the total sample and contains 4 g/Lof fluxing agent.

NOTE 2—Graphite crucibles may be used instead of platinum for the fusion. The graphite crucibles are not to be immersed in the digestion solution. Pour the red-hot melt directly from the crucible into the acid solution and proceed with stirring and heating as written above.

NOTE 3—If the stirring is not constantly maintained, some of the constituents may precipitate, primarily silicic acid, as a result of heating in the highly acidic solution. The analysis must then be repeated.

10.3.1.2 If a flame is used for heating, rotate the platinum dish in the flame until a clear melt is obtained. If automated fusion equipment is being used, follow the manufacturer's programmed steps. If the crucible is inserted manually into the flame using platinum-tipped tongs, stir by swirling for at least 5 min. When a clear melt is obtained, either pour the hot melt into 50 mL of 5 + 95 nitric acid (see 7.5.4) in a clean 250- or 400-mL beaker containing a Teflon-coated magnetic stirring bar or cool the crucible and transfer the solid pellet to this solution. (It is the analyst's responsibility to ensure that the entire sample is transferred to the nitric acid solution). Immediately place the beaker on a stirring hot plate. Stir and heat the solution to just below boiling and maintain the near boiling condition until the pellet is dissolved or for not more than 30 min (see Note 3). Transfer the solution quantitatively to a 100-mL volumetric flask. Add 1 mL of internal standard to the flask and dilute to the 100-mL mark with water. This solution is 1000 ppm with respect to the total sample and contains 4 g/Lof fluxing agent.

10.3.2 *Mixed Acid Dissolution*—Weigh 0.1g (to the nearest 0.1 mg) of the ash sample as prepared in 9.1 or 9.2 into a 250-mL polycarbonate bottle with an O-ring seal and screw cap 9see Note 4. The bottle should be capable of withstanding a temperature up to 130°C, the pressure developed during digestion, and resistant to oxidation. (Warning—With repeated use the polycarbonate bottles will become brittle and

develop cracks. They should be inspected before each use. A convenient way to do this is to hold them up to a light source. If any evidence of cracks is noted, the bottle should be discarded.)

NOTE 4— Some combustions residues may contain sulfite sulfur. If sulfite is known to be present or is suspected, add 1-mL of  $30\% H_2O_2$  to the digestion bottle before proceeding to 10.3.2.1. The peroxide will oxidize sulfite species to sulfate which is quantitatively retained in the digestion process. If peroxide is added, make the appropriate adjustment to the final sample volume used in the calculation of results in Section 12.

10.3.2.1 Add 5.0 mL of the 70/30 HCl/HF mixed acid solution (7.5.5) and 2.0 mL of HNO<sub>3</sub> to the sample and tighten the screw cap (see Note 5). Heat the bottle at 100 to 130°C in a boiling water bath, on a steam bath, or in an oven for at least 2 h. Remove the bottle from the heat source, and add 93.0 mL of 1.5% boric acid (H<sub>3</sub>BO<sub>3</sub>) solution. Return the bottle to the heat source and continue heating for 1 h. Cool the solution to room temperature before analysis. If the samples are not analyzed immediately, they may be stored in their original digestion bottles or transferred to polyethylene bottles. Prepare a method blank using the above procedure.

NOTE 5—The 70/30 HCl/HF mixed acid solution (see 7.5.5) can be prepared and stored until use, whereas an aqua regia mixture (HCl and HNO<sub>3</sub>) is not stable. Using the mixed acid solution and concentrated HNO<sub>3</sub> is equivalent to using aqua regia and HF.

10.3.3 Prepare calibration standards using appropriate values of standard stock solutions (see 7.3). Add 1-mL internal standard solution (see 7.4) per 100-mL volume used. Dilute to the mark with the proper diluents.

#### **11. Instrument Operation**

11.1 Consult the manufacturer's instructions for operation of the ICP spectrometer. The present method assumes that good operating procedures are followed. Design differences among instruments and different selected analytical wavelengths for individual spectrometers make it impractical to list detailed conditions.

11.2 To ensure the validity of the data obtained from an ICP analysis, the following QC elements shall be considered the minimum for each analyte wavelength.

11.2.1 Initial and periodic instrument performance verification (also to be performed after major maintenance):

11.2.1.1 All manufacturer specified spectral alignment practices (such as Mercury lamp alignment) shall be followed.

11.2.1.2 The reference peak intensity shall be monitored following the manufacturer recommendations. A Manganese solution is often used for this purpose.

11.2.1.3 The minimum detectable limit shall be verified every 6 months for analytes previously determined to be within ten times the minimum detectable limit. The minimum detectable limit must be less than or equal to the reporting limit.

11.2.1.4 Select peak wavelengths to minimize/eliminate spectral interferences.

11.2.1.5 Inter-element interference corrections (spectral interferences) and background point corrections shall be verified every 3 months according to manufacturer specifications. 11.2.2 *Calibration* :

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11.2.2.1 All analysis results must fall within the concentration range of the calibration standards. If a sample result occurs above the high calibration standard, dilute the sample and reanalyze for that element.

11.2.2.2 All calibration solutions shall be matrix matched (in relation to the dissolution background such as  $LiB_4O_7$  and acids) to the sample solutions.

11.2.2.3 The calibration shall include a minimum of a calibration blank and three calibration standard concentrations, assuming a linear calibration. The recommended relative concentrations for the calibration standards are:

(1) The middle standard should be near the mid-point of the expected sample concentration range.

(2) The high standard should be approximately two times the middle standard

(3) The low standard should be approximately one-tenth  $(\frac{1}{10})$  of the middle standard.

(4) The linear correlation of the calibration regression shall be 0.995 or greater.

Analyte	Calcium	Silicon
Estimated sample concentration	Calcium 10 mg/L to 20 mg/L in solution	180 mg/L to 230 mg/L in solution
Recommended middle standard concentration	15 mg/L	200 mg/L
Recommended high standard concentration	30 mg/L	400 mg/L
Recommended low standard concentration	1.5 mg/L	20 mg/L

#### 11.2.3 Initial calibration verification:

11.2.3.1 A successful calibration shall be verified with an initial calibration verification standard(s) and the calibration blank prior to the analysis of any samples.

11.2.3.2 The initial calibration verification recovery shall be within 5% of the known value. See 7.12 and 7.13.

11.2.3.3 The initial calibration blank reported concentration shall be below the reporting limit.

11.2.4 Periodic calibration verification:

11.2.4.1 The calibration shall be verified after every 10th analysis and at the end of the batch or shift using a periodic calibration verification standard(s) and a calibration blank.

11.2.4.2 The periodic calibration verification recovery shall be within 10% of the known value. See 7.12 and 7.13.

11.2.4.3 The periodic calibration blank reported concentration shall be below the reporting limit.

11.2.5 If a calibration verification fails to meet the criteria, it shall be rerun once. If it still fails the calibration is suspect and any samples analyzed after the last acceptable calibration verification shall be re-analyzed.

11.2.6 Preparation batch Quality Control Checks:

11.2.6.1 Various checks are necessary to ensure that the dissolution process applied to the samples provides accurate recovery without contamination.

11.2.6.2 All preparation batch quality control shall be performed once for each batch or for every 40 samples, whichever is more frequent.

11.2.6.3 *Method blank*—Absolute results for this blank shall be less than the reporting limit.

11.2.6.4 *Preparation duplicates*—A sample prepared in duplicate following the procedure in section 10 of this standard. If the duplicates fail to meet the repeatability specifications of this test method, reanalyze the sample solution and the duplicate solution once. If these results still fail to meet the repeatability specifications consider the preparation batch in question and investigate the problem.

11.2.6.5 Secondary Control sample—A secondary measurement standard shall be processed following the procedure in section 10 of this standard. Results for the control sample shall be within the ASTM Reproducibility limits and within the laboratory's process control limits (as defined in ASTM Manual  $7^6$  or other appropriate process control limit definition).

11.2.6.6 *Primary Control sample*—A primary measurement standard shall be processed through the entire sample digestion scheme. This sample shall be performed a minimum of once per quarter. Results for the primary control sample shall be within the ASTM Reproducibility limit and within the laboratory's process control limit (as defined in ASTM Manual 7 or other appropriate process control limit definition).

11.2.7 Secondary QC verifications—Post analysis verifications include verification of the sum of the oxides as a weight percent of the sample. The undetermined content shall not exceed 5% when all major and minor analytes and SO3 are included. If the undetermined value exceeds 5%, the analysis shall be considered suspect, and verification steps shall be taken when the cause for a high undetermined percent is not already known.

