Designation: C1109 – 23

Standard Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy[1]

This standard is issued under the fixed designation C1109; 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, using inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

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 This practice should be used by analysts experienced in the use of ICP-AES, the interpretation of spectral and non-spectral interferences, and procedures for their correction.

1.5 No detailed operating instructions are provided because of differences among various makes and models of suitable ICP-AES instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

1.6 This practice contains notes that are explanatory and are not part of the mandatory requirements of the method.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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.*

1.9 *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]

C859 Terminology Relating to Nuclear Materials

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste

D1193 Specification for Reagent Water

D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E288 Specification for Laboratory Glass Volumetric Flasks

E438 Specification for Glasses in Laboratory Apparatus

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus and Operator Qualification

3. Terminology

3.1 For definitions of pertinent terms not listed here, see Terminology C859.

3.2 *Definitions:*

3.2.1 *atomic emission*—characteristic radiation emitted by an electronically excited atomic species. **D7035**

[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.

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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.

3.2.1.1 *Discussion*—In atomic (or optical) emission spectrometry, a very high-temperature environment, such as a plasma, is used to create excited state atoms. For analytical purposes, characteristic emission signals from elements in their excited states are then measured at specific wavelengths.

3.2.2 *background correction*—process of correcting the intensity at an analytical wavelength for the intensity due to the underlying spectral background of a blank. **D7035**

3.2.2.1 *Discussion*—During sample 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).

3.2.3 *bias*—difference between the expectation of the test results and an accepted reference value. **E177**

3.2.4 *calibration blank solution*—calibration solution prepared without the addition of any reference solutions. **D7035**

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

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

3.2.7 *critical limit (L_C)*—minimum significant value of an estimated net signal or concentration, applied as a discriminator against background noise. **(1)**

3.2.8 *inductively coupled plasma (ICP)*—a high-temperature discharge generated by a flowing conductive gas, normally argon, through a magnetic field induced by a load coil that surrounds the tubes carrying the gas. **D7035**

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

3.2.10 *interelement correction*—a spectral interference correction technique in which emission contributions from interfering elements that emit radiation at the analyte wavelength are subtracted from the apparent analyte emission after measuring the interfering element concentrations at other wavelengths. **D7035**

3.2.11 *limit of detection (L_D)*—value for which the false negative error is *B* using a given critical limit. **(1)**

3.2.11.1 *Discussion*—If the analytical standard deviation is constant with respect to concentration, this can be computed as 3.7 times the standard deviation of the analytical results from ten matrix blank samples spiked at approximately the anticipated detection limit; otherwise, see references (**1**, **2**)[3] for additional guidance.

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

3.2.13 *linearity check solution(s)*—solution(s) of nitric acid with a volume fraction concentration of 1 % 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.

3.2.14 *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.

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

3.2.16 *spectral interference*—an interference caused by the emission from a species other than the analyte of interest. **D7035**

3.2.16.1 *Discussion*—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 Practice

4.1 Aqueous leachates are prepared, using Test Method C1220, for analysis using this practice.

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

4.3 Samples 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 material removed from a waste form by aqueous leaching.

4.4 Additional general guidelines are provided in Guide C1009, Specification D1193, Terminology C859, and Terminology E135.

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 % 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

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