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Standard Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Furnace Atomic Absorption Spectrometry[1]

This standard is issued under the fixed designation D6357; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods pertain to the determination of antimony, arsenic, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, vanadium, and zinc in coal and coke. These test methods can also be used for the analysis of residues from coal combustion processes.

NOTE 1—These test methods may be applicable to the determination of other trace elements.

1.2 The values stated in SI units ~~shall~~ are to be regarded as ~~the~~ standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.2.1 All percentages are percent mass fractions unless otherwise noted.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke
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
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
D7448 Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke
D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Documents:*[3]

EPA/600/4-91/010 Methods for the Determination of Metals in Environmental Samples

[1] These test methods are under the jurisdiction of ASTM Committee D05 on Coal and Coke and are the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Superintendent of Documents, U.S. Printing Office, Washington, DC 20402.

3. Terminology

3.1 *Definitions*—Definitions applicable to these test methods are listed in Terminology D121.

4. Summary of Test Method

4.1 The coal or coke to be analyzed is ashed under controlled conditions, digested by a mixture of aqua-regia and hydrofluoric acid, and finally dissolved in 1 % nitric acid. Combustion residues are digested on an as-received basis. The mass concentration of individual trace elements is determined by either inductively coupled plasma atomic emission spectrometry (ICPAES) or inductively coupled plasma mass spectrometry (ICPMS). Selected elements that occur at mass concentrations below the detection limits of ICPAES can be quantitatively analyzed by graphite furnace atomic absorption spectrometry ~~(GFAA).~~(GFAAS) or ICPMS.

5. Significance and Use

5.1 Coal contains several elements whose individual ~~concentrations~~ mass fractions are generally less than 0.01 %. These elements are commonly and collectively referred to as trace elements. These elements primarily occur as part of the mineral matter in coal. The potential release of certain trace elements from coal combustion sources has become an environmental concern.

5.2 The ash prepared in accordance with these provisional test methods quantitatively retains the elements listed in 1.1 and is representative of their ~~concentrations~~ mass fractions in the coal or coke.

6. Apparatus

6.1 *Inductively Coupled Plasma Atomic Emission Spectrometer (ICPAES)*—The spectrometer system may be either simultaneous or sequential, vacuum or purged, but must include computer-controlled background correction.

6.1.1 *Argon Gas ~~Supply—~~Supply,* ~~High~~high purity ~~(99.99 %).~~(≥ 99.996 %).

6.1.2 *Mass Flow Controllers*—A mass-flow controller to regulate the nebulizer gas is required. Mass flow controllers on the intermediate and outer torch gas flows are recommended.

6.2 *Inductively Coupled Plasma Mass Spectrometer (ICPMS)*—The spectrometer system must be capable of scanning the mass range of the elements to be analyzed.

6.2.1 *Argon Gas Supply,* high purity ~~(99.99 %).~~(≥ 99.996 %).

6.2.2 The use of a variable speed peristaltic pump for delivering sample solution to the nebulizer, a mass-flow controller on the gas supply to the nebulizer, and a water-cooled spray chamber are highly recommended.

6.3 *Atomic Absorption Spectrometer with Graphite Furnace ~~(GFAA),~~(GFAAS),* having background correction capable of removing nonspecific absorbance.

6.3.1 *Single-Element Hollow Cathode or Single-Element Electrodeless Discharge Lamps.*

6.3.2 *Single-Output Device,* capable of recording and evaluating peak area and peak shape.

6.3.3 *Pyrolytic Coated Graphite Tubes and Platforms.*

6.3.4 *Argon Gas Supply,* high purity (99.99 %).

6.3.5 *Autosampler*—Although not specifically required, the use of an autosampler is highly recommended.

6.4 *Muffle Furnace,* with temperature control and with air circulation as specified in 9.1.

6.5 *Analytical Balance,* capable of weighing to 0.1 mg.