# 12. Calculation or Interpretation of Results

12.1 Calculate the percentage (by weight) of each element in the ash using the following equation:

$$\% E = (C \times V)/W \times D \times 100 \tag{1}$$

where:

$$E$$
 = element analyzed,

- C = concentration in mg/L (ppm or g/g) of M in the analyzed solution,
- V = volume (in litres) of sample solution prepared in Section 10,

W = weight of sample in milligrams, and

D = dilution factor; = final volume of analyzed solution divided by the amount of the prepared solution (see Section 10) used for the dilution.

12.2 Use Practice D3180 to calculate as-determined results to other bases.

12.3 Convert concentrations in the ash to the dry sample basis for reporting as follows:

$$C = A \times B/100 \tag{2}$$

where:

C = percent of element in the dry sample,

A = percent of element determined in the ash, and

B = % ash in the dry sample.

<sup>&</sup>lt;sup>6</sup> Manual of Presentation of Data and Control Chart Analysis, ASTM MNL7A, ASTM International, 2002.

#### TABLE 3 Concentration Ranges and Limits for Repeatability and Reproducibility for Major and Minor Elemental Oxides in Ash from Coal, Coke, and Solid Combustion Residues

Note—The precision and bias study for SO3 was performed only by the mixed acid dissolution (10.3.2) and does not include data from fusion and dissolution (10.3.1).

Elemental Oxide	Concentration Range	Repeatability Limit ( <i>r</i> )	Reproducibility Limit ( <i>R</i> )
$SiO_{2}$ $Al_{2}O_{3}$ $Fe_{2}O_{3}$ $MgO$ $CaO$ $TiO_{2}$ $K_{2}O$ $P_{2}O_{5}$ $Na_{2}O$	2.04-73.73 % 1.04-29.54 % 0.39-47.94 % 0.40-7.29 % 1.04-44.03 % 0.06-1.47 % 0.09-2.53 % 0.10-1.34 % 0.17-7.44 %	$\begin{array}{c} -0.13 + 0.09 \ \bar{x}^{A} \\ 0.17 + 0.06 \ \bar{x}^{A} \\ 0.13 \ \bar{x}^{A} \\ 0.02 + 0.08 \ \bar{x}^{A} \\ 0.01 \ \bar{x}^{A} \\ 0.02 + 0.07 \ \bar{x}^{A} \\ 0.06 + 0.11 \ \bar{x}^{A} \\ 0.06 + 0.11 \ \bar{x}^{A} \\ 0.06 + 0.09 \ \bar{x}^{A} \end{array}$	$\begin{array}{c} 2.00 + 0.10 \ \bar{x}^{A} \\ 0.86 + 0.07 \ \bar{x}^{A} \\ 0.23 \ \bar{x}^{A} \\ 0.11 + 0.11 \ \bar{x}^{A} \\ 0.25 \ \bar{x}^{A} \\ 0.05 + 0.12 \ \bar{x}^{A} \\ 0.14 + 0.30 \ \bar{x}^{A} \\ 0.11 + 0.31 \ \bar{x}^{A} \\ 0.10 + 0.17 \ \bar{x}^{A} \end{array}$
MnO <sub>2</sub> BaO BaO SrO SO <sub>3</sub> <sup>B</sup>	198–834 ppm 266–950 ppm 0.13–3.00 % 285–10460 ppm 0.14–10.0	0.16 $\bar{x}^{A}$ 0.07 $\bar{x}^{A}$ 0.17 $\bar{x}^{A}$ 33 + 0.076 $\bar{x}^{A}$ 0.03 $\bar{x}$ +0.06	0.42 $\bar{x}^{A}$ 190 ppm 0.30 $\bar{x}^{A}$ 73 + 0.164 $\bar{x}^{A}$ 0.08 $\bar{x}$ +0.06

<sup>A</sup>Where  $\bar{x}$  is the average of two single test results.

<sup>B</sup>SO<sub>3</sub> vales are applicable to mixed acid dissolution only. See 10.3.

### 13. Precision and Bias

13.1 *Precision*—The precision of this test method for the determination of major and minor elements in ash from coal, coke, and solid combustion residues are shown in Table 3. The precision characterized by the repeatability  $(S_r, r)$  and reproducibility  $(S_R, R)$  is described in Table A1.1 in the Annex A1.

13.1.1 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 from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

13.1.2 *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 %.

13.2 *Bias*—A standard reference material (1633b-coal fly ash) from the National Institute for Standards and Technology

(NIST) was included in the ICP interlaboratory study to ascertain possible bias between reference material values and those determined by the new method. A comparison of the NIST values and those obtained in the interlaboratory study are given in Table 4. The results show a very small (positive) bias

TABLE 4 Comparison of Certified Values for Standard Reference
Material 1633b with Interlaboratory Study Values for Major and
Minor Elemental Oxides in Ash from Coal, Coke, and Solid
Combustion Besidues

	Combustion neside	103
Elemental	ICP-RR	NIST
Oxide	Value	Value
SiO <sub>2</sub>	50.47 ± 1.21	49.25 ± 0.17
Al <sub>2</sub> O <sub>3</sub>	$29.00 \pm 0.63$	$28.44 \pm 0.51$
Fe <sub>2</sub> O <sub>3</sub>	$11.92 \pm 0.35$	$11.12 \pm 0.33$
MgO	$0.81 \pm 0.027$	$0.80 \pm 0.01$
CaO	$2.28 \pm 0.16$	$2.11 \pm 0.08$
TiO <sub>2</sub>	$1.32 \pm 0.037$	$1.32 \pm 0.02$
K <sub>2</sub> O	$2.33 \pm 0.067$	$2.35 \pm 0.04$
P <sub>2</sub> O <sub>5</sub>	$0.61 \pm 0.036$	0.53 <sup>A</sup>
Na <sub>2</sub> O	$0.27 \pm 0.04$	$0.27 \pm 0.004$
MnO <sub>2</sub> <sup>B</sup>	$207.3 \pm 5.6$	$208.6 \pm 2.7$
BaO <sup>B</sup>	821 ± 38	792 ± 30
SrO <sup>B</sup>	$1237 \pm 54$	1231 ± 17
SO3	$0.51\pm0.02$	$0.52\pm0.01$

<sup>A</sup>Noncertified value.

<sup>B</sup>Value in ppm.

for the reported values for iron.

13.3 An interlaboratory study, designed consistent with Practice E691, was conducted in 1997, and twelve labs participated.<sup>7</sup>

13.4 A second interlaboratory study, designed consistent with Practice E691, was conducted in 2003. The purpose of the interlaboratory study was to include sulfur in the suite of elements analyzed by ICP-AES. The details of the study and supporting data are given in a Research Report filed at ASTM headquarters.<sup>8</sup>

# 14. Keywords

14.1 coal; coal ash; inductively coupled plasma-atomic emission spectrometry; major and minor element

<sup>&</sup>lt;sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1035.

<sup>&</sup>lt;sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1032.

# 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 materials described in Table A1.1.

A1.3 *Reproducibility Standard Deviation*  $(S_R)$ —The standard deviation of test results obtained under reproducibility conditions.

A1.2 Repeatability Standard Deviation  $(S_r)$ —The standard deviation of test results obtained under repeatability conditions.

