



Designation: **D6349 – 13** **D6349 – 21**

# 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 ( $\epsilon$ ) 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.

NOTE 1—There are two interlaboratory studies associated with this test method. The first was conducted in 1997 (RR:D05-1035)<sup>2</sup> and the second was conducted in 2007 (RR:D05-1032).<sup>3</sup> Sulfur trioxide was only included in the 2007 study, and that study only included combustion residues derived from ash and no combustion residues derived from coke.

1.2 Units—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>4</sup>

[D121 Terminology of Coal and Coke](#)

[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](#)

<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>2</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1035. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

<sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1032. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

<sup>4</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke](#)  
[D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal](#)  
[D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases](#)  
[D7348 Test Methods for Loss on Ignition \(LOI\) of Solid Combustion Residues](#)  
[D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis](#)  
[D8146 Guide for Evaluating Test Method Capability and Fitness for Use](#)  
[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)  
 2.2 *ISO Standard*:<sup>5</sup>  
[ISO/IEC Guide 99:2007 International vocabulary of metrology -- Basic and general concepts and associated terms \(VIM\)](#)

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology [D121](#).

### 4. Summary of Test Method

4.1 The sample to be analyzed is ashed under standard conditions and ignited to constant ~~weight~~-mass. 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 photomultiplier 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.

### 5. Significance and Use

5.1 A compositional analysis of coal and coke and their associated combustion residues are often useful in assessing 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~~mass fraction 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.

5.2 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 commercial-scale burning of the coal.

### 6. Interferences

6.1 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.

6.1.1 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 mass 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.

6.1.2 [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](#).

<sup>5</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

6.1.3 **Table 2<sup>6</sup>** 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 mass 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.

6.1.4 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/Humidifying 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.

6.1.5 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, (such as, incident power, gas flows, observation position, and so forth), height, and sample uptake rate~~), 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.

## 7. Apparatus

7.1 *Ashing Furnace*, with an adequate air circulation and capable of having its temperature regulated at ~~500°C~~500 °C and ~~750°C~~750 °C.

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

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

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

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

7.6 *Polycarbonate Bottles*, ~~250-mL~~250 mL capacity with an O-ring seal and screw cap, capable of withstanding temperatures of

<sup>6</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 Mass Concentration Equivalents (mg/L) Arising from Interference at the 100 ppm (mg/L) Level<sup>6</sup>**

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

Analyte Elements	Wavelengths, nm	Interferents					
		Al	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

100°C 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 during the digestion.

7.7 *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 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.

NOTE 2—The abbreviation ICPAES is used throughout this test method to refer to Inductively Coupled Plasma Atomic Emission Spectrometry, and it is understood that some manufacturers will instead use the abbreviation ICPOES. In all cases, it is understood that ICPAES and ICPOES refer to the same technique.

## 8. Reagents

8.1 *Purity of Reagents*—Reagent 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>7</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.

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

8.3 *Standard Stock Solutions*—Stock solutions of 1000 mg ppm (mg/L) for each element are needed for preparation of dilute standards in the range from <math>0.1</math> mg/L to 100 mg ppm/L. Prepare standard stock solutions from 99.99% purity metals or salts. Alternatively, one can use commercially available stock solutions specifically prepared for ICP-AES spectroscopy.

8.3.1 If multi-element stock standards are prepared, care should be taken to take into account contamination present for other elements of interest in each single element stock solution. It is recommended that multi-element stocks be purchased from a reputable standards vendor and include a certification of the major element mass concentrations as well as a table of contaminant elements and mass concentrations.

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

<sup>7</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.

## 8.5 Acids:

8.5.1 *Hydrochloric Acid*—Concentrated HCl, ~~sp gr~~ hydrochloric acid, 12 N, specific gravity 1.19.

8.5.2 *Hydrofluoric Acid*—Concentrated HF, ~~sp gr~~ hydrofluoric acid, 29 N, specific gravity 1.17.

8.5.3 *Nitric Acid*—Concentrated HNO<sub>3</sub>, ~~sp gr~~ nitric acid, 16 N, specific gravity 1.42.

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

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

8.6 *Fluxing Agents*—Lithium tetraborate, Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, or mixtures of lithium tetraborate (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) and anhydrous lithium metaborate (LiBO<sub>3</sub>).

8.7 *Boric Acids Solution*—1.5 %.

8.8 *Hydrogen Peroxide*—~~30%~~ 30 %.

8.9 *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.

8.10 *Standard Solution Diluent*—Use either 8.10.1 or 8.10.2.

8.10.1 ~~Weigh~~ Transfer 4 g, determined to the nearest 0.0001 g, of fluxing agent (see 8.6) into a clean ~~1000-mL~~ 1000 mL beaker containing a magnetic stirring bar. Add 500 mL of 5 + 95 nitric acid (see 8.5.4) 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 13). Quantitatively transfer the warm solution to a ~~1000-mL~~ 1000 mL volumetric flask. After the solution cools to room temperature, dilute to 1000 mL with reagent grade water.

8.10.2 ~~Weigh~~ Transfer 4 g, determined to the nearest 0.0001 g, of fluxing agent (see 8.6) into a platinum dish (or crucible). Heat to ~~1000°C~~ 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~~ 1000 mL beaker. Add 500 mL of 5 + 95 nitric acid (see 8.5.4) 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~~ 30 min (see Note 13). 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~~ 1000 mL volumetric flask. After the solution cools to room temperature, dilute to ~~1000-mL~~ 1000 mL with reagent grade water.

NOTE 3—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.

8.11 *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.