6.6 *Teflon Beakers,* ~~100-~~100 mL or ~~200-mL~~200 mL capacity.

6.7 *Hot Plate,* capable of regulating temperature between ~~90~~90 °C to ~~150°C.~~150 °C.

6.8 *Volumetric Flasks,* ~~100-~~10 mL and ~~10-mL~~100 mL capacity.

6.9 *HDPE Bottles,* ~~100-mL~~100 mL capacity.

6.10 *Crucibles,* ~~50-mL~~50 mL quartz or high silica.

7. Reagents

7.1 *Purity of Reagents*—All acids used in these test methods must be trace metal purity grade or equivalent. Redistilled acids are acceptable.

7.2 *Purity of Water*—The purity of the water used in these test methods shall be equivalent to ASTM Type II reagent water of Specification D1193.

7.3 *Aqua Regia Solution*—Mix one part concentrated nitric acid (HNO_3, sp. gr. 1.42) and three parts concentrated hydrochloric acid (HCl, sp. gr. 1.9).

7.4 *Hydrofluoric ~~Acid—~~Acid,* ~~Concentrated~~concentrated (HF, sp. gr. 1.15).

7.5 *ICP Calibration Standards*—Aqueous multielement solutions made up in 1 % HNO_3 are used for calibration of ICPAES and ICPMS systems. The stock standards may be purchased or prepared from high-purity grade chemicals or metals.

7.5.1 *GFAAGFAAS Stock Standard Solution (1000 (1000 µg ppm) /mL)*—Single-element standards either purchased or prepared from high-purity grade chemicals or metals.

7.5.2 *GFAAGFAAS Intermediate Stock Standard Solution (1(1 µg ppm) /mL)*—Add 0.1 mL of stock standard solution (7.5.1) and 1 mL of concentrated nitric acid to a 100-mL100 mL volumetric flask. Dilute to volume with water.

NOTE 2—Accuracy of the pipette was not stated in the instructions for the interlaboratory study for the determination of this method's precision, and so it is not stated here; however, the volumetric measurement accuracy should be considered relative to the repeatability of the method.

7.6 *Magnesium Nitrate Solution*—Matrix modifier (106 g/L(106 g/L $Mg(NO_3)_2 \cdot 6H_2O$) for the determination of arsenic and antimony, equivalent to 10 000 ppm10 000 µg/mL magnesium.

7.6.1 A matrix modifier is used to minimize GFAAGFAAS interference effects by selective volatilization of either the analyte or the matrix components. Other matrix modifiers such as nickel nitrate or palladium nitrate can be used. The analyst should compare modifiers to establish optimum performance as outlined in 10.1.

7.7 *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.

7.7.1 *Calibration Blank*—A 1 % volume fraction nitric acid solution. When using matrix modifiers of GFAA,GFAAS, the calibration blank shall also contain the same equivalent concentration.mass concentration of the matrix modifier.

7.7.2 *Method Blank*—Consists of all the reagents in the same volumes as used in preparing the samples. The method blank shall be processed through the entire sample digestion scheme.

8. Analysis Sample

8.1 Samples of coal and coke shall be prepared in accordance with Practice D2013 or Practice D346.

8.2 Standard practices for the sampling and preparation of residues from coal utilization processes have not been established. Some of these materials are highly abrasive. The use of high speed pulverizers for size reduction shall be avoided. The use of jaw crushers followed by final preparation in an agate mortar and pestle is recommended to prevent contamination of the sample.

8.3 Analyze separate test portions for moisture content in accordance with Test Methods D3173 andor D7582 so that calculations to other bases can be made.