TABLE A1.1 Repeatability (S	,, r) and Reproducibility (S <sub>R</sub> , R) Parameters Used for Calculation of Precision Stateme	₽nt
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Material	Average	Sr	SiO₂ SR		R	Material	Average	Sr	Al <sub>2</sub> O <sub>3</sub> SR		R
Material	Average	51	эн	r	п	waterial	Average	51	3R	r	п
SL 16	2.04	0.1757	0.9016	0.4919	2.5245	SL 16	1.04	0.1176	0.2075	0.3293	0.5811
FBC 9	24.96	0.8585	1.3907	2.4039	3.8941	FBC 9	9.30	0.4087	0.5491	1.1445	1.5376
91three	25.34	0.8172	1.4299	2.2883	4.0036	1635	12.80	0.2692	0.6918	0.7537	1.9371
89one	27.76	0.7742	2.0337	2.1677	5.6943	89one	13.58	0.4246	1.0916	1.189	3.056
WAL 1	30.49	0.7935	1.537	2.2219	4.3036	90five	15.88	0.3715	0.625	1.0402	1.7501
WAL 6	30.78	0.7847	1.6418	2.197	4.5971	91three	17.28	0.471	1.0166	1.3188	2.8464
90five	34.69	1.2085	2.1835	3.3837	6.1137	WAL 7	18.37	0.5599	0.7047	1.5678	1.9731
1635	35.17	0.8463	2.4193	2.3695	6.7741	WAL 1	19.38	0.3037	0.5593	0.8505	0.5661
2776	47.49	1.819	2.2528	5.0931	6.3078	WAL 6	19.69	0.2795	0.5646	0.7825	1.5809
1632c	49.31	1.6403	2.5153	4.5927	7.0429	WAL 4	23.06	0.2947	0.7231	0.825	2.0246
1633b	50.47	1.2066	1.8074	3.3785	5.0608	1632c	24.08	0.4669	0.8512	1.3073	2.3834
WAL 4	55.66	1.3734	2.9156	3.8455	8.1638	2776	28.20	0.9721	1.5042	2.722	4.2117
89ten	58.28	2.1858	2.7337	6.1201	7.6545	1633b	29.00	0.6332	0.918	1.773	2.5703
WAL 7	63.73	2.1713	3.2922	6.0797	9.2183	89ten	29.54	0.8364	0.9418	2.3419	2.637
		F	-e <sub>2</sub> O <sub>3</sub>						MgO		
Material	Average	Sr	SR	r	R	Material	Average	Sr	SR	r	R
SL 16	0.39	0.0494	0.0873	0.1382	0.2445	91three	0.40	0.022	0.055	0.061 46	0.153 88
89ten	4.21	0.2511	0.449	0.7031	1.2572	SL 16	0.52	0.0221	0.0299	0.061 94	0.083 77
90five	4.98	0.2102	0.3113	0.5886	0.8716	89one	0.60	0.0271	0.0406	0.075 9	0.113 54
WAL 7	5.02	0.1823	0.2039	0.5105	0.571	1632c	0.79	0.0346	0.0557	0.096 93	0.155 81
WAL 4	5.03	0.1993	0.2765	0.5581	0.7743	1633b	0.82	0.0271	0.0388	0.075 9	0.108 52
	5.30	0.1498	0.2647	0.4195	0.741	89ten	0.83	0.0324	0.056	0.090 76	0.156 80
WAL 6			0.4979	0.779	1.394	2776	1.03	0.0395	0.116	0.110 48	0.324 89
WAL 6 FBC 9	5.91	0.2782	0.4979				2.03	0.0742	0.2447	0.207 73	0.685 13
FBC 9		0.2782 0.2527	0.3738	0.7075	1.0467	WAL 7	2.03	0.0742	0.2447	0.201 10	
FBC 9	5.91			0.7075 0.6692	1.0467 1.0061	WAL 7 WAL 4	2.03	0.0483	0.1237	0.135 36	0.346 29
FBC 9 WAL 1 1635	5.91 6.46	0.2527	0.3738								
FBC 9 WAL 1	5.91 6.46 7.23	0.2527 0.239	0.3738 0.3593	0.6692	1.0061	WAL 4	2.47	0.0483	0.1237	0.135 36	0.346 29
FBC 9 WAL 1 1635 91three	5.91 6.46 7.23 9.45	0.2527 0.239 0.4187	0.3738 0.3593 0.6115	0.6692 1.1723	1.0061 1.7122	WAL 4 1635	2.47 3.27	0.0483 0.104	0.1237 0.1369	0.135 36 0.291 18	0.346 29 0.383 42 0.589 36
FBC 9 WAL 1 1635 91three 1633b	5.91 6.46 7.23 9.45 11.92	0.2527 0.239 0.4187 0.3534	0.3738 0.3593 0.6115 1.2383	0.6692 1.1723 0.9896	1.0061 1.7122 3.4673	WAL 4 1635 FBC 9	2.47 3.27 3.34	0.0483 0.104 0.1144	0.1237 0.1369 0.2105	0.135 36 0.291 18 0.320 34	0.346 29 0.383 42

