



Standard Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste¹

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1. Scope

1.1 This test method covers the relative chemical durability of simulated and radioactive monolithic waste forms, such as glasses, ceramics, or cermets, in various test solutions at temperatures $<100^{\circ}\text{C}$ under low surface-area-to-volume (S/V) ratio conditions.

1.2 This test method can be used to distinguish differences in the leaching behavior of various simulated or radioactive waste forms under the specific conditions of the test based on analysis of the test solution. Data from this test are used to calculate the normalized elemental mass loss from specimens exposed to aqueous solutions at temperatures $<100^{\circ}\text{C}$.

1.3 Specimen surfaces may be altered during this test. These altered surfaces may be used to study the reaction of monolithic waste forms during static exposure to solutions.

1.4 This test method must be performed in accordance with all applicable quality assurance requirements for acceptance of the data.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.3.2.

2. Referenced Documents

2.1 ASTM Standards:²

C 1109 Test Method for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectrometry

C 1174 Practice for Prediction of the Long-Term Behavior of Waste Package Materials Including Waste Forms Used

in the Geologic Disposal of High-Level Nuclear Waste
D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

D 1293 Test Methods for pH of Water

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

2.2 EPA Document:³

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

3. Terminology

3.1 Definitions:

3.1.1 *accumulated dose*—the sum of the absorbed doses received by the system considered regardless of whether it is exposed to radiation in a continuous or discontinuous fashion.

3.1.2 *accuracy*—the closeness of agreement between the accepted reference value and individual results (Practice E 177).

3.1.2.1 *Discussion*—In its usage in this test method, *accuracy* includes the effects of precision and bias. The term is applied to measurements wherein a specific standard reference is available such as NIST standard mass and reference solutions traceable to a standards organization. The term “accurate to within” a specified range means that individual measurements on a reference standard are always within the specified range, for example, within 2°C of a certified NIST thermocouple, within 0.5 mg of a NIST standard mass or within 10 % of the value for a reference solution.

3.1.3 *actinide*—any element with atomic number of 89 to 103.

3.1.4 *bias of a measurement process*—a generic concept related to a consistent or systematic difference between a set of test results from the process and an accepted reference value of the property being measured (Practice E 177).

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.07 on Waste Materials.

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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 SW846A, 3rd Ed., Revision 1, U.S. Environmental Protection Agency, Washington, DC, December 1987.

3.1.5 *chemical durability*—the resistance of a glass, ceramic, or cermet test specimen to the release of its constituents to an aqueous solution under the specific conditions of this test.

3.1.5.1 *Discussion*—The response of glass, ceramics, or cermets under other conditions is outside the scope of this test method.

3.1.6 *closed system*—a system that precludes the transport of matter into or out of the system.

3.1.7 *high-purity water*—ASTM Type I or Type II water with a maximum total matter content of 0.1 g/m³, a minimum electrical resistivity of 16.67 MΩ·cm at 25°C, and no detectable soluble silica (see Specification D 1193 and Terminology D 1129).

3.1.8 *ion selective electrode (ISE)*—a device for measuring F⁻.

3.1.9 *leachant*—a solution used, or intended for use, in leaching.

3.1.10 *leachate*—the solution resulting from a leach test.

3.1.11 *leaching*—the action of removing soluble constituents from a solid into a solution.

3.1.12 *monolithic specimens*—specimens that are physically one coherent piece, as opposed to powdered specimens that consist of many small pieces of irregular configuration. Monolithic specimens may consist of several individual phases, but they must be bound in a stable coherent configuration.

3.1.13 *nuclear waste forms*—solid materials in which radioactive wastes have been immobilized.

3.1.14 *open system*—a system that permits the transport of matter into or out of the system, e.g., O₂ and/or CO₂ diffusion into or out of the system.

3.1.15 *polytetrafluoroethylene (PTFE)*—a material produced from various monomers that are polymerized into the plastic.

3.1.16 *precision of a measurement process*—a generic concept related to the closeness of agreement between test results obtained under prescribed like conditions from the measurement process being evaluated (Practice E 177). In this test method, precision will be measured by either standard deviation or relative standard deviation.

3.2 *Abbreviations:*

3.2.1 *EDX*—energy-dispersive x-ray fluorescence instrument (or analysis).

3.2.2 *ICP*—inductively coupled plasma.

3.2.3 *PFA*—perfluoroalkoxy.

3.2.4 *SEM*—scanning electron microscope (or microscopy).

3.2.5 *TEM*—transmission electron microscope (or microscopy).