9. Procedure

9.1 *Ashing*—Weigh to the nearest 0.1 mg 0.1 mg enough of the coal or coke sample that will yield approximately 0.5 g 0.5 g of ash into an open 50-mL50 mL quartz or high-silica crucible. Place the crucible in a cold muffle furnace. Adjust the temperature control so that the furnace reaches a temperature of 300°C300 °C in 1 h and then 500°C500 °C in the second hour. Maintain the furnace temperature at 500°C500 °C for a minimum of 2 h, stirring the sample occasionally. Ashing is complete when no visible evidence of carbonaceous material remains. Cool the samples to room temperature under conditions that minimize the absorption of water. Grind the ash to pass a 150-µm150 µm (No. 100) U.S.A. standard sieve in an agate mortar then reignite at 500°C500 °C for 1 h. Cool the ash and store in a desiccator. Determine the percentage of ash by analyzing under the same conditions a separate portion of the analysis sample.Allow the ash to cool under conditions that minimize the absorption of water.

NOTE 3—If all the ash from 9.1 is quantitatively transferred for digestion in 9.2, it is not necessary to sieve and grind the ash. Results from 11.2.3, 12.3, or 13.1.4.8 are then ppm mass fraction of the element in the as-determined sample.

9.1.1 If necessary for results calculations, determine the percentage of ash in the analysis sample according to Test Methods D3174 or D7582, modifying the ashing temperatures in those methods to those specified in 9.1 above.

9.2 *Dissolution*—Weigh 0.200000.2000 g to 0.5000 g 0.5000 g of the thoroughly blended ash prepared according to 9.1 into a 100-100 mL or 200-mL200 mL Teflon beaker. Add 20 mL 20 mL of aqua regia and 20 mL 20 mL of concentrated hydrofluoric acid to the beaker. Place the beaker on a hot plate that has been adjusted to 130130 °C to 150°C.150 °C. Heat the mixture to dryness, but do not bake. After the solution has evaporated, rinse the beaker walls with deionized water and heat this solution to dryness, again being careful not to bake the sample. Remove the beaker from the hot plate and allow it to cool to room temperature. Add 1 mL of concentrated nitric acid and 20 mL 20 mL of deionized water to the beaker. Heat the contents on a hot plate at 9090 °C to 100°C100 °C until the sample is in solution. If a residue remains after 1 h of heating, it may be ignored. The trace elements are considered to be quantitatively extracted at this point. Remove the beaker from the hot plate and allow the solution to cool to room temperature. Transfer Quantitatively transfer the cool solution to a 100-mL100 mL volumetric flask and dilute to volume with deionized water. If the solution is not to be analyzed immediately, transfer it to a HDPE bottle to avoid adsorption of lead during storage. Prepare a method blank (7.7.2) with each batch of samples to be analyzed. To minimize contamination, clean laboratory ware in a volume fraction of 50 % HNO_3 solution followed by a volume fraction of 50 % HCl solution, then rinse thoroughly with water.

NOTE 3—To minimize contamination, clean laboratory ware in a 1:1 solution of HNO_3 followed by a 1:1 solution of HCl then rinse thoroughly with deionized water.

10. Analysis

10.1 ~~Because of the differences~~ Difference between various makes and models of ~~instruments, all~~ instruments will occur, so instrumental operating instructions cannot be provided. Instead, the analyst shall refer to the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, linear dynamic range, interference effects, and appropriate background correction shall be investigated and established for each individual analyte on that particular instrument.

11. Test Method A—Inductively Coupled Plasma Atomic Emission Spectroscopy

11.1 Table 1 shows the elements listed in 1.1 along with some suggested wavelengths for inductively coupled plasma atomic emission spectrometry (ICPAES). Other wavelengths may be substituted if they can provide the needed sensitivity and are treated according to the provisions of 10.1. Also shown are estimated detection limits.

11.2 *Calibration Procedure*—Calibrate the instrument according to the procedure recommended by the manufacturer using a calibration blank and aqueous multielement standards made up in a volume fraction of 1 % trace metal grade HNO_3. All calibration solutions must also contain an internal standard (see Note 4). Records for all calibrations must be in accordance with ~~Guide~~Practice D7448. Internal standards need to be added to all analytical solutions, samples, calibration standards, and quality control samples. Addition of an internal standard can be done either online or manually.