# ∰ D6349 – 09

TABLE A1.1 Continued

Interini         Average         Sr						TADLE AT		θu				
Betern (1835)         1.0075         0.0299         0.14137         0.00849         0.2987         5L.16         0.0081         0.0201         0.01417         0.014407         0.014407         0.014407         0.014407         0.014407         0.014407         0.014407         0.014407         0.014407         0.004400         0.00245         0.0214         0.01417         0.014407         0.014107         0.014107         0.01411         0.01411         0.014111         0.014111         0.01411         0.014111         0.01411         0.01411         0.01411         0.01411         0.01411         0.01411         0.01411         0.01411         0.01411				CaO						TiO <sub>2</sub>		
1633b       2.2183       0.167       0.2047       0.4677       0.6732       FEC 9       0.44       0.0251       0.0304       0.070 30       0.084 80       0.0876 80       0.148 43         Bone       3.2775       0.145       0.3560       0.4061       0.4825       Blane       0.73       0.0248       0.0304       0.078 30       0.028 80       0.135 84         WAL 4       8.3385       0.3040       0.4863       0.8838       1.5502       1.552       1.003       0.0714       0.078 80       0.028 80       0.128 81         WAL 4       8.3385       0.5040       0.5755       1.8861       3.557       916/ree       1.28       0.0304       0.078 80       0.0273       0.010 81       0.127 21         WAL 1       2.2877       1.8804       2.587       916/ree       1.32       0.0374       0.078 80       0.0278 50       0.272 40       0.068 0       0.128 80       0.776 41         St 1.62       1.4271       0.2755       1.8402       2.877       1.835       1.32       0.0374       0.068 90       0.176 40       0.272 40       0.064 0       0.069 80       0.176 40       0.273 40       0.466 2.273 40       0.146 0       0.273 40       0.466 2.273 40       0.166 0       0	Material	Average	Sr	SR	r	R	Material	Average	Sr	SR	r	R
1633b       2.2183       0.167       0.2047       0.4677       0.6732       FEC 9       0.44       0.0251       0.0304       0.070 30       0.084 80       0.0876 80       0.148 43         Bone       3.2775       0.145       0.3560       0.4061       0.4825       Blane       0.73       0.0248       0.0304       0.078 30       0.028 80       0.135 84         WAL 4       8.3385       0.3040       0.4863       0.8838       1.5502       1.552       1.003       0.0714       0.078 80       0.028 80       0.128 81         WAL 4       8.3385       0.5040       0.5755       1.8861       3.557       916/ree       1.28       0.0304       0.078 80       0.0273       0.010 81       0.127 21         WAL 1       2.2877       1.8804       2.587       916/ree       1.32       0.0374       0.078 80       0.0278 50       0.272 40       0.068 0       0.128 80       0.776 41         St 1.62       1.4271       0.2755       1.8402       2.877       1.835       1.32       0.0374       0.068 90       0.176 40       0.272 40       0.064 0       0.069 80       0.176 40       0.273 40       0.466 2.273 40       0.146 0       0.273 40       0.466 2.273 40       0.166 0       0	80ton	1 0375	0 0200	0 1/13	0.0838	0 3957	SI 16	0.06	0.0053	0.0231	0.014.87	0.064.80
1632c       2.8042       0.0657       0.1329       0.0481       0.0268       0.01341       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.01354       0.0268       0.0358       0												
Bönen         3.2775         0.1461         0.03280         0.4061         0.0228         1.682         Bönen         7.7         0.02260         0.02860         0.03380         0.02131           WAL 7         5.9115         0.2381         0.4085         0.6868         0.6828         1.5707         WAL 7         1.01         0.0332         0.07761         0.07761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.03761         0.04861         0.0327         0.10461         0.02821         0.02761         0.02761         0.02761         0.04971         0.10461         0.02826         0.0374         0.03641         0.04861 </td <td></td>												
2776         3.4013         0.0814         0.4185         0.228         1.1634         WAL 4         0.85         0.0198         0.0781         0.0238         0.0282         0.0338         0.0282         0.1358         0.0282         0.0338         0.0282         0.1358         0.0338         0.0282         0.1358         0.1358         0.1358         0.1358         0.1358         0.1358         0.1358         0.1358         0.1358         0.0274         0.0474         0.0768         0.0338         0.0338         0.0338         0.0338         0.0338         0.0338         0.0338         0.0338         0.0138         0.0138         0.17721           WAL 1         2.2144         0.6577         1.0357         1.0374         0.0164         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0166         0.0164         0.0166         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01734         0.0166         0.01746         0.01734         0.0166 </td <td></td>												
WAL 7         5.9115         0.2381         0.4489         0.6665         1.3707         WAL 7         1.01         0.0323         0.0465         0.06297         0.132 67           173.5         17.2344         0.4666         0.5246         1.1358         2.5888         WAL 1         1.17         0.0353         0.100 31         0.172 2         0.141 00         0.2297         0.0148         0.0653         1.02297         0.0148         0.0653         0.102 47         0.0448         0.0653         0.102 47         0.0414         0.00274         0.0474         0.0473         0.01297         0.112 67         0.01297         0.01297         0.0124         0.0141         0.0098         0.112 67         0.0163         0.0174         0.0174         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0164         0.0174         0.0164         0.0174         0.0164         0.0174         0.0164         0.0174         0.0164         0.0174         0.0164         0.0174         0.0174         0.0174         0.0174         0.0174         0.0174         0.0174         0.01714         0.0174         0.01714												
WAL4         8.3385         0.3049         0.5893         0.4502         1.09         0.0274         0.0474         0.075 E8         0.177 21           WAL6         20.0223         0.6715         1.2575         1.8501         3.549         90five         1.28         0.0364         0.0821         0.0857         0.227 67           WAL1         2.2174         0.5000         0.488         F.         1.850         1.22         0.0544         0.0673         0.116 47         0.1664         0.227 67           UPL         2.2458         4.822         2.776         1.520         0.0444         0.2276         1.22         0.0414         0.126 67         0.126 67         0.126 67         0.126 67         0.0414         0.0734         0.126 67         0.167 64         0.176 61         0.167 64         0.176 61         0.176 61         0.176 61         0.176 61         0.177 21         1.128         0.0448         0.0414         0.176 61         0.017 61         0.578         7         R         Material         Average         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S         S												
1635       17.2344       0.4066       0.12846       1.1389       2.5888       VAL 1       1.17       0.0356       0.0633       0.100 31       0.1272         90five       21.8719       0.5909       0.9488       1.6544       2.6567       91three       1.32       0.0304       0.0623       0.114 100       0.2297         90five       32.458       0.8782       1.7329       2.4588       4.852       2.776       1.32       0.0447       0.0474       0.116 46       0.7744       0.124 66       0.778 49         181       33.5577       1.977       6.3473       10.38       88ten       1.47       0.0385       0.0146       0.0128 0       0.0273       0.0146       0.124 26       0.124 26       0.124 26       0.124 26       0.124 26       0.126       0.014       0.028 0       0.024 26       0.024 0       0.004       0.0146 6       0.024 27       0.048 60       0.019 36       0.042 7       0.048 60       0.019 37       0.024 9       0.0667       0.0667 1       0.045 72       0.188 26         91three       0.09       0.016 86       0.044 75       0.048 60       0.019 37       0.031 0       0.027 1       0.056 27       0.0667       0.0667 1       0.045 70       0.181 26 <td></td>												
WAL6         20.0223         0.036 07         0.027         0.036 07         0.027 65           WAL1         22.8144         0.0672         1.0270         0.0376 65         0.0776         1.32         0.0374         0.0673         0.1416 07         0.1616 07         0.0270 65           WAL1         22.8144         0.6572         1.0270         5.4983         1.32         0.0374         0.0281 7         0.1614         0.0164 1         0.0498         0.0774 40           PIthee         34.2450         0.7872         5.2923         1.7443         WAL6         1.36         0.0484         0.0276 7         0.0281 7         0.0281 7         0.0280 7         0.0281 7         0.0281 7         0.0281 7         0.0281 7         0.0281 7         0.0281 7         0.0282 92           Material         Average         F         S         S         S         r         R         Average 7         F         C         0.0481 7 </td <td></td>												
96/hve         21/6719         0.5909         0.9488         1.6444         2.6567         91/hree         1.32         0.0374         0.01742         0.111 00         0.207 65           91/hree         32.459         0.8782         1.7239         2.4588         4.852         2776         1.32         0.0374         0.0374         0.0464         0.2734         0.124 66         0.765 40 <sup>4</sup> SL 16         38.577         1.997         6.2295         5.5923         1.7443         WAL         1.338         0.0357         0.1046         0.0176         0.0273         0.248 98         0.776 40 <sup>4</sup> 91/hree         0.06         0.0136         0.042 75         0.048 80         0.095 7         0.576         r         R           91/hree         0.09         0.016 85         0.042 75         0.045 80         0.019 70         0.057 7         0.057 7         0.027 30         0.021 70         0.037 10         0.053 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 71         0.53 7         0.271 73												