3.2.6 *XRD*—x-ray diffractometer (or diffraction).

4. Summary of Test Method

4.1 Specimens of known volume and geometric surface area are immersed in the reference leachants without agitation for defined time periods at defined temperatures. The S/V ratio is held constant within 0.5 of 10.0 m⁻¹. Three reference temperatures, 40°, 70°, and 90°C, and a number of specific time periods are identified in a series of test matrices established to meet objectives that include evaluation of waste forms for comparative purposes. In the test method, three reference

leachants are used: high-purity water and two solutions (silicate/bicarbonate and brine) that approximate fluids that the waste form may encounter in a geologic repository. In addition to the reference leachants, others may be used. The test is for application to simulated waste forms and to radioactive specimens.

4.1.1 PTFE test vessels and PTFE specimen supports are used, provided the integrated dose to a PTFE component from all radiation (alpha, beta, or gamma) does not exceed 10⁴ rad (100 Gy), which has been shown to not damage PTFE.⁴ If the integrated dose to the test vessel and specimen support exceeds 10⁴ rad, 304L stainless steel or fused silica vessels and specimen supports are to be used (in such tests involving brine leachants, fused silica vessels and components must be used because of the corrosion of stainless steel by the brine) (see Note 1).

NOTE 1—These modifications to the test method are required when using highly radioactive waste forms.

4.2 Separate specimen and leachant volumes are required for each data point. The test results are based on leachate analyses in all cases and include examination of the leached specimen surface after long-duration tests.

5. Significance and Use

5.1 This test method is intended principally to distinguish differences in the leaching behavior of candidate monolithic, inorganic, radioactive waste forms under low S/V ratio conditions. The test method can be used to produce altered solid specimens to study the reaction of monolithic waste forms during static exposure to solutions. Data from this test may form part of the larger body of data that is necessary in the logical approach to long-term prediction of waste form behavior, as described in Practice C 1174. In particular, solution concentrations and characterization and altered surfaces may be used in the testing of geochemical modelling codes. This test method excludes study of powdered or organic materials. This test method can be used as either a “characterization” or “accelerated” test under the protocol of Practice C 1174, mentioned above.

5.2 The total absorbed dose for each PTFE test vessel may not exceed 10⁴ rad (100 Gy) during the lifetime of the vessel. Hence, a record of the absorbed dose each vessel receives must be maintained.

5.2.1 More radiation-resistant materials are used when testing in radiation fields where the accumulated absorbed dose exceeds 10⁴ rad (see Note 1).

5.3 Both aerated and deaerated solutions may be used in this test method. However, when testing highly radioactive specimens, tests with deaerated solutions are mandatory to minimize radiolysis effects of nitrogen (see Note 1). Control of the oxygen fugacity is not part of this test method. Such control and measurement may be required for specific uses of leaching data but are beyond the scope of this test method. Preparation of deaerated leachants is covered in 7.2.2.

⁴ Strachan, D. M., “Effect of Gamma Irradiation on Simulated Waste Glass Leaching and on the Leach Vessel,” *Journal of the American Ceramic Society* 66[9], C-158-C-160, 1983.

5.4 The use of PTFE test vessels may result in some F⁻ release from the vessel to the solution. For PTFE vessels that meet the qualification requirements of this test method (see 6.4), the amount of release at low radiation levels, <10⁴ rad, is not significant. Fluoride, at the concentrations encountered in tests where the radiation dose is limited to less than 10⁴ rad and qualified PTFE is used, has not been demonstrated to have an effect on leaching behavior.⁴ The primary reason for limiting the integrated dose to PTFE vessels and specimen supports to 10⁴ rad and requiring that the PTFE vessels be qualified is to ensure that excessive fluoride releases do not occur (see Note 1). In order to monitor fluoride releases, which could have an influence on test results, analysis for F⁻ concentration is a test requirement.

6. Apparatus and Analytical Requirements

6.1 Fig. 1 illustrates the basic features of the test equipment. The specimen is held near the centroid of the leach volume, for example with a monofilament or by use of a coarsely woven support screen. The specimen surface-area-to-leachant-volume (S/V) ratio must be within 0.5 of 10.0 m⁻¹.

6.2 *Test Vessel Material*—The choice of material for construction of the test vessels will depend on the radiation field. When testing is performed in fields that yield an absorbed dose of less than 10⁴ rad, use PTFE PFA vessels that have been qualified (see 6.4).