NOTE 4—An internal standard is needed to compensate for:
1. Differences in physical properties (such as viscosity) between the calibration standard and the test ~~samples and~~samples,
2. Drift caused by thermal changes in the laboratory which will affect the instrument ~~optics.~~optics, and
3. Drift caused by changes in the sample introduction system, including tubing wear and nebulizer performance.

An appropriate internal standard element should:
(i) not be naturally present in the test samples in appreciable ~~concentrations;~~mass fractions,
(ii) not present spectral interferences with any analyte,
(iii) be a strong emitter so that its relative mass concentration can be kept low, and
(iv) be as chemically similar to the analyte as possible.

11.2.1 *Initial Calibration Verification*—Before analyzing test samples, analyze the method blank and verify the proper calibration of the instrument by analyzing a reference material that has traceability to an internationally recognized certifying agency such as NIST. Results for the reference material must be within the stated uncertainty limits or the calibration procedure must be repeated.

11.2.2 *Periodic Calibration Verification and Recalibration*—In accordance with ~~Guide~~Practice D7448, analyze a control sample ~~such as NIST~~ on a periodic basis. Results obtained for the control sample must be within ~~10 % of the stated value~~ established limits or all results obtained since the last successful control check of that element must be rejected and the calibration procedure repeated.

11.2.3 *Calculation*—Calculate the ~~concentration~~ mass fraction of the element (dry basis) in the ash as follows:

$$C = (A \times df)/(W \times 100) \quad (1)$$

TABLE 1 Suggested Wavelengths for ICPAES

Element	Wavelength, nm	Estimated Detection Limit, µg/L[A]
As[B]	189.042, 228.812, 193.759	53
Be	313.042	0.3
Cd[B]	226.502	4
Co	228.616	7
Cr	267.716, 205.552	7
Cu	324.754	6
Mn	257.610	30
Mo	202.030, 203.844	8
Ni	231.604	15
Pb	220.353	42
Sb[B]	217.581, 206.833	32
V	292.402, 292.464	8
Zn	213.856	2

[A] ~~Detection~~ Detection limits are given for informational purposes only and represent the lowest mass concentration that produces ~~a~~an instrumental response statistically different from an aqueous blank solution. Detection limits should not be confused with quantitation limits. Detection limits are sample and matrix dependent. They will vary from instrument to instrument and should be established by each user of these test methods. These values (3 sigma) are based on data contained in EPA/600/4-91/010, Method 200.7 Revision 5.4 (1994).

[B] ~~As,~~ As, Cd, and Sb are typically present in coal at ~~concentrations~~ mass fractions that are below the detection limits of ICPAES.

$$w_a = \left(\frac{cVD}{m}\right) \quad (1)$$

where:

~~*C* = weight percent of the element in the ash,~~
~~*df* = dilution factor,~~
~~*A* = ppm of the element in solution, and~~
~~*W* = weight of the sample in grams.~~

w_a = mass fraction of the element in the ash (dry basis), µg/g,
c = mass concentration of the element in solution, µg/mL,
V = final volume of the 100 mL flask containing the dissolved solution of the ash, mL,
D = analytical dilution factor (if used), dimensionless, and
m = mass of the ash sample, g.

12. Test Method B—Inductively Coupled Plasma Mass Spectrometry

12.1 Table 2 shows the elements listed in 1.1, the isotope, and its abundance used for ICPMS determinations. Also shown are some potential molecular ~~interferents.~~interferences.

