



Standard Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy¹

This standard is issued under the fixed designation C 1109; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is applicable to the determination of low concentration and trace elements in aqueous leachate solutions produced by the leaching of nuclear waste materials.

1.2 The nuclear waste material may be a simulated (non-radioactive) solid waste form or an actual solid radioactive waste material.

1.3 The leachate may be deionized water or any natural or simulated leachate solution containing less than 1 % total dissolved solids.

1.4 The analysis must be conducted with an inductively coupled plasma-atomic emission spectrometer.

1.5 The values stated in SI units are to be regarded as the standard.

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

2. Referenced Documents

2.1 ASTM Standards:²

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Wastes

D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

¹ This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved June 1, 2004. Published July 2004. Originally approved in 1988. Last previous edition approved in 1998 as C 1109 – 98.

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. Terminology

3.1 Definitions:

3.1.1 *Inductively Coupled Plasma Emission Spectroscopy (ICP-AES)*—refer to Terminology E 135.

3.1.2 *water*—refer to Terminology D 1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *analytical curve*—the plot of net signal intensity versus elemental concentration using data obtained during calibration.

3.2.2 *calibration*—the process by which the relationship between net signal intensity and elemental concentration is determined for a specific element analysis.

3.2.3 *calibration blank*—a 1 % (v/v) solution of nitric acid in deionized water.

3.2.4 *calibration reference solution(s)*—solutions containing known concentrations of one or more elements in 1 % (v/v) nitric acid for instrument calibration.

3.2.5 *detection limits (DL)*—the concentration of the analyte element equivalent to three times the standard deviation of ten replicate measurements of the matrix blank.

3.2.6 *instrument check solution(s)*—solution(s) containing all the elements to be determined at concentration levels approximating the concentrations in the specimens. These solutions must also contain 1 % (v/v) nitric acid.

3.2.7 *linear dynamic range*—the elemental concentration range over which the analytical curve remains linear to within the precision of the analytical method.

3.2.8 *linearity check solution(s)*—solution(s) containing the elements to be determined at concentrations that cover a range that is two to ten times higher and lower than the concentration of these elements in the calibration reference solutions. These solutions also contain 1 % (v/v) nitric acid.

3.2.9 *non-spectral interference*—changes in the apparent net signal intensity from the analyte due to physical or chemical processes that affect the transport of the analyte to the plasma and its vaporization, atomization, or excitation in the plasma.

3.2.10 *off-peak background correction*—during specimen analysis, measurements are made of the background intensity near the peak wavelength of the analytical lines. Correction of

the analytical line peak intensity to yield the net line intensity can be made by subtraction of either (a) a single intensity measurement performed on the high or low wavelength side of the analytical line (single-point background correction), or (b) an interpolated background intensity from background measurements acquired on both the high and low wavelength sides of the analytical line (double-point background correction).

3.2.11 *on-peak spectral interference correction*—adjustments made in observed net intensity of peak of interest to compensate for error introduced by spectral interferences.

3.2.12 *sensitivity*—the slope of the linear dynamic range.

3.2.13 *spectral interference*—light emission from spectral sources other than the analyte element that contributes to the apparent net signal intensity of the analyte. Sources of spectral interference include spectral line overlaps, broadened wings of intense spectral lines, ion-atom recombination continuum emission, molecular band emission, and stray (scattered) light effects.

4. Summary of Practices

4.1 The general principles of emission spectrometric analysis are given in Ref (1).³ In this practice, elemental constituents of aqueous leachate solutions are determined simultaneously or sequentially by inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

4.2 Specimens are prepared by filtration if needed to remove particulates and acidification to match calibration reference solutions. Filtration should be the last resort to clarify a solution since leach studies are designed to determine the absolute amount of glass dissolved.

4.3 Additional general guidelines are provided in Guide C 1009, Terminology D 1129, Specification D 1193, and Terminology E 135.

5. Significance and Use

5.1 This practice may be used to determine concentrations of elements leached from nuclear waste materials (glasses, ceramics, cements) using an aqueous leachant. If the nuclear waste material is radioactive, a suitably contained and shielded ICP-AES spectrometer system with a filtered exit-gas system must be used, but no other changes in the practice are required. The leachant may be deionized water or any aqueous solution containing less than 1 % total solids.

5.2 This practice as written is for the analysis of solutions containing 1 % (v/v) nitric acid. It can be modified to specify the use of the same or another mineral acid at the same or higher concentration. In such cases, the only change needed in this practice is to substitute the preferred acid and concentration value whenever 1 % nitric acid appears here. It is important that the acid type and content of the reference and check solutions closely match the leachate solutions to be analyzed.

5.3 This practice can be used to analyze leachates from static leach testing of waste forms using C 1220.

6. Apparatus

6.1 *Inductively Coupled Plasma-Atomic Emission Spectrometer*, with a spectral bandpass of 0.05 nm or less, is required to provide the necessary spectral resolution. The spectrometer may be of the simultaneous multielement or sequential scanning type. The spectrometer may be of the air-path, inert gas-path, or vacuum type, with spectral lines selected appropriately for use with the specific instrument. Either an analog or digital readout system may be used.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ 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 reagent water as defined by Type I of Specification D 1193 or water exceeding these specifications.

7.3 *Nitric Acid* (specific gravity 1.42)—Concentrated nitric acid (HNO₃).

7.4 *Nitric Acid, High-Purity*—Nitric acid of higher purity than reagent grade, specially prepared to be low in metallic contaminants. The acid may be prepared by sub-boiling distillation (2), or purchased from commercial sources.

7.5 *Stock Solutions*— May be purchased or prepared from metals or metal salts of known purity. Stock solutions should contain known concentrations of the element of interest ranging from 100 to 10 000 mg/L.

7.6 *Calibration Reference Solutions, Instrument Check Solutions, and Linearity Check Solutions:*

7.6.1 Prepare single-element or multielement calibration reference solutions by combining appropriate volumes of the stock solutions in acid-rinsed volumetric flasks. To establish the calibration slope accurately, provide at least one solution with element concentration that is a minimum of 100 times the detection limit for each element. Add sufficient nitric acid to bring the final solution to 1 % HNO₃. Prior to preparing the multielement solutions, analyze each stock solution separately to check for strong spectral interference and the presence of impurities (3). Take care when preparing the multielement solutions to verify that the components are compatible and stable (they do not interact to cause precipitation) and that none of the elements present exhibit mutual spectral interference. Transfer the calibration reference solutions to acid-leached FEP TFE-fluorocarbon or polyethylene bottles for storage. Calibration reference solutions must be verified initially using a quality control sample and monitored periodically for stability.

³ The **boldface** numbers in parentheses refer to the list of references at the end of this standard.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."