6.2.1 For fields where the absorbed dose exceeds 10⁴ rad, use fused silica or 304L stainless steel. If the vessel dose exceeds 10⁴ rad and the brine leachant is employed, fused silica vessels are to be used (because of the corrosion of stainless steel by the brine). Use of the same vessel material throughout the test matrix will allow an evaluation of the contribution to the leachate by the vessel, for example, silicon from fused silica. (See Note 1.)

6.2.2 Teflon vessels are considered to provide open systems because they are pervious to carbon dioxide and some water loss.

6.2.3 Stainless steel and fused silica vessels are considered to provide closed systems because they are impervious to carbon dioxide, and water loss is usually negligible.

6.3 *Test Vessel Size*—The vessels shall have a diameter-to-height ratio between 0.5 and 2.0. Leach vessel volumes will generally be between 20 mL and 1 L. The vessels shall be sufficiently impervious and have a tight-fitting lid to limit leachant loss during the test to less than 10 % of the original volume. The specimen support shall be constructed of the same material as the vessel or of equally inert material and designed to maintain the specimen near the centroid of the leach volume, but must not contact more than 5 % of the specimen surface area.

6.4 *Identification of Vessels and Cleaning History*—Vessel identification and the cleaning history of each vessel must be maintained during testing.

6.4.1 *Identification Marking*—A unique identifying number should be permanently marked on each leach vessel. The same number should be permanently marked on the companion lid.

6.4.2 *Record of Vessel Cleaning History*—Each batch of cleaned vessels shall be labeled with a unique batch number. A log book of the leach vessel number and date of cleaning shall be kept. The date can be used as the batch number identifier if only one batch has been cleaned on that date. Alternatively, a separate batch number can be assigned and recorded. In this manner, any inconsistent test responses might be traced to improper cleaning of a batch of vessels or to a problem vessel.

6.5 *Qualification of PTFE Lot for Use as Test Vessels*—Variations in manufacturing practice may cause particular lots of PTFE to have unacceptable amounts of fluoride release from PTFE vessels during leach tests. Therefore, the vessels from a particular lot must be qualified for use by performing a blank test for 28 days to ascertain and document that the fluoride release is acceptably low for 28-day tests. The suitability of a particular lot of PTFE for longer-term tests is dependent on similar checks of fluoride release in blank tests conducted for the longer test durations, which is a test requirement. That is, the fluoride level must always be checked on the blanks and leachates used during testing. It is imperative that the vessels for the blanks be from the same lot as the other vessels used for testing. Measurement of pH shall also be determined in these qualification tests, as well as in the analyses of test leachates. The test matrices in 9.5 require the use of blanks, which will further document that excessive F⁻ release from the vessel has not occurred during testing.

6.5.1 To qualify a lot of PTFE, clean three vessels as described in 6.6 and run a 28-day blank test using the three vessels at 90°C with deionized water. Measure the pH and F⁻ concentration. If the pH is in the range of 5.0 to 7.0 and the F⁻ is below 0.5 µg/ml, the lot of PTFE is acceptable for use.

6.6 *Preparation of Vessel*—New PTFE vessels and supports must be cleaned to reduce the amount of F⁻ released during testing. This cleaning is done by a special procedure using a NaOH solution, described in 6.6.1. PTFE vessels can be reused after testing provided they are cleaned before reuse. However, reuse of PTFE test vessels and supports is not allowed in this test method when these components have been used in tests with actinide-doped specimens. This is because actinides are