12.2 *Calibration*—In conjunction with 11.2, calibrate the instrument by analyzing a blank consisting of ~~deionized~~ water and appropriate internal ~~standards, and a~~ standards in a volume fraction of 1 % solution of HNO_3 containing 0 ~~ppb~~ng/mL of the elements to be analyzed and internal standards. Continue the calibration by analyzing three solutions that cover the expected mass concentration range of the elements to be analyzed. ~~One of the solutions must be lower, one in the range, and one higher in concentration than that of the analyte. Suggested~~ The mass concentrations of the calibration standards should bracket the expected mass concentrations of the analytes. Suggested mass concentration ranges are ~~10,~~10 ng ~~50,~~/mL, 50 ng/mL, and ~~250~~250 ng ~~ppb.~~/mL.

12.2.1 *Internal Standards*—Internal standards are needed ~~to compensate for instrument drift.~~ for the reasons stated in 11.2. Drift associated with ICPMS instruments ~~is typically~~ can in part be mass dependent. Therefore, it is recommended that the analyst use a series of internal standards that covers the mass range and ionization potentials of the elements to be analyzed. Elements used as internal standards should not be present in the samples to be analyzed in appreciable quantities. ~~Li, Ge, In, and Bi are recommended as~~ Refer to the manufacturer for recommendations of internal standards for the list of elements in 1.1. ~~However,~~ If Li is used as an internal standard, an enriched (95 % or better) ^{6}Li must be used because of the significant ~~concentration of~~ mass fraction of naturally occurring ^{7}Li in most coals. Because they are not present in coal in appreciable ~~concentrations, any~~mass fractions, isotopes of Ge, In, and Bi may be ~~used~~used.

12.2.2 *Initial Calibration Verification*—Before analyzing test samples, analyze the method blank and verify the proper calibration of the instrument by analyzing a reference material that has traceability to an internationally recognized certifying agency such as NIST. Results for the reference material must be within the stated uncertainty limits or the calibration procedure must be repeated.

TABLE 2 Isotopes Used for ICPMS Trace Element Determinations

Element	Isotope	Abundance	Interferant	Estimated Detection Limit, µg/L[A]
As	75	100	^{40}Ar ^{35}Cl+	0.9
Be	9	100		0.1
Cd	114	28.8	^{96}Mo ^{16}O+	0.1
Co	59	100		0.03
Cr	52	83.8		0.07
Cu	63	69.1	^{47}Ti ^{16}O+	0.03
Mn	55	100		0.1
Mo	98	23.8		0.1
Ni	60	26.1		0.2
Pb	206	52.4		0.08
Sb	121	57.3		0.08
V	51	99.8	^{35}Cl ^{16}O+	0.02
Zn	68	18.6	^{36}S ^{16}O ^{16}O+	0.2

[A] ~~Detection~~ Detection limits are given for informational purposes only and represent the lowest mass concentration that produces an instrument response statistically different from an aqueous blank solution. Detection limits should not be confused with quantitation limits. Detection limits are sample and matrix dependent. They will vary from instrument to instrument and should be established by each user of these test methods. The values (3 sigma) are based on data contained in EPA/600/4-91/010, Method 200.8, Revision 5.4 (1994).

12.2.3 *Periodic Calibration Verification and Recalibration*—In accordance with ~~Guide~~Practice D7448, analyze a control sample on a periodic basis. Results obtained for the control sample must be within ~~10 % of the stated value~~ established limits or all results obtained since the last successful control check for that element must be rejected and the calibration procedure repeated.

12.3 *Calculation*—Calculate the ~~concentration~~ mass fraction of the element in the ash ~~as follows:~~according to

$$C = (A \times df)/(W \times 100) \quad (2)$$

Eq 1 in 11.2.3.

~~where:~~

~~C = weight percent of the element in the ash,~~
~~df = dilution factor,~~
~~A = ppm of the element in solution, and~~
~~W = weight of the sample in grams.~~

13. Test Method C—Graphite Furnace Atomic Absorption Spectrometer

13.1 *Calibration and Sample Solution Preparation:*

13.1.1 Use the intermediate stock standard solution (7.5.2) to prepare at least five working standards to cover the optimum mass concentration ranges specified by the instrument manufacturer for the element to be analyzed. Add an aliquot of concentrated nitric acid to obtain a final ~~concentration~~ volume fraction of 1 % HNO_3. When preparing arsenic or antimony working standards, add 2 mL of magnesium nitrate solution (7.6~~).~~) per 10 mL of working standard solution.

