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

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1. Scope

1.1 This test method 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 (nonradioactive) 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:*

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Wastes[2]

D 1129 Terminology Relating to Water[3]

D 1193 Specification for Reagent Water[3]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *deemission spectroscopy*—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 *nonspectral 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*)

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[1] This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] *Annual Book of ASTM Standards*, Vol 12.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

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 *specific 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 Test Method

4.1 The general principles of emission spectrometric analysis are given in Ref **(1)**.[5] In this test method, 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 absolutre 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 test method 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 test method are required. The leachant may be deionized water or any aqueous solution containing less than 1 % total solids.

5.2 This test method 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 test method 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 test method 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.[6] 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 (HNO_3).

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 % HNO_3. 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.

NOTE 1—Solutions in polyethylene bottles are subject to transpiration losses that may affect the assigned concentration values.

7.6.2 Prepare the instrument check solution(s) and linearity check solutions in a similar manner.

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

[6] "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."

7.6.3 Fresh solutions should be prepared as needed with the realization that concentrations can change with age.

8. Specimen Preparation

8.1 Filter the leachate through a clean, inert membrane filter having pore size of 0.45 µm or smaller, using an inert filter support (avoid the use of fritted glass supports). Examine the filtered leachate to verify the absence of visible solids or suspended colloids. Compare the analyses of filtered and unfiltered aliquots of the original leachate solution to determine whether the filter membrane contributes to the blank level of the filtered solution. The deposit on the filter may be analyzed separately if required.

8.2 Add sufficient high-purity concentrated nitric acid to bring the leachate sample solution to volume 1 % HNO_3. If the leachate is known to be a chloride solution, or nitric acid is undesirable for other experimental reasons, concentrated hydrochloric or other mineral acid may be used instead. The acid conditions of the calibration and check solutions used in the analytical procedure must match those of the leachate specimen.

9. Analytical Conditions

9.1 *Analytical Lines*—Suggested analytical lines for typical elements are given in Table 1. Additional lines for these and other elements of interest, and information about possible interfering lines, can be found in compilations of analytical lines for ICP-AES **(4-12)**.

9.2 *Selection of Analytical Conditions*—Select an optimum combination of analytical lines, background correction methods, plasma viewing position, and plasma/spectrometer operating conditions to obtain the following for each element:

9.2.1 The lowest attainable detection limit,

9.2.2 The acceptable linear dynamic range,

9.2.3 Avoidance or minimization of spectral and nonspectral interference, and

9.2.4 Best attainable precision.

9.3 Follow the spectrometer manufacturer's recommendation wherever possible in establishing operating conditions. For simultaneous multielement systems, the optimum plasma viewing position and set of operating conditions is usually a compromise **(13)**. The combination of conditions selected must be used in all subsequent ICP operations, including determination of detection limits, calibrations, specimen analyses, and performance checks. Systematic use of an appropriate method such as reference to an atom-to-ion emission intensity ratio **(3)** to ensure reproducible conditions is recommended.

9.4 Determine the detection limit and the upper limit for linear calibration for each element. Use these analytical limits as a guide in the preparation of the calibration reference solutions and linearity check solutions. Determine three sigma control limits for each element through repetitive analysis of the instrument check solution(s). For a particular element, these control limits are the known elemental concentration plus and minus three standard deviations. At least ten independent analyses, distributed randomly with respect to time and laboratory operating conditions, are recommended for estimation of the standard deviations.

TABLE 1 1Suggested Analytical Wavelengths of Typical Elements for ICP-AES[A]

Element	Suggested Wavelength, nm	Estimated Detection Limit, mg/L	Alternative Wavelength, nm	Estimated Detection Limit, mg/L
Aluminum	308.22	0.04	237.32	0.03
Americium	283.23	0.01	292.06	>0.01
Arsenic	193.70	0.05	189.04	0.01[B]
Barium	493.41	0.002	455.40	0.001
Beryllium	234.86	0.0003	313.04	0.0003
Boron	249.77	0.005	249.68	0.005
Cadmium	214.44	0.002	...	...
Calcium	317.93	0.01	393.37	0.0002
Cerium	418.66	0.05	413.76	0.05
Chromium	267.72	0.007	205.55	0.006
Dysprosium	353.17	0.01	205.50	...
Gadolinium	342.25	0.01	...	...
Iron	259.94	0.006	273.96	0.02
Lanthanum	408.67	0.01	379.48	0.01
Lead	217.00	0.09	220.35	0.04
Lithium	670.78	0.002	..	...
Magnesium	279.55	0.0001	279.08	0.03
Manganese	257.61	0.001	294.92	0.008
Molybdenum	202.03	0.008	203.84	0.01
Neodymium	406.11	0.1	401.22	0.05
Neptunium	382.91	0.09	456.04	0.13
Nickel	231.60	0.02	221.65	0.01
Phosphorus	214.91	0.08	178.29	[B]
Plutonium	300.06	0.03	297.25	0.03
Potassium	766.49	0.04	...	...
Rhodium	343.49	0.06	233.48	0.04
Ruthenium	240.27	0.03	...	...
Samarium	442.43	0.05	...	...
Selenium	203.99	0.1	196.03	0.08[B]
Silicon	288.16	0.03	212.41	0.02
Sodium	589.00	0.03	330.24	1.9
Strontium	421.55	0.0008	407.77	0.0004
Sulfur	180.73	[B]	...	...
Technetium	254.32	0.002	261.00	0.002
Tellurium	214.28	0.04	214.72	0.2
Thorium	401.91	0.08	...	...
Titanium	337.28	0.007	334.94	0.004
Uranium	385.96	0.25	367.01	0.3
Vanadium	292.40	0.008	...	...
Zinc	213.86	0.002	206.20	0.006
Zirconium	343.82	0.008	339.20	0.008