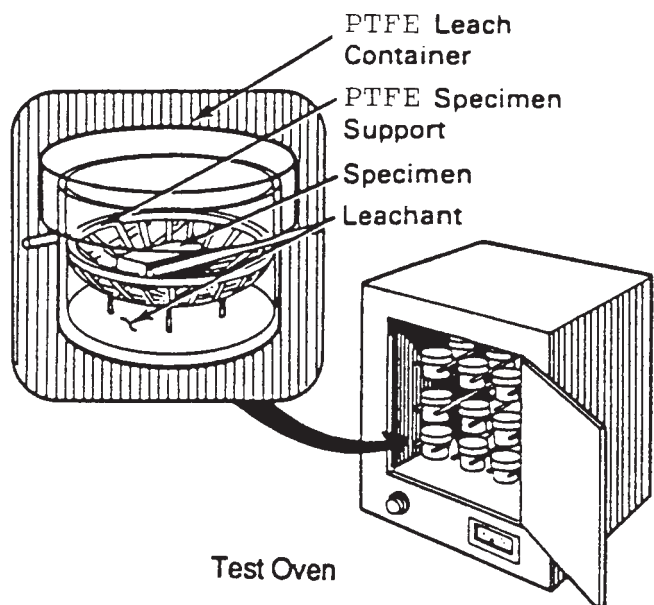


FIG. 1 Example Apparatus for Static Leach Test Method

difficult to remove, and may not be sufficiently removed from the PTFE vessel walls by leachate acidification and the vessel/specimen support structure cleaning procedure. As these dopants may be present in very low concentrations in a leachate, contamination from the vessel walls could be potentially significant. New PTFE test vessels and supports, except fine monofilaments, shall be heated in a $200 \pm 10^\circ\text{C}$ oven for one week prior to cleaning. Both new and used PTFE vessels and PTFE specimen supports, except for fine filaments, must be cleaned according to the specified procedures. Stainless steel and fused silica vessels are cleaned according to different procedures, described in 6.6.3.

6.6.1 *New PTFE Vessels and Supports*—For new PTFE vessels and supports, clean according to 6.6.1.1-6.6.1.20. For used PTFE vessels (cleaned previously according to 6.6.1.1-6.6.1.20), skip 6.6.1.1-6.6.1.20 and clean according to 6.6.2.1-6.6.2.8.

6.6.1.1 Rinse vessels, lids, and supports with fresh high-purity water. Use at least three vessel volumes for each vessel at ambient temperature.

6.6.1.2 Fill vessels approximately 90 % full with 5 wt % NaOH solution.

6.6.1.3 Tighten lids and place vessels in an oven preheated to $110 \pm 10^\circ\text{C}$.

NOTE 2—Use this temperature when using PTFE vessels rated to 0.5 MPa or higher (see 9.4.7). For vessel designs not rated to 0.5 MPa, use an oven temperature of $95 \pm 2^\circ\text{C}$.

6.6.1.4 Retighten the vessel lids after 12 to 24 h in oven.

6.6.1.5 After 7 days in oven, remove vessels and allow to cool to room temperature.

6.6.1.6 Remove lids carefully and dispose of NaOH solution.

6.6.1.7 Rinse vessels and lids in fresh high-purity water.

6.6.1.8 Repeat 6.6.1.7.

6.6.1.9 Place vessels and lids in fresh, boiling high-purity water for a minimum of 1 h.

6.6.1.10 Repeat 6.6.1.7 and 6.6.1.9.

6.6.1.11 Allow vessels and lids to air dry for a minimum of 16 h at $90 \pm 10^\circ\text{C}$.

6.6.1.12 Fill vessels about 90 % full with fresh high-purity water at ambient temperature.

6.6.1.13 Tighten lids and place vessels in oven preheated to $90 \pm 2^\circ\text{C}$ for a minimum of 16 h.

6.6.1.14 Remove vessels and allow to cool to room temperature.

6.6.1.15 Take an aliquot of liquid from each vessel and measure pH.

6.6.1.16 If pH is below 5, repeat 6.6.1.1-6.6.1.16 until pH is above 5.

6.6.1.17 If pH is above 7, repeat 6.6.1.7-6.6.1.17.

6.6.1.18 If pH is between 5.0 and 7.0 take a second aliquot and send for ISE F⁻.

6.6.1.19 If ISE F⁻ level is $>0.5 \mu\text{g/mL}$, repeat 6.6.1.7-

6.6.1.19. If ISE F⁻ is still $>0.5 \mu\text{g/mL}$ repeat 6.6.1.1-6.6.1.19.

6.6.1.20 If ISE F⁻ level is $<0.5 \mu\text{g/mL}$, a vessel is acceptable for use.

6.6.2 *Used Vessels*—Clean used PTFE vessels and supports (that is, vessels and supports that were cleaned according to 6.6.1.1-6.6.1.20 and then used for testing) according to 6.6.2.1-6.6.2.8.

6.6.2.1 Rinse vessels, lids, and supports with fresh high-purity water. Use at least three vessel volumes of water for each vessel.

6.6.2.2 Soak vessels and supports for 1 h in 0.16 M HNO₃ (1 wt % HNO₃) at $90 \pm 10^\circ\text{C}$.

6.6.2.3 Rinse again as specified in 6.6.2.1.

6.6.2.4 Soak for 1 h in high-purity water at $90 \pm 10^\circ\text{C}$.

6.6.2.5 Fill the vessels approximately 90 % full with fresh high-purity water with support in place. Close the lids and hold for at least 16 h at $90 \pm 2^\circ\text{C}$ and then measure the pH of the water from each vessel. Take an aliquot of the water from at least two vessels from each vessel batch and submit for ISE F⁻.