WAL 1         22.8144         0.6572         1.0275         1.8402         2.877         1633b         1.32         0.0373         0.104 67         0.160 47           SL 16         38.3577         1.9973         6.2295         5.5923         17.443         WAL 6         1.38         0.0446         0.0273         0.124 67         0.179 49           FBC 9         4.40221         0.268         3.7107         0.39         Bilen         1.47         0.0385         0.10446         0.0273         0.024 80         0.229 29           Material         Average         Sr         FSC         N         R         Material         Average         Sr         F2O         S.16         0.023 90         0.243 50         0.042 75         0.445 80         0.019 70         0.063 70         0.053 70         0.273 00         0.210 80         0.071 10         0.053 70         0.210 40         0.0118 30         0.016 80         0.0116 30         0.021 70         0.066 70         0.021 70         0.021 70         0.021 80         0.071 10         0.052 81         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80         0.011 80												
91thce         32,459         0.8782         1.7229         2.4688         4.452         2776         1.32         0.0446         0.2734         0.124 68         0.776 49           FBC 9         44.0321         0.2669         3.7107         6.3473         10.39         89ten         1.47         0.0385         0.1046         0.0176         0.292 92           Material         Average         Sr         K-0         Sr         R         R         Material         Average         Sr         SR         r         R           911tree         0.00         0.013 66         0.002 25         0.038 26         0.090 59         FBC 9         0.10         0.0138         0.0057         0.058 77         0.243 24           901tw         0.39         0.42 26         0.084 79         0.120 00         0.237 40         89one         0.19         0.0251         0.0752         0.070 16         0.214 43           WAL 6         0.318         0.117 08         0.524 16         WAL 16         0.313         0.0251 50         0.530         0.0271 60         0.021 56         0.014 80         0.021 56         0.014 80         0.021 56         0.0171 60         0.284 24         0.0173         0.064 20         0.020 57         0.0260	90five	21.6719	0.5909	0.9488	1.6544	2.6567	91three	1.32	0.0504	0.0742	0.141 00	0.207 65
SL 16         38.3577         1.9973         6.2265         5.5923         17.443         WAL 6         1.36         0.0385         0.0641         0.0998         0.179 49           Material         Average         Sr         P.05         r         R           Olimon         0.031 66         0.032 25         0.098 26         0.099 59         FBC 9         0.10         0.0118         0.033 7         0.028 27         0.046 27           St. 16         0.09         0.013 66         0.042 75         0.045 80         0.119 70         896ne         0.13         0.024 90         0.0073 1         0.053 97         0.227 10           1835         0.44         0.041 81         0.118 70         0.117 70         0.224 14         WAL 6         0.311         0.024 90         0.0071 0         0.024 70         0.080 70         0.027 70         0.021 43           WAL 6         0.33         0.041 82         0.014 12         0.112 70         0.523 16         WAL 6         0.311         0.022 90         0.027 80         0.021 43           WAL 6         0.33         0.041 82         0.114 25         0.123 20         0.55 80         0.077 80         0.027 80         0.213 44           WAL 7         0.051 20	WAL 1	22.8144	0.6572	1.0275	1.8402	2.877	1633b	1.32	0.0374	0.0573	0.104 67	0.160 41
FBC 9         44.0321         0.2669         3.7107         6.3473         10.39         89ten         1.47         0.0385         0.1046         0.107.68         0.292 92           Material         Average         Sr         K20         SR         r         R         Material         Average         Sr         P20         SR         r         R           911tree         0.09         0.013 66         0.032 35         0.038 26         0.090 50         FBC 9         0.10         0.0119         0.013         0.0274         0.045 80         0.271 70         0.044 80         0.0752         0.0752         0.0710 16         0.271 70         0.271 70         0.0254 16         WAL 1         0.053 0         0.0721         0.066 80         0.211 70         0.386 80         WAL 1         0.00         0.046 80         0.211 70         0.386 80         WAL 1         0.40         0.0244         0.0791         0.057 25         0.201 35           WAL 6         0.133         0.026 40         0.181 00         0.172 45         0.386 81         Y776         0.40         0.0264 0         0.0719         0.057 25         0.201 35           WAL 6         0.033         0.0379         0.041 90         0.141 45         0.585 7 <td< td=""><td>91three</td><td>32.459</td><td>0.8782</td><td>1.7329</td><td>2.4588</td><td>4.852</td><td>2776</td><td>1.32</td><td>0.0446</td><td>0.2734</td><td>0.124 86</td><td>0.765 40<sup>A</sup></td></td<>	91three	32.459	0.8782	1.7329	2.4588	4.852	2776	1.32	0.0446	0.2734	0.124 86	0.765 40 <sup>A</sup>
FBC 9         44.0321         0.2669         3.7107         6.3473         10.39         89ten         1.47         0.0385         0.1046         0.107.68         0.292 92           Material         Average         Sr         K20         SR         r         R         Material         Average         Sr         P20         SR         r         R           911tree         0.09         0.013 66         0.032 35         0.038 26         0.090 50         FBC 9         0.10         0.0119         0.013         0.0274         0.045 80         0.271 70         0.044 80         0.0752         0.0752         0.0710 16         0.271 70         0.271 70         0.0254 16         WAL 1         0.053 0         0.0721         0.066 80         0.211 70         0.386 80         WAL 1         0.00         0.046 80         0.211 70         0.386 80         WAL 1         0.40         0.0244         0.0791         0.057 25         0.201 35           WAL 6         0.133         0.026 40         0.181 00         0.172 45         0.386 81         Y776         0.40         0.0264 0         0.0719         0.057 25         0.201 35           WAL 6         0.033         0.0379         0.041 90         0.141 45         0.585 7 <td< td=""><td>SL 16</td><td>38.3577</td><td>1.9973</td><td>6.2295</td><td>5.5923</td><td>17.443</td><td>WAL 6</td><td>1.36</td><td>0.0357</td><td>0.0641</td><td>0.099 88</td><td>0.179 49</td></td<>	SL 16	38.3577	1.9973	6.2295	5.5923	17.443	WAL 6	1.36	0.0357	0.0641	0.099 88	0.179 49
Material         Average         Sr         SR         r         R         Material         Average         Sr         P2.0           SI, 16         0.09         0.013 66         0.042 35         0.036 50         0.019 0         0.019 0         0.053 97         0.242 92           SI, 16         0.09         0.013 66         0.042 75         0.045 80         0.019 70         0.019         0.0241         0.0697         0.0242 0         0.0697         0.0242 1         0.0697         0.0242 1         0.0697         0.0242 1         0.0647         0.0249 1         0.0647         0.0241 1         0.0647         0.0241 6         0.0647         0.0647 6         0.0648 6         0.018 1         0.210 43           WAL 6         0.73         0.056 45         0.113 87         0.153 02         0.388 86         WAL 1         0.40         0.0205 0         0.0647         0.0648 6         0.021 55           WAL 7         0.93         0.065 62         0.118 277         0.384 80         0.515 0         1832b         0.53         0.0376         0.0772         0.0768 0         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0.221 94         0												
Material         Average         Sr         SR         SR         r         R         Material         Average         Sr         SR         r         R           91three         0.09         0.013 66         0.023 25         0.038 26         0.039 0         PRC 9         0.10         0.019 0         0.0657         0.243 24         0.243 24           901we         0.39         0.042 86         0.044 75         0.120 00         0.237 40         890ne         0.19         0.0252         0.06477         0.046 80         0.217 14         0.045 00         0.071 16         0.210 16         0.211 74         0.331         0.0251         0.034 0         0.034 60         0.071 16         0.210 48           WAL 1         0.53         0.037 16         0.046 70         0.141 10         0.237 15         1632         0.330         0.0379         0.0716         0.210 48           WAL 6         0.130 0.065 20         0.142 25         0.386 31         2776         0.40         0.0336         0.0779         0.0762         0.106 0         0.213 42           WAL 4         1.30         0.059 9         0.126 60         0.142 77         0.384 49         1633b         0.1035         0.10032         0.1035         0.1010 40												
91         Number         0.09         0.013 66         0.032 25         0.038 26         0.095 97         FBC 9         0.10         0.0193         0.0731         0.053 97         0.243 24           SL 16         0.09         0.016 36         0.042 75         0.045 80         0.119 70         89ten         0.16         0.0195         0.06677         0.06677         0.06772         0.168 67         0.0672         0.06973         0.06731         0.06731         0.06731         0.06732         0.0712         0.06973         0.064 70         0.161 10         0.221 41         1.0251         1.0752         0.0712         0.064 80         0.221 43           WAL 6         0.731         0.054 65         0.131 87         0.133 02         0.386 66         WAL 1         0.40         0.0234         0.0752         0.0762         0.1066         0.213 55           FPC 9         1.03         0.056 24         0.183 03         0.15746         0.535 65         9         Whree         0.036         0.1032         0.10140         0.243 84           MAL 7         0.040 126         0.191 04         0.243 10         0.157 26         0.157 26         0.10140         0.243 84         0.247 947           16320         0.241 14         0.233 44			-			_			_			_
SL 16       0.09       0.016 36       0.042 75       0.04 80       0.119 70       89ten       0.16       0.0195       0.06677       0.026772       0.0186         90five       0.39       0.042 86       0.044 70       0.120 00       0.237 40       0.0251       0.0752       0.0752       0.0771 6       0.2271 71         1635       0.037 18       0.044 70       0.110 10       0.2374 40       WAL 6       0.31       0.0251       0.0752       0.0762       0.069 73       0.069 61       0.2271 71         WAL 6       0.73       0.054 65       0.131 67       0.133 02       0.388 66       WAL 1       0.40       0.0234       0.0772       0.096 80       0.201 55         FBC 9       1.03       0.056 24       0.113 03       0.157 48       0.512 50       1632c       0.631       0.0379       0.0762       0.106 06       0.213 42         VRL 4       0.023 00 90       0.126 60       0.142 77       0.354 48       1633b       0.051       0.0355       0.04890       0.0779       0.2424 02         VRL 4       0.237 00       0.241 41       0.238 94       0.114       0.238 04       0.555 78       91hree       0.76       0.057       0.227 20       0.227 20       0.227 20	Material	Average	Sr	SR	r	R	Material	Average	Sr	SR	r	R