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

8.11.2 *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.

8.12 ~~Initial calibration verification standard(s):~~ Calibration Verification Standard(s):



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

8.12.2 Where possible the initial calibration verification standard(s) shall be traceable to a primary standard such as a NIST CRM: National Institute of Technology Standard Reference Material (NIST SRM).

8.13 ~~Periodic calibration verification standard(s)~~—Calibration Verification Standard(s)—The source of these materials can be the same as the calibration materials.

8.14 *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).

8.15 *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).

## 9. Sample Preparation

9.1 *Coal and Coke*—Prepare the analysis sample in accordance with Practice **D2013** for coal or Practice **D346** for coke by pulverizing the material to pass a 250 µm (No. 60) U.S.A. standard sieve.

9.1.1 Analyze separate test portions for moisture and ash contents in accordance with Test Methods **D3173**, **D3174**, or **D7582** so that calculation to other bases can be made.

9.2 *Laboratory Ashing of Coal and Coke Analysis Sample*—Prepare the ash from a thoroughly mixed analysis sample of coal or coke (see **9.1**). Spread the coal and coke in a layer not over 6 mm 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~~500 °C ± ~~10~~10 °C at the end of 1 h. Continue the gradual heating until the temperature rises from ~~500~~500 °C ± ~~10~~10 °C to ~~750~~750 °C ± ~~15~~15 °C at the end of 1 h. Maintain the ~~750~~750 °C temperature until the test specimen reaches a constant mass or for an additional ~~two hours~~2 h. Allow the dish to cool, transfer to an agate mortar, and grind to pass a 75 µm (No. 200) U.S.A. standard sieve. Reignite the ash at ~~750~~750 °C for 1 h, ~~cool rapidly, and weigh~~ allow to cool to room temperature, and determine the mass of portions for analysis.

9.3 *Solid Combustion Residue*—Dry a representative portion of the solid residue to constant mass at ~~107~~107 °C ± ~~3~~3 °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 75 µm (No. 200) U.S.A standard sieve. Use a mill that minimizes metal contamination.

9.4 *Ashing Solid Combustion Residue*—Spread an appropriate amount of the prepared sample 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~~500 °C ± ~~10~~10 °C at the end of 1 h. Continue the gradual heating until the temperature rises from ~~500~~500 °C ± ~~10~~10 °C to ~~750~~750 °C ± ~~15~~15 °C at the end of 1 h. Maintain the ~~750~~750 °C temperature until the combustion residue reaches a constant mass or for an additional ~~two hours~~2 h. Cool the test specimen, grind to pass a 75 µm (No. 200) U.S.A standard sieve, and reignite at ~~750~~750 °C for 1 h.

9.5 If previously-ignited samples are stored and the absorption of moisture, or CO<sub>2</sub>, or both, is in question, reignite the ash at ~~750~~750 °C before use. Alternatively, determine loss on ignition using Test Method **D7348** on a separate sample ~~weighed out whose mass is determined~~ 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 mass 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 mass 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  $\text{HNO}_3$  (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, tabletops, 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~~: fusion and mixed acid. 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 mass 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 0.1 g (to Transfer 0.1 g (determined to the nearest 0.1 mg))~~ 0.1 mg of the ash sample as prepared in 9.5 or 9.4 into a platinum dish (or crucible) (see Note 24). ~~Weigh 0.4 g (to Transfer 0.4 g (determined 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~~ 1000 °C to ~~1200~~ 1200 °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~~ 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~~ 250 mL or ~~400~~ 400 mL beaker. Place a clean TFE-fluorocarbon coated magnetic stirring bar in the platinum dish and add ~~50~~ 50 mL of ~~5~~ 5 + 95  $\text{HNO}_3$  nitric acid (see 8.5.4) 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~~ 30 min. Constantly stir and heat the solution, and repeat the analysis if precipitate is formed (see Note 35). Remove the platinum dish from the beaker, rinse the dish with small amounts of reagent water, and quantitatively transfer the solution to a ~~100~~ 100 mL volumetric flask. Add 1 mL of internal standard to the flask and dilute to the ~~100~~ 100 mL mark with water. This solution is ~~1000~~ 1000 mg ppm/L with respect to the total sample and contains ~~44 g~~ 4 g/L of fluxing agent. Prepare a method blank using the above procedure.

NOTE 4—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 5—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~~ 50 mL of ~~5~~ 5 + 95 nitric acid (see 8.5.4) in a clean ~~250~~ 250 mL or ~~400~~ 400 mL beaker containing a Teflon-coated TFE-fluorocarbon 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).~~ If it cannot be completely transferred, the sample should be discarded and re-prepared. 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~~ 30 min. Constantly stir and heat the solution, and repeat the analysis if precipitate is formed (see Note 35). Transfer the solution quantitatively to a ~~100~~ 100 mL volumetric flask. Add ~~1~~ 1 mL of internal standard to the flask and dilute to the ~~100~~ 100 mL mark with water. This solution is ~~1000~~ 1000 ppm/L with respect to the total ash sample and contains ~~4~~ 4 g/L of fluxing agent. Prepare a method blank using the above procedure.

10.3.2 *Mixed Acid Dissolution*—~~Weigh 0.1 g (to Transfer 0.1 g (determined to the nearest 0.1 mg))~~ of the ash sample as prepared in 9.5 or 9.4 into a ~~250~~ 250 mL polycarbonate bottle with an O-ring seal and screw cap ~~9~~ see cap. Note 4. The bottle should be capable of withstanding a temperature up to ~~130~~ 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.)