6.6.2.6 Repeat 6.6.2.4 and 6.6.2.5 until the pH is in the range of 5.0 to 7.0 and the F⁻ is $<0.5 \mu\text{g/mL}$.

6.6.2.7 If the pH and fluoride requirements cannot be achieved by three repetitions of 6.6.2.4 and 6.6.2.5, then repeat the cleaning procedure starting at 6.6.2.1.

6.6.2.8 Dry vessels and lids at $90 \pm 10^\circ\text{C}$ for a minimum of 16 h and store inside a clean environment until used.

6.6.3 *Stainless Steel and Fused Silica Vessels*—The procedures in 6.6.1 and 6.6.2 are specifically for PTFE vessels. When using other inert vessels, such as fused silica or 304L stainless steel, variations of these procedures are appropriate.

6.6.3.1 Clean fused silica vessels using 6.6.2.1-6.6.2.8 except delete the check for F⁻, which is specific to PTFE containers.

6.6.3.2 Degrease new 304L stainless steel vessels and lids without gaskets and ultrasonically clean in 95 % ethanol for approximately 5 min (in order to remove any residual grease or oil left from machining operations) and then clean using the following procedure:

6.6.3.3 Rinse three times in high-purity water.

6.6.3.4 Submerge in 0.16 M HNO₃ (1 wt % HNO₃) for 1 h at $90 \pm 10^\circ\text{C}$.

6.6.3.5 Rinse three times with high-purity water at ambient temperature.

6.6.3.6 Submerge the vessels and lids in fresh high-purity water for 1 h at $90 \pm 10^\circ\text{C}$.

6.6.3.7 Rinse with fresh high-purity water at ambient temperature.

6.6.3.8 Fill the vessel 80 to 90 % full with high-purity water. Close the lid and leave in a $90 \pm 2^\circ\text{C}$ oven for a minimum of 16 h.

6.6.3.9 Remove the vessels from the oven and cool to room temperature, then take a cooled aliquot of the water and measure the pH.

6.6.3.10 If the pH is not in the range of 5.0 to 7.0, repeat 6.6.3.6-6.6.3.9.

6.6.3.11 If the pH is not in the range of 5.0 to 7.0 after 3 repetitions of 6.6.3.6-6.6.3.9, repeat the cleaning steps starting at 6.6.2.2.

6.6.3.12 Dry the vessels in a $90 \pm 10^\circ\text{C}$ oven for a minimum of 16 h and then cool to room temperature. If the

vessels are not used immediately, close the vessels and store in a clean environment until needed.

6.6.4 Cleaning of Used Stainless Steel and Fused Silica Vessels—When stainless steel or fused silica vessels are reused subsequent to their use with radioactive specimens, residual contamination may be present. The vessels shall be cleaned before reuse using 0.16 M HNO₃ (1 wt % HNO₃) and high-purity water until the level of the radioactive element of interest is below the detectable level using the analytical method employed for concentration measurement of the leachate. Stainless steel vessels are also checked for Si contamination before reuse.

6.6.4.1 Rinse the vessel and lid with high-purity water. Fill the vessel 80 to 90 % full with 0.16 M HNO₃ (1 wt % HNO₃). Reseal the vessel and place in an oven at 90 ± 2°C for a minimum of 16 h to acid strip any radionuclides adhering to the interior of the vessel.

6.6.4.2 Check the acid stripped solution for radioactivity. Repeat 6.6.4.1 until the radioactivity of the acid strip solution is reduced below the background.

6.6.4.3 Remove the gasket and discard. Rinse vessels and lids thoroughly with high-purity water at ambient temperature. Take precautions to prevent contamination of the vessel interior with any radionuclides present on the exterior of the vessel or in the work environment.

6.6.4.4 Fill the vessel 80 to 90 % full with fresh high-purity water. Close the lid using a new, cleaned gasket (see 6.6.5) and place in oven at 90 ± 2°C for at least 24 h.

6.6.4.5 Remove vessels from oven, then take an aliquot of the water and measure the pH. Take another aliquot and measure the radioactivity. For stainless steel vessels, also measure the Si content of the solution.

6.6.4.6 If the pH is not in the range of 5.0 to 7.0 or the measured radioactivity is not at the background level, or Si is detected for stainless steel vessels, repeat 6.6.4.3–6.6.4.5.