SL 16       0.09       0.016 36       0.042 75       0.04 80       0.119 70       89ten       0.16       0.0195       0.06677       0.026772       0.0186         90five       0.39       0.042 86       0.044 70       0.120 00       0.237 40       0.0251       0.0752       0.0752       0.0771 6       0.2271 71         1635       0.037 18       0.044 70       0.110 10       0.2374 40       WAL 6       0.31       0.0251       0.0752       0.0762       0.069 73       0.069 61       0.2271 71         WAL 6       0.73       0.054 65       0.131 67       0.133 02       0.388 66       WAL 1       0.40       0.0234       0.0772       0.096 80       0.201 55         FBC 9       1.03       0.056 24       0.113 03       0.157 48       0.512 50       1632c       0.631       0.0379       0.0762       0.106 06       0.213 42         VRL 4       0.023 00 90       0.126 60       0.142 77       0.354 48       1633b       0.051       0.0355       0.04890       0.0779       0.2424 02         VRL 4       0.237 00       0.241 41       0.238 94       0.114       0.238 04       0.555 78       91hree       0.76       0.057       0.227 20       0.227 20       0.227 20	Olthroo	0.00	0.012.66	0 022 25	0.038.36	0.000.50		0.10	0.0102	0.0721	0.052.07	0 242 24
96/live         0.39         0.042 86         0.084 79         0.120 00         0.237 40         890ne         0.13         0.0249         0.097         0.0697 30         0.271 41           1635         0.45         0.041 11         0.172 00         0.117 00         0.524 16         WAL 6         0.031         0.0221         0.0647         0.0647         0.0647         0.0647         0.0647         0.0648 6         0.113 10           WAL 6         0.73         0.054 65         0.113 10         0.238 68         WAL 1         0.40         0.0346 10         0.0420         0.0205         0.071 9         0.057 25         0.201 58           WAL 4         1.30         0.056 64         0.183 00         0.157 46         0.152 41         0.535 01         0.0351         0.1035         0.1104         0.288 89           Borne         1.49         0.202 66         0.055 13         901re         1.07         0.0351         0.1032         0.148 28         0.283 89         0.288 89         0.277 81         0.277 81         0.277 81         0.277 81         0.277 81         0.278 91         0.288 94         0.555 91         91three         0.077 6         0.178 00         0.248 94         0.555 31         0.571         0.114 10         0.237 10												
1835       0.041 81       0.187 20       0.117 08       0.524 16       WAL 6       0.0215       0.0252       0.06732       0.0071 6       0.210 43         WAL 6       0.73       0.034 65       0.131 87       0.153 02       0.386 66       WAL 1       0.40       0.0320       0.0671 6       0.210 43         WAL 7       0.033       0.056 42       0.133 03       0.157 48       0.512 50       1632c       0.533       0.0379       0.0762       0.106 06       0.213 42         WAL 4       1.30       0.056 24       0.182 03       0.157 48       0.512 50       1632c       0.53       0.0379       0.0762       0.106 06       0.213 42         WAL 4       1.30       0.056 24       0.182 05       0.556 9       911nree       0.70       0.053       0.10360       0.017 42       0.243 24         WAL 7       1.038 0.051 26       0.104 0       0.433 3       0.505 13       901re       1.07       0.0776       0.1712       0.217 84       0.473 47         1632c       1.74       0.057 28       0.241 91       0.085 59       911nree       0.76       0.553       0.1032       0.148 38       0.428 47         1632c       1.74       0.056 3       0.245 19 7       0												
NAL 1         0.03         0.03 18         0.064 70         0.104 11         0.237 15         1635         0.038         0.0232         0.064 70         0.064 96         0.181 26           WAL 6         0.73         0.054 65         0.131 87         0.153 0         0.389 81         2776         0.40         0.0361         0.0719         0.067 25         0.201 56           WAL 4         1.30         0.056 99         0.126 60         0.142 77         0.384 48         1633b         0.61 40         0.0361         0.1035         0.0668         0.620 156           Bisone         1.49         0.202 66         0.565 3         0.565 76         0.858 59         9111ree         0.776         0.1172         0.213 8         0.488 42           1633c         1.74         0.067 30         0.241 91         0.885 59         9111ree         0.776         0.1121         0.2173 8         0.488 41           1633c         2.73         0.141 86         0.487 37         0.396 70         1.094 44         1.34         0.0974         0.2269         0.227 82         0.653 33           391tree         0.77         0.017 56         0.028 40         0.445 35         0.565         1.484         1.574         41.00         1.633b												
WAL 6         0.73         0.054 65         0.131 67         0.153 02         0.386 86         WAL 1         0.40         0.0346         0.072         0.036 80         0.201 55           FBC 9         1.03         0.056 24         0.142 25         0.175 48         0.512 50         1632c         0.035         0.0379         0.0752         0.201 35           FBC 9         1.03         0.056 24         0.183 03         0.555 76         0.858 27         WAL 7         0.76         0.035         0.0869         0.097 92         0.243 24           2776         1.68         1.101 39         0.234 14         0.283 99         91hree         0.76         0.033         0.1032         0.148 38         0.286 94           1632c         1.74         0.057 30         0.241 91         0.188 43         0.677 36         WAL 4         1.34         0.097 80         0.171 8         0.171 80         0.479 47           1633b         2.34         1.067 36         0.286 51         0.997 70         1.084 4         0.677 36         WAL 4         1.34         0.097 80         0.226 9         0.635 33           91three         0.17         0.177 45         0.028 64         0.049 17         0.0080 18         B4ten         1.98         <												
WAL 7         0.93         0.061 62         0.142 25         0.172 55         0.398 31         2776         0.40         0.0205         0.0719         0.067 25         0.201 32           WAL 4         1.30         0.056 24         0.180 30         0.157 48         0.512 50         1632         0.133         0.057 25         0.201 32           WAL 4         1.30         0.056 20         0.366 53         0.565 76         0.858 27         WAL 7         0.035         0.0360         0.0079         0.0224         0.201 32           V101 39         0.2241 14         0.283 90         0.655 59         911mee         0.76         0.035         0.1032         0.148 38         0.2028 94           1633b         2.34         0.067 30         0.241 91         0.184 44         0.677 36         WAL 4         1.34         0.0767 4         0.171 2         0.217 38         0.479 47           1633b         2.34         0.067 30         0.241 91         0.184 44         0.677 36         WAL 4         1.34         0.0767 4         0.177 2         0.217 85         0.479 47         0.229         0.227 82         2.63 33           81616         0.029 14         0.046 85         0.056 10         0.028 39         1.6335         207												
FBC 9         1.03         0.056 24         0.183 03         0.157 48         0.512 50         1632c         0.0379         0.0762         0.106 06         0.213 42           830me         1.49         0.202 06         0.306 53         0.685 76         0.888 27         WAL 7         0.70         0.0351         0.0685         0.0489         0.243 24           2776         1.69         1.013         0.224 14         0.280 0         0.655 59         911me         0.76         0.053         0.0489         0.0433         0.243 24           1633b         2.34         0.057 30         0.241 91         0.188 44         0.677 30         0.479 47         0.357 37         0.397 70         1.046 47         0.0976         0.0766         0.1712         0.217 82         0.479 47           1633b         2.74         0.017 56         0.286 44         0.049 17         0.046 67         WAL 4         1.34         0.097 6         1.852         19.78         51.87           780 tr         Sr         Sr         Sr         Sr         Sr         Sr         Sr         Sr         R         A1.00           1633b         0.27         0.177 6         0.028 44         0.049 17         0.060 16         1633b												
WAL4         1.30         0.050 99         0.126 60         0.142 77         0.354 48         1633b         0.61         0.0381         0.1035         0.101 40         0.289 88           2776         1.69         1.101 39         0.234 14         0.283 90         0.655 59         91three         0.76         0.035         0.0659         0.243 24           1632b         2.34         0.067 30         0.241 91         0.188 44         0.677 86         WAL 4         1.34         0.097 80         0.227 82         0.855 33           83ten         2.53         0.141 68         0.387 37         0.396 70         1.084 64         1.34         0.097 4         0.2269         0.227 82         0.855 33           91three         0.17         0.017 56         0.028 64         0.049 17         0.066 51         0.128 98         f633b         207         5.82         14.84         15.74           PIC 9         0.20         0.020 18         0.046 55         0.069 28         PIC 9         212         15.27         3.58         4.27.4         100.39           1633b         0.27         0.017 74         0.033 49         0.046 6         0.105 20         2.776         27.16         2.16.3         3.89.3         66.16					0.172 55							
880me         1.49         0.202 06         0.306 53         0.656 76         0.658 27         WAL 7         0.70         0.035         0.0869         0.097 20         0.248 24           2776         1.69         1.101 39         0.234 14         0.283 90         0.655 59         91five         0.76         0.053         0.1032         0.148 38         0.479 47           1632b         2.34         0.067 30         0.241 91         0.188 44         0.677 36         WAL 4         1.34         0.0974         0.2269         0.272 82         0.655 33           89ten         2.53         0.114 68         0.387 37         0.396 70         1.084 64         0.677 36         WAL 4         1.34         0.0974         0.2269         0.272 82         0.655 33           91three         0.17         0.017 56         0.028 64         0.049 17         0.081 88         91633b         207         5.62         14.64         15.74         41.003           183ba         0.27         0.017 76         0.038 49         0.046 65         0.105 26         20.029         1632c         275         2.363         38.93         66.16         108.99           1133b         0.27         0.017 76         0.021 74         0.052 57	FBC 9	1.03	0.056 24	0.183 03	0.157 48	0.512 50	1632c	0.53	0.0379	0.0762	0.106 06	0.213 42