[A]See Refs **(4-12)** for a more complete list. Check those references also to identify any possible interfering spectral lines from components such as rare earths, actinides, or high-concentration components.

[B]Vacuum spectrometer.

10. Calibration

10.1 *Calibration of the Spectrometer System*:

10.1.1 Follow the spectrometer manufacturer's instructions for forming and stabilizing the plasma. Allow sufficient time for plasma stabilization before making measurements.

10.1.2 Calibrate the spectrometer system using the calibration blank and the appropriate calibration reference solutions, following the same procedure as for specimen analysis (see Section 11). Obtain separate intensity measurements at the analytical line peak position while introducing the calibration blank and the calibration reference solution. Subtract the analytical line peak intensity determined during analysis of the calibration blank from that of the calibration reference solution analysis to determine the net intensity related to concentration. Then check the accuracy of the calibration by analyzing the instrument check solution(s). The values obtained must fall within the previously determined control limits (3σ) or the instrument must be recalibrated. Finally, check calibration linearity by analyzing the linearity check solutions. The values

obtained must be accurate to within 5 % of the known solution concentration for all elements. It is not necessary to check for calibration linearity every time the spectrometer is calibrated provided that the analysis conditions have not been altered.

10.1.3 The calibration range for each element should be limited to a linear ratio of the net signal intensity and concentration.

10.1.4 Frequency of calibration required depends on instrument stability. Common practice is to verify correct calibration daily at start-up and to recalibrate whenever the check solutions analysis indicates the need (see Section 11).

11. Procedure

11.1 *Leachate Solution Analysis*:

11.1.1 Follow the spectrometer manufacturer's instructions for forming and stabilizing the plasma. Allow sufficient time for plasma stabilization before making measurements.

11.1.2 Analyze specimens using the instrument operating procedure recommended by the manufacturer. A minimum of three sequential spectral integrations is recommended for determining the average (reported) concentration for a single specimen analysis. Provide a dilute acid rinse between specimens, with sufficient rinse time to ensure analyte signals return to blank concentrations. The calibration blank may be used for this purpose. If a high concentration of any element is observed in the specimen, the calibration blank should be analyzed to verify that no carryover memory effect has occurred. If carryover is observed, repeat rinsing until the correct blank value is obtained. After introducing each specimen or blank, allow sufficient time for complete equilibration before initiating data collection. Analysis for elemental concentrations beyond the linear calibration range should be conducted by reducing the specimen concentration to the linear range by appropriate dilution and acidification of the specimen.

11.1.3 Analyze the instrument check solution(s) after every group of ten specimens has been analyzed. Precede the analysis of the instrument check solution(s) by analysis of the calibration blank to verify that the routine rinse procedure prevents memory effects from previous specimen analyses. Analyze the instrument check solution(s) more frequently if nonroutine leachate specimens or analytical conditions are suspected. The concentrations obtained should fall within the three sigma precision limits. If not, the spectrometer must be recalibrated and all specimens analyzed since the last satisfactory check solution analysis must be reanalyzed. If serious instrumental drift occurs, the analysis of specimens should be halted and cause of the instability sought and eliminated. Concentrations must fall within the linear range of each element. Otherwise, specimens must be diluted and reanalyzed.

11.1.4 The stability of the baseline for each element is determined by comparison of successive analyses of the calibration blank. For automated instruments for which negative analytical values are reported only as <0, the baseline stability should be monitored by periodic analysis of instrument check solution(s) containing concentrations approximately twice the detection limit concentrations. Analysis for baseline drift is recommended after every group of ten specimens.

12. Corrections

12.1 *Spectral Interference*—Spectral interference can usually be avoided by judicious choice of analytical wavelengths **(14)**. When spectral interference cannot be avoided, the necessary corrections should be made using the empirical method of spectral interference correction, using either the operating computer software supplied by the spectrometer manufacturer or the manual method detailed below. The empirical correction method cannot be used with scanning spectrometer systems if both the analytical and interfering lines cannot be located precisely and reproducibly. With any instrument, the analyst must always be alert to the possible presence of unexpected elements producing interfering spectral lines.

12.1.1 The empirical method of spectral interference correction uses interference correction factors **(15)**. Interference correction factors are determined by analyzing the single-element, high-purity stock solutions under conditions matching as closely as possible those used for specimen analysis. Unless plasma conditions can be accurately reproduced from day to day, or for longer periods, interference correction factors found to affect the results significantly must be redetermined each time specimens are analyzed **(3, 16, 17)**.

12.1.2 Interference correction factors, K_{ij}, are calculated from apparent concentrations observed in the analysis of the high-purity stock solutions.

$$K_{ij} = (A_i - C_i)/B_j \quad (1)$$

where:
K_{ij} = interference correction factors,
A_i = apparent concentration observed for element i,
C_i = concentration of i observed for the blank, and
B_j = actual concentration of interfering element j.

12.1.3 Specimen concentrations observed for element i (already corrected for baseline drift) are corrected for spectral interferences from elements j, k, and l, for example:

$$S_i = [(A_i - K_{ij})\cdot B_j] - [K_{ik}\cdot(B_k - K_{il})\cdot B_l] \quad (2)$$

where:
S_i = concentration of element i corrected for spectral interference,
A_i = observed concentration of i,
B_j = observed concentration of interfering element j,
B_k = observed concentration of interfering element k,
B_l = observed concentration of interfering element l,
K_{ij} = interference correction factor,
K_{ik} = interference correction factor, and
K_{il} = interference correction factor.

12.1.4 Interference correction factors may be negative if off-peak background correction is employed for element i. A negative K_{ij} can result where an interfering line is encountered at the background correction wavelength rather than at the peak wavelength. The concentrations of interfering elements j, k, and l must have been determined within their respective linear ranges to approximate the actual concentrations as closely as possible. Mutual interferences (i interferes with j and j interferes with i) require iterative or matrix methods for calculation.

12.2 *Nonspectral Interference*—Nonspectral interference is not likely to occur in ICP-AES if the matrix of the specimens matches the matrix of the standards. This condition generally

holds for the low-concentration leachants to which this test method applies. Further ensurance of avoiding nonspectral interference can be achieved by preparing all standard solutions using the original leachant solution as the matrix (matrix matched standards) instead of simply pure water or acid.

12.2.1 If nonspectral interference correction is determined to be necessary, the method of standard additions can be used **(18)**. The method of standard additions is applicable under the following conditions:

12.2.1.1 The chemical and physical form of the analyte in the standard addition is the same as the analyte in the specimen, or the analytical source (ICP) is capable of converting the analyte in both specimen and addition to the same form so that transport, automization, and excitation processes do not differ;

12.2.1.2 The interference effect is independent of analyte concentration over the concentration range of standard additions; and

12.2.1.3 The analytical calibration curve is linear over the concentration range of standard additions.

12.2.2 The method of standard additions involves the addition of a known concentration of analyte to the specimen. The concentration of the addition should be not less than 50 % nor more than 100 % of the analyte concentration in the specimen so that measurement precision will not be degraded and so that interferences which exhibit a dependence on analyte/interfering element ratios will not cause erroneous results. The method must be applied to all elements in the specimen set individually. Multielement standard addition **(19)** can be used if it has been determined that added elements do not produce interferences.

13. Precision and Bias

13.1 The precision of this test method will depend on the choice of instrumentation, analytical wavelengths, operating conditions, etc. Typical values for short-term precision (based on replicate measurements performed at concentrations at least 100 times the detection limit) range from 0.3 to 2 % relative standard deviation. Precision degrades with decreasing concentration to approximately 25 % relative standard deviation at approximately two times the detection limit.

13.2 The bias of this test method is dependent on the reliability of the calibration reference solutions, the amount of spectral and nonspectral interference, the accuracy of interference corrections, and the adherence to the calibration drift specifications **(20)**. Using accurate calibration reference solutions, and with all necessary corrections applied correctly, the relative analytical bias for element concentrations that are at least ten times the detection limit will be approximately equal to the calibration drift.

13.3 A negative bias will result from the specimen dilution that occurs if addition of acid is required during specimen preparation (8.2). This bias can be eliminated by correcting the analyzed concentration using the appropriate dilution factor.

13.4 Table 2 gives typical data for ICP-AES analyses of a simulated leachate used to evaluate analytical precision and bias **(21)**.

TABLE 2 Standard Deviation of ICP-AES Analysis of Simulated Leachate Solutions SL-1[A]

Element	Concentration, mg/L	Standard[B] Deviation, mg/L	Relative Standard Deviation,%
Boron	47.1	0.5	1.06
Calcium	0.021	0.0053	24.8[C]
Cerium	2.01	0.04	1.99
Iron	0.014	0.003	21.4[C]
Molybdenum	1.92	0.03	1.56
Neodymium	1.60	0.03	1.88
Silicon	18.9	0.18	0.95
Sodium	79.6	1.4	1.76
Strontium	5.13	0.03	0.58

[A] Analytical data from twelve analyses conducted at random times on each of 4 successive days. Each analysis consisted of the average of triplicate contiguous burns of the solution (routine procedure).
[B] Standard deviation of individual analysis: $n = 12$ (See Ref. **(21),** Table 6.11).
[C] Concentration of Ca and Fe is approaching the detection limit.

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