6.6.4.7 If three repetitions of 6.6.4.3–6.6.4.5 do not result in a pH of 5.0 to 7.0, low radioactivity, and Si <1 ppm for stainless steel vessels, then repeat the cleaning starting at 6.6.4.1.

6.6.4.8 Dry vessels, lids, and gaskets at 90 ± 2°C for a minimum of 16 h and store in a clean environment until needed.

6.6.5 Cleaning of New PTFE Gaskets for Stainless Steel Vessels—Clean new PTFE gaskets for stainless steel vessels using the following method:

6.6.5.1 Handle the gaskets only with clean tongs.

6.6.5.2 Clean each gasket ultrasonically in 95 % ethanol for approximately 10 min.

6.6.5.3 Clean each gasket under flowing high-purity water at ambient temperature for approximately 3 min.

6.6.5.4 Bake each gasket in an oven at 200 ± 10°C for a minimum of 4 h.

6.6.5.5 Immerse each cooled gasket in fresh high-purity water in a boiling water bath for a minimum of 2 h.

6.6.5.6 Dry gaskets at 90 ± 10°C for a minimum of 16 h and store in a clean environment until needed.

6.7 Oven—The test oven must be capable of controlling the temperature of the test vessels to within 1°C over the range of 40° to 100°C. Determine the zone within the loaded chamber that is constant within 1°C of the target temperature using at least ten points of temperature measurement. A temperature recorder or other monitoring device must be provided to ensure that the desired temperature has been maintained for the duration of the test. When radioactive specimens are used, ensure that self-heating does not prevent the maintenance of the desired leaching temperature (see Note 1). Brief fluctuations from the desired temperature are allowable when specimens are placed in or removed from the test oven, but the cumulative time of these fluctuations outside 1°C of the target temperature must not exceed 5 % of the test period and no fluctuation may be more than 5°C above the target temperature.

6.8 Balances—Balances shall provide the following accuracies, depending on the materials being weighed:

Leachant + vessels	within 0.25 % of the leachant mass
Chemical reagents	within 1 % of the reagent mass
Specimens	within 0.5 mg

TABLE 1 Required Calibration Schedule

Measurement	Device	Frequency Check and Methods
Temperature	thermocouple or thermometer	6 months NIST standard or ice/boiling water
	electronics or temperature probe (without sensor)	6 months against a calibrated millivolt source
Length	micrometer	6 months standard foils, gage blocks
Mass	balance	3 months NIST standard masses
Chemical concentration	analytical method	3 months NIST standards, where possible, 2 times daily (routine), secondary standards
pH	pH meter	each day of use with commercial buffer solutions, and at intervals of 30 to 60 min during measurements. See Test Methods D 1293 and 6.11 for pH measurement guidance
Volume	volumetric flasks	use certified flasks 3 months by measuring the mass of pure water contained
	pipettes	3 months by measuring the mass of pure water contained
Activity	counting techniques	twice a day, before and after counting NIST or NIST-traceable standard isotope source of interest

6.9 *Volume Measurement*—Measure leachant volumes gravimetrically or with pipettes, burettes, or flasks calibrated as described in Table 1 (see also 6.10) and accurate to within 1 % or better.

6.10 *Solution Analysis*—Measure solute concentrations using equipment standardized with standards traceable to NIST, preferably, or other recognized organizations, such as EPA or USGS. Determine and report precision and bias. Although analytical results should normally be accurate within 10 % of the reference solution when checked by individual measurements on reference solutions, this may not be possible when concentrations in the solution approach detection limits. The detection limits for each analysis must accompany the reported result. Various analytical techniques can be used to determine the solute concentrations in leachates, including inductively coupled plasma spectroscopy (see Test Methods C 1109 or EPA SW846a, or both), direct current plasma spectroscopy, atomic absorption emission spectroscopy, and neutron activation. Selection of a specific technique depends on specific test objectives and the particular solutes of interest. For radioactive elements such as actinides and fission products, where low amounts may be of interest, radiochemistry/radiation counting may be needed or desirable. Use of blanks and simulated leachates, as discussed in Section 10, help ensure that high-quality data are obtained.

6.11 *pH Measurement*—Measure the pH to an accuracy of 0.1 unit using a calibrated meter. Use Test Methods D 1293, Method A and commercial buffers to make this measurement. When measuring the pH of deaerated solutions, make the measurement under an argon atmosphere.