2776       1.69       1.101 39       0.234 14       0.283 90       0.655 59       91three       0.76       0.053       0.1032       0.148 38       0.288 94         1632b       1.74       0.067 30       0.241 91       0.184 35       0.655 13       90five       1.34       0.0974       0.2129       0.212 82       0.635 33         B8ten       2.53       0.141 68       0.387 37       0.396 70       1.084 64       1.34       0.0974       0.226 91       0.272 82       0.635 33         Bytime       0.17       0.017 56       0.028 64       0.049 17       0.080 18       89ten       198       7.06       18.52       19.78       51.87         FBC 9       0.20       0.021 18       0.045 85       0.056 51       0.128 39       1632b       207       5.62       14.64       15.74       41.00         1633b       0.27       0.017 74       0.099 49       0.046 6       0.110 56       FBC 9       212       15.27       35.85       42.74       10.83         89one       0.32       0.033 12       0.079 32       0.062 70       0.303 08       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.037	WAL 4	1.30	0.050 99	0.126 60	0.142 77	0.354 48	1633b	0.61	0.0361	0.1035	0.101 04	0.289 88
1632b       1.74       0.067 30       0.180 40       0.143 53       0.505 13       90five       1.07       0.0776       0.1712       0.217 38       0.479 47         B38ben       2.53       0.067 30       0.241 91       0.386 737       0.396 70       1.084 64       0.677 36       WAL 4       1.34       0.0776       0.1712       0.217 38       0.635 33         Material       Average       Sr       Material       Material <t< td=""><td>89one</td><td>1.49</td><td>0.202 06</td><td>0.306 53</td><td>0.565 76</td><td>0.858 27</td><td>WAL 7</td><td>0.70</td><td>0.035</td><td>0.0869</td><td>0.097 92</td><td>0.243 24</td></t<>	89one	1.49	0.202 06	0.306 53	0.565 76	0.858 27	WAL 7	0.70	0.035	0.0869	0.097 92	0.243 24
1838b       2.34       0.067 30       0.241 91       0.18 44       0.677 36       WAL 4       1.34       0.0974       0.2269       0.272 82       0.635 33         Material       Average       Sr       Sr       SR       r       R       Material       Average       Sr       SR       r       R         91three       0.17       0.017 56       0.028 64       0.049 17       0.090 18       89ten       198       7.06       18.52       19.78       51.87         FBC 9       0.20       0.017 76       0.029 46       0.046 6       0.110 56       FBC 9       212       15.27       35.85       42.74       100.39         89ten       0.22       0.017 76       0.029 74       0.052 5       0.069 28       90five       240       18.89       49.5       52.89       138.60         89one       0.32       0.053 64       0.069 27       0.303 08       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.023 89       0.063 64       0.069 20       0.150 20       2776       47.13       34.16       86.79       52.89       138.60         1632c       0.59       0.636 40       0.027 33 <td>2776</td> <td>1.69</td> <td>1.101 39</td> <td>0.234 14</td> <td>0.283 90</td> <td>0.655 59</td> <td>91three</td> <td>0.76</td> <td>0.053</td> <td>0.1032</td> <td>0.148 38</td> <td>0.288 94</td>	2776	1.69	1.101 39	0.234 14	0.283 90	0.655 59	91three	0.76	0.053	0.1032	0.148 38	0.288 94
1633b       2.34       0.067 ab       0.241 9b       0.184 4b       0.677 36       WAL 4       1.34       0.0974       0.2269       0.272 82       0.635 33         Baterial       Average       Sr       Sr       SR       r       R       Material       Average       Sr       SR       r       R         91three       0.17       0.017 56       0.028 46       0.049 17       0.008 18       89ten       198       7.06       18.52       19.78       51.87         FBC 9       0.200 10.0020 18       0.045 65       0.028 46       0.046 17       0.008 18       89ten       198       7.06       18.52       19.78       51.87         B3ben       0.27       0.017 74       0.039 49       0.046 6       0.110 56       FBC 9       212       15.27       35.85       42.74       100.39         B3ben       0.23       0.033 12       0.023 49       0.052 6       0.069 28       90five       240       18.89       49.5       52.89       138.60         B3one       0.32       0.033 12       0.064 14       0.066 30       0.150 20       2776       47.13       34.16       86.71       95.66       242.79         WAL 7       0.64			0.051 26	0.180 40		0.505 13	90five	1.07			0.217 38	0.479 47
89ten         2.53         0.141 68         0.387 37         0.396 70         1.084 64           Material         Average         Sr         SR         r         R         Material         Average         Sr         SR         r         R           91three         0.17         0.017 56         0.028 64         0.049 17         0.000 18         89ten         198         7.06         18.52         19.78         51.87           FBC 9         0.20         0.020 18         0.046 55         1.0128 39         1633b         207         5.62         14.64         15.74         41.00.39           89ten         0.22         0.018 76         0.024 74         0.052 5         0.069 28         90five         240         18.89         49.5         52.89         138.60           89ten         0.22         0.018 76         0.024 74         0.052 0         2776         23.63         38.93         66.16         108.99           WAL 4         0.56         0.091 77         10.082 4         0.257         0.303 08         WAL 4         463         16.85         60.98         47.13         34.16         86.71         95.66         242.79           WAL 7         0.64         0.047 05				0 241 91						0 2269		
Material         Average         Sr         Sr         SR         r         R         Material         Average         Sr         SR         r         R           91three         0.17         0.017         0.017         6         0.020         18         0.066         10.128         39         1633b         207         5.62         14.64         15.74         41.00           1633b         0.27         0.017         74         0.039         49         0.046         0.110.56         FBC 9         212         15.27         35.85         42.74         100.39           89ten         0.29         0.018         76         0.024         74         0.052         0.069         28         90five         240         18.89         49.5         52.89         138.60           89one         0.32         0.053         4         0.052         0.092         2776         471         34.16         86.71         95.66         242.79           WAL 7         0.64         0.022         73         0.064         1.049         890ne         62         49.7         90.52         19.9.5         53.45           90five         1.99         0.118         47												
Material         Average         Sr         SR         r         R         Material         Average         Sr         SR         r         R           91three         0.17         0.017         0.017         0.020         0.020         0.020         0.045         65         0.128         91         1633b         207         5.62         14.64         15.74         41.00           B30ne         0.229         0.017.76         0.039.49         0.046         0.110.56         FBC 9         212         15.27         35.85         42.74         100.39           89one         0.22         0.013.76         0.024         74         0.052.5         0.069.28         901ive         240         18.89         49.5         52.89         138.60           89one         0.023         0.033.12         0.079.32         0.092.7         0.203.08         WAL 4         463         16.85         60.98         47.18         170.74           1632c         0.59         0.023.89         0.053.64         0.066 90         0.150.20         277.6         471         34.16         86.71         95.66         242.79           WAL 7         0.64         0.047.05         0.053.26         0.131 7		2.00	0.111.00	0.007 07	0.00070	1.001.01						
$\begin{array}{c c c c c c c c c c c c c c c c c c c $												
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Material	Average	Sr	SR	r	R	Material	Average	Sr	SR	r	R
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	91three	0.17	0.017.56	0 028 64	0 049 17	0.080.18	89ten	198	7.06	18.52	19 78	51.87
1633b       0.27       0.017 74       0.039 49       0.046 6       0.110 56       FBC 9       212       15.27       35.85       42.74       100.39         89ene       0.29       0.018 76       0.024 74       0.052 5       0.069 28       90five       240       18.89       49.5       52.89       138.60         89one       0.32       0.033 12       0.079 20       0.222 09       1632       275       23.63       38.93       66.16       108.99         WAL 4       0.56       0.091 77       0.108 24       0.257       0.303 08       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.023 89       0.063 44       0.066 90       0.150 20       2776       471       34.16       86.71       95.66       242.79         VAL 7       0.64       0.047 05       0.652 66       0.131 7       0.149 13       89one       622       49.77       90.52       133.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         VAL 1       3.80       0.127 63       0.405 17												
89ten       0.29       0.018 76       0.024 74       0.052 5       0.069 28       90five       240       18.89       49.5       52.89       138.60         89one       0.32       0.033 12       0.079 32       0.092 7       0.222 09       1632c       275       23.63       38.93       66.16       108.99         WAL 4       0.56       0.091 77       0.108 24       0.257       0.303 8       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.023 89       0.053 64       0.066 90       0.150 20       2776       471       34.16       86.71       95.66       242.79         WAL 7       0.64       0.022 73       0.064 14       0.063 6       0.179 60       WAL 6       565       16.48       100.9       46.14       282.51         2776       0.64       0.047 05       0.053 74       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         90five       7.44       0.256 06       0.481       0.717       1.366 9       WAL 7       699       34.75       90.79       97.30       254.22         MAL 6       7.44       0.256 06       0.488       0.												
89one       0.32       0.033 12       0.079 32       0.092 7       0.222 09       1632c       275       23.63       38.93       66.16       108.99         WAL 4       0.56       0.091 77       0.108 24       0.257       0.303 08       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.023 88       0.053 64       0.066 90       0.150 20       2776       471       34.16       86.71       95.66       242.79         WAL 7       0.64       0.047 05       0.053 26       0.131 7       0.149 13       89one       622       49.77       90.52       139.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       153 5       653       31.63       104.83       88.58       293.52         WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         I635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       91three       770       39.04       142.03       109.32       39.67         WAL 6       7.4       0.266 06 <td></td>												