13.1.2 *Sample Aliquot*—Add an aliquot of the sample solution (9.2) in the optimum mass concentration range for the element to be determined to a ~~10-mL~~10 mL volumetric flask. To estimate the aliquot of sample solution, it may be necessary to analyze the original sample solution (9.2). In some cases, only by trial and error can the correct aliquot of sample be determined. Alternatively, ICPAES can be used to screen samples to determine which elements may require analysis by ~~GFAA.~~GFAAS.

13.1.3 Add nitric acid ~~to obtain a 1 % solution.~~ so that the 10 mL of solution will have a final volume fraction of 1 % nitric acid. The determination of arsenic and antimony require the addition of 2 mL of magnesium nitrate solution (7.6~~).~~) per 10 mL of working standard solution. Dilute to volume with water.

13.1.4 *Instrument Parameters*—As stated in 10.1, because of differences in equipment, it is impossible to specify instrument operating parameters (for example, wavelength, slit, lamp power, drying, ashing and atomization temperatures, and so forth). Instead, the analyst shall initially program the system according to the instrument manufacturer's instructions for a particular analyte. Optimize instrument performance for each analyte according to the following sections.

13.1.4.1 *Drying Temperature*—Make an injection of both a sample and a working standard solution according to 13.1.4. Use a mirror to observe the samples through the introduction port. The drying temperature should be high enough to evaporate the sample smoothly but not so hot that the sample begins to boil or spatter.

13.1.4.2 *Ashing Temperature*—As the ashing step begins, no sizzle or popping sounds should be heard. The ashing temperature should be high enough to eliminate most of the background but not so hot as to volatilize the analyte. A high flow rate of inert gas is required during the ashing stage to sweep the furnace of unwanted background material.

13.1.4.3 *Atomization Temperature*—Adjust the atomization temperature as necessary to eliminate low, broad, misshapen, or doublet peaks. Adjustments should be made in ~~100°C~~100 °C increments. Peak shape may also dictate the mode of measurement (peak height or peak area) and the choice of graphite tube and platforms. Graphite platforms significantly improve instrument performance for the determination of Cd, Pb, As, and Sb. It is strongly recommended that they be tried as part of optimizing instrument performance for each element to be determined.

13.1.4.4 Refer to the instrument manufacturer's instructions for further information on optimizing performance.

13.1.4.5 Repeat the steps in 13.1 through ~~13.3.4~~13.1.4 for each element to be determined.

13.1.4.6 *Initial Calibration Verification*—Before analyzing test samples, analyze the method blank and verify the proper calibration of the instrument by analyzing a reference material that has traceability to an internationally recognized certifying agency such as NIST. Results for the reference material must be within the stated uncertainty limits or the calibration procedure must be repeated.

NOTE 5—**Caution:** Matrix problems are prevalent when analyzing the types of samples described in 1.1 by ~~GFAA.~~GFAAS. If the sample matrix varies significantly from that of the reference material, validation of the test methods with the reference material may lead to an incorrect assumption that the test methods are applicable to other matrices.

13.1.4.7 *Periodic Calibration, Verification, and Recalibration*—In accordance with ~~Guide~~Practice D7448, analyze a control sample ~~such as NIST~~ on a periodic basis. Results obtained for the control sample must be within ~~10 % of the stated value~~ established limits or all results obtained since the last successful control check for that element must be rejected and the calibration procedure repeated.

13.1.4.8 *Calculation*—Calculate the ~~concentration~~ mass fraction of the element according to Eq 1 in 11.2.3~~the ash as follows:~~.

$$C = (A \times df)/(W \times 100) \quad (3)$$