6.12 *pH Measurement in Brines:*

6.12.1 Determination of pH, defined as the negative logarithm of hydrogen ion activity, in concentrated brines using standard glass electrodes is complicated by two principal factors: (1) a significant liquid junction potential and (2) significant differences between hydrogen ion concentrations and activities. The result is usually a measured pH value that is significantly smaller than the actual value.

6.12.2 A potential at the junction between the reference electrode filling solution and the sample solution (“liquid junction potential”) is present any time these two solutions are different. The potential arises from the interdiffusion of ions in the two solutions. Since these ions diffuse at different rates, the electrical charge will be carried unequally across the junction. This results in a potential whose magnitude and stability depends on the composition of the solutions as well as the type of junction. Glass pH electrodes are usually standardized against buffers to establish the pH scale. These buffer solutions, however, are of much lower ionic strength than concentrated brines, so that significantly different liquid junction potentials are present in the two cases. The usual result in a saturated brine is a measured pH value that is one or more pH units smaller than the actual value.

6.12.3 In addition, changes in ion activity coefficients as a function of ionic strength can be important in pH measurements. (Activity is equal to the product of concentration and the activity coefficient.) Activity coefficients are usually unity in dilute solutions. However, in solutions of high ionic

strength, average cation-anion activity coefficients can be much larger than unity (for example, greater than 10 for HCl in saturated magnesium chloride solutions). Because glass electrodes respond to ion activities, not to concentration, there can be a large effect on the measured pH value. The situation is complicated by a lack of activity coefficient data in concentrated brines and a similar lack of theoretical models applicable to such solutions.

6.12.4 Considerable caution must be exercised, therefore, when attempting to interpret brine pH measurements. Liquid junction and ion activity coefficient effects will result in measured pH values being shifted significantly from the true pH (based on H⁺ ion activities). However, for a given brine system, these contributions should be constant for a large portion of the pH scale, such that trends in the pH will be unaffected. If major changes in brine composition occur during a given experiment, even trends in measured pH may be suspect. In this test method, the reference brine is a diluted brine derived from analysis of Brine A for the Salado Region in New Mexico.⁵ Other simulated, site-specific reference brines may be used depending on the particular application of the test. The bulk composition of the brine solution is not likely to change significantly in leach tests, and the measurement of pH to indicate trends of the actual pH during testing is expected to be valid. For distilled water and the silicate water, the measurement of pH is relatively straightforward.

6.13 *Calibration and Standards*—Calibrate all instruments used in these tests initially, and periodically, to minimize possible errors due to drift. Table 1 shows the methods and the minimum frequency of calibration for the various devices used. Use standardization procedures that are published by recognized authorities such as NIST or ASTM.

7. Leachant Preparation and Storage

7.1 *General Chemicals and Procedures*—Use chemicals of reagent grade or better that conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶

7.1.1 It is recommended that each chemical be analyzed to determine if impurities, once the leachant is prepared, will exceed detection limits of the leachate analysis system to be used. If impurities will cause detection limits to be exceeded, obtain a different batch of the chemical or use an ultrapure chemical. Good laboratory practice should be used at all times to minimize contamination of the leachant.

7.2 *Water*—The water referred to in this procedure is air-saturated (except when deaerated solutions are required), reagent water Type I or II conforming to Specification D 1193, which has a total impurity level, including organics, of less than 0.1 mg/L.

⁵ Molecke, M. A., “A Comparison of Brines Relevant to Nuclear Waste Experimentation,” *Sandia Report SAND83-0516*, Sandia National Laboratories, 1983.

⁶ *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 *Annual 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.

7.2.1 When working with radioactive materials, radiolysis of dissolved gases in water becomes an important factor; therefore deaerated leachants should be used.

7.2.2 To deaerate water for use in leachant preparation, purge the boiling, high-purity water for 15 min with argon. Immediately place the hot water under an argon atmosphere. Prepare the leachants as described below using the cooled, deaerated water in an argon atmosphere.

7.3 *Preparation of Brine Leachant*—Prepare the brine by dissolving 48.2 g KCl, 90.0 g NaCl, and 116.0 g MgCl₂ (247.9 g MgCl₂·6H₂O) in sufficient water to make approximately 900 mL of solution. Adjust the pH to fall within the range of 6.4 to 6.6 by dropwise addition of 0.01 M NaOH or 0.01 M HCl. Then add water to make 1.00 L of solution. Analyze the leachant to verify the composition and to determine impurity concentration. Discard the leachant if the concentration of any constituent is in error by more than 10 % from the calculated recipe concentration.