WAL 4       0.56       0.091 77       0.108 24       0.257       0.303 08       WAL 4       463       16.85       60.98       47.18       170.74         1632c       0.59       0.023 89       0.053 64       0.066 90       0.150 20       2776       471       34.16       86.71       95.66       242.79         2776       0.64       0.047 05       0.053 26       0.131 7       0.149 13       890ne       622       49.77       90.52       139.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       99.72       148.50       279.23         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       99.72       148.50       279.23         PIthee       381       20.6												
1632c       0.59       0.023 89       0.053 64       0.066 90       0.150 20       2776       471       34.16       86.71       95.66       242.79         WAL 7       0.64       0.022 73       0.064 14       0.063 6       0.179 60       WAL 6       565       16.48       100.9       46.14       282.51         2776       0.64       0.047 05       0.053 26       0.131 7       0.149 13       890ne       622       49.77       90.52       139.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         1635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       91three       770       39.04       142.03       109.32       397.67         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       50.8       58.3       142.3         890ne       343       20.6       89.6 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>												
WAL 7       0.64       0.022 73       0.064 14       0.063 6       0.179 60       WAL 6       565       16.48       100.9       46.14       282.51         2776       0.64       0.047 05       0.053 26       0.131 7       0.149 13       890ne       622       49.77       90.52       139.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         1635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       91three       770       39.04       142.03       109.32       397.67         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       99.72       148.50       279.23         Waterial       Average       Sr       SR       R       R       Material       Average       Sr       SR       R       R         Boone       246       17.1       87.2       47.8       244.1       89one       285       20.8       50.8       58.3       142.3         Boone       343       20.6       89.6       57.8 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>												
2776       0.64       0.047 05       0.053 26       0.131 7       0.149 13       89one       622       49.77       90.52       139.35       253.45         90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         1635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       9three       770       30.04       142.03       109.32       397.67       279.23         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       99.72       148.50       279.23         Material       Average       Sr       SR       r       R       Material       Average       Sr       SR       r       R         1632       266       17.1       87.2       47.8       244.1       89one       285       20.8       50.8       58.3       142.3         901hree       381       29.7       51.5       <												
90five       1.99       0.118 47       0.152 37       0.331 7       0.426 63       1635       653       31.63       104.83       88.58       293.52         WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         1635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       91three       770       39.04       142.03       109.32       397.67         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       99.72       148.50       279.23         Material       Average       Sr       SR       r       R       Material       Average       Sr       SR       r       R         Byone       343       20.6       89.6       57.8       250.9       SL 16       338       36.2       121.4       101.3       340.0       260.9         91three       381       29.7       51.5       83.2       144.2       FBC 9       734       58.4       76.3       163.6       213.7         1632c       645       19.6       43.2       54.8												
WAL 1       3.80       0.127 63       0.405 17       0.357 4       1.134 7       WAL 7       699       34.75       90.79       97.30       254.22         1635       6.65       0.218 39       0.371 48       0.611 50       1.040 14       91three       770       39.04       142.03       109.32       397.67         WAL 6       7.44       0.256 06       0.488 1       0.717       1.366 69       WAL 1       834       53.04       90.79       97.30       254.22         Material       Average       Sr       SR       r       R       Material       Average       Sr       SR       r       R         FBC 9       266       17.1       87.2       47.8       244.1       890ne       285       20.8       50.8       58.3       142.3         89one       343       20.6       89.6       57.8       250.9       SL 16       338       36.2       121.4       101.3       340.0         91three       381       29.7       51.5       83.2       144.2       FBC 9       734       58.4       76.3       163.6       213.7         1632c       645       19.6       43.2       54.8       121.0       89ten	2776	0.64	0.047 05	0.053 26	0.131 7	0.149 13	89one	622	49.77	90.52	139.35	253.45
1635 WAL 66.65 7.440.218 39 0.256 060.371 48 0.488 10.611 50 	90five	1.99	0.118 47	0.152 37	0.331 7	0.426 63	1635	653	31.63	104.83	88.58	293.52
WAL 6         7.44         0.256 06         0.488 1         0.717         1.366 69         WAL 1         834         53.04         99.72         148.50         279.23           Material         Average         Sr         SR         r         R         Material         Average         Sr         SR         r         R           FBC 9         266         17.1         87.2         47.8         244.1         890ne         285         20.8         50.8         58.3         142.3           890ne         343         20.6         89.6         57.8         250.9         SL 16         338         36.2         121.4         101.3         340.0           91three         381         29.7         51.5         83.2         144.2         FBC 9         734         58.4         76.3         163.6         213.7           1632b         821         38.2         59.9         107.1         167.6         1632c         1047         25.3         56.7         70.8         158.8           1633b         821         38.2         59.9         107.1         167.6         1632c         1047         25.3         56.7         70.8         158.8           2776	WAL 1	3.80	0.127 63	0.405 17	0.357 4	1.134 7	WAL 7	699	34.75	90.79	97.30	254.22
WAL 6         7.44         0.256 06         0.488 1         0.717         1.366 69         WAL 1         834         53.04         99.72         148.50         279.23           Material         Average         Sr         SR         r         R         Material         Average         Sr         SR         r         R           FBC 9         266         17.1         87.2         47.8         244.1         890ne         285         20.8         50.8         58.3         142.3           890ne         343         20.6         89.6         57.8         250.9         SL 16         338         36.2         121.4         101.3         340.0           91three         381         29.7         51.5         83.2         144.2         FBC 9         734         58.4         76.3         163.6         213.7           1632b         821         38.2         59.9         107.1         167.6         1632c         1047         25.3         56.7         70.8         158.8           1633b         821         38.2         59.9         107.1         167.6         1632c         1047         25.3         56.7         70.8         158.8           2776	1635	6.65	0.218 39	0.371 48	0.611 50	1.040 14	91three	770	39.04	142.03	109.32	397.67
MaterialAverageSrSrSRrRMaterialAverageSrSRrRFBC 926617.187.247.8244.189one28520.850.858.3142.389one34320.689.657.8250.9SL 1633836.2121.4101.3340.091three38129.751.583.2144.2FBC 973458.476.3163.6213.71632c64519.643.254.8121.089ten86737.993.2106.0260.91633b82138.259.9107.1167.61632c104725.356.770.8158.889ten95036.962.8103.5175.9WAL 71 22952.688.7147.3248.427761 353141.0141.0394.9394.91633b1 23753.7113.8150.4318.816351 898250.1321.4700.3899.927761 67046.9116.1131.2325.2WAL 73 540134.5198.0376.5554.391three1 93557.21 65.51 60.04 63.590five6 034321.7328.9900.7921.0WAL 42 94857.4207.41 60.6580.6WAL 46 899142.6400.93 99.41122.590five2 95997.11 67.3 <td></td> <td></td> <td></td> <td>0.488 1</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>				0.488 1								
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91three38129.751.583.2144.2FBC 973458.476.3163.6213.71632c64519.643.254.8121.089ten86737.993.2106.0260.91633b82138.259.9107.1167.61632c104725.356.770.8158.889ten95036.962.8103.5175.9WAL 7122952.688.7147.3248.427761353141.0141.0394.9394.91633b123753.7113.8150.4318.816351 898250.1321.4700.3899.927761 67046.9116.1131.2325.2WAL 73 540134.5198.0376.5554.391three1 93557.2165.5160.0463.590five6 034321.7328.9900.7921.0WAL 42 94857.4207.4160.6580.6WAL 46 899142.6400.9399.41122.590five2 95997.1167.3271.9468.5WAL 617 120697.01614.91951.64521.816353 43588.4230.2247.4644.5WAL 130 4792101.03542.75882.79919.6WAL 69 290290.5519.9813.41455.6												
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WAL 6         17 120         697.0         1614.9         1951.6         4521.8         1635         3 435         88.4         230.2         247.4         644.5           WAL 1         30 479         2101.0         3542.7         5882.7         9919.6         WAL 6         9 290         290.5         519.9         813.4         1455.6												
WAL 1         30 479         2101.0         3542.7         5882.7         9919.6         WAL 6         9 290         290.5         519.9         813.4         1455.6												
WAL I IU 400 265 /12 /98.0 1993.0			21VI.V	0042.1	0002.7	3313.0	VVAL 0	3 2 30	230.0	019.9	013.4	1400.0
		000							00F	710	700 0	1002.0

# **↓** D6349 – 09

# TABLE A1.1 Continued

SO3					
Material	Average	Sr	SR	r	R
NIST	0.5062	0.0205	0.0277	0.0574	
1633b					0.0774
APBA	0.1396	0.0288	0.0579	0.0806	0.1621
BD	0.8700	0.0201	0.0692	0.0564	0.1938
PHA	4.6179	0.0586	0.3237	0.1642	0.9064
ESP5	8.7171	0.1419	0.2234	0.3973	0.6256
LP1	3.6971	0.0546	0.0727	0.1528	0.2036
LP2	10.0279	0.0942	0.3698	0.2637	1.0355
SDA	6.0992	0.0992	0.3014	0.2778	0.8439
SFA	4.7088	0.0968	0.0987	0.2709	0.2765

<sup>A</sup>Value-treated as an outlier.

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