7.3.1 The density of the brine leachant at 23°C is within 0.005 of 1.1790 g/cm³. This value may be used if the aliquots of brine leachant for the individual leach tests are measured by weight rather than volume.

7.3.2 **Caution**—When using brine in radiation fields, hydrogen gas will be generated and may pressurize the test vessel. Take precaution when making such studies. Use sturdy test vessels or vessels with gas vents, or both. Also, since chloride brines can become very corrosive under high radiation fields, use caution when selecting the test vessel material. (See Note 1.)

7.4 *Preparation of Silicate Water Leachant*—Prepare the silicate water leachant by dissolving 0.179 g NaHCO₃ and 0.058 g SiO₂ as silicic acid in sufficient water to make about 900 mL of solution. Adjust the pH to within 0.1 of 7.5 with 0.01 M HCl. Add water to make 1.00 L of solution. Analyze the leachant to verify the composition and to determine impurity concentration. Discard the leachant if the concentration of any constituent is in error by more than 10 %.

7.5 *Repository Waters*—When additional leachants, representative of specific repository waters, are used, the rigor with which the data are obtained must be the same as for the three reference leachants. Record the type of repository water used, where and when it was obtained (or how it was prepared if made up in the laboratory), and its chemical analysis. In addition, verify that the repository water remained stable for the duration of the test and did not react significantly with the test vessel. This information will include the quantity, composition, and identity of any precipitate that may have been formed in the blanks during the test.

7.6 *Leachant Storage*—Use polyethylene or polypropylene bottles with tight-fitting lids to store the leachants. Before use, rinse these bottles with three volumes of 6 M HNO₃, each rinse equal to 20 % of the vessel volume; three volumes of water, each equal to 20 % of the vessel volume; and two volumes of freshly prepared leachant, each equal to 10 % of the vessel volume.

7.6.1 Use the leachant immediately or place in a dark location and store in a sealed vessel until beginning the test. If the leachant is not used immediately, report the storage time. Verify the composition by analysis before use.

7.6.2 Store deaerated solutions in vessels with an argon atmosphere above the liquid level and for no longer than one week.

8. Test Specimen Preparation

8.1 The specimens may be either fabricated individually or cut from larger samples of the waste-form material. When cutting specimens from larger samples, avoid the use of wax or adhesives to hold the sample to be cut. If such materials must be used, none of the surfaces on the final leach test specimens shall be surfaces to which the adhesive was applied.

8.2 When performing experiments using deaerated solutions, a final specimen cleaning step must be carried out under an argon atmosphere using deaerated deionized water. Sawing and cutting of the test specimens need not be done in argon.

8.3 *Characterization of Test Material*—Document the fabrication method and fabrication conditions for the sample from which test specimens are prepared. Provide information on how specimens were selected from the fabricated material. The researcher must include information on the chemical and radiochemical (if applicable) composition and compositional variations of the sample from which the test specimens are obtained. This information should be obtained from bulk chemical and radiochemical analyses. Include information on chemical-composition variations within the fabricated material, as well as within and between specimens. For certain radioactive samples, autoradiography may be necessary to demonstrate homogeneity with respect to alpha-emitting isotopes. The researcher may also wish to characterize the sample and/or test specimens by optical microscopy, XRD, and SEM-EDX. Use these techniques to document microcracking, phase identification, relative concentrations of phases, and homogeneity both within and between specimens. For specimens in which an as-fabricated surface is to be leached, analyses by surface spectroscopy of a surface cross-section by SEM-EDX or other applicable techniques are also required to determine whether the surface composition differs from the bulk composition. If differences exist, discuss the effect on the report of test results.

8.3.1 When the waste form is heterogeneous, or multiphase, the test operator must ensure that the test specimens contain a representative distribution of the different phases, as documented by optical microscopy, SEM-EDX, or other applicable techniques. Further, for some multiphase waste forms, individual phase particles may be quite large. To ensure that the presence of a large individual phase particle does not bias the result obtained with any single test specimen, the following requirements must also be met: individual test specimens shall be sized such that the largest dimension of any individual phase particle does not exceed one-half the smallest dimension of the test specimen. Also, the surface area of any individual phase particle shall not exceed 10 % of the total geometric surface area of the test specimen.

8.4 *Test Specimens*—The test specimen is monolithic and is generally a single piece with regular shape, such that the area

exposed to the leachant is easily determined. The specimens must be representative of the bulk waste form. Only cut surfaces are to be leached. All surfaces exposed to the leachant must be prepared in a consistent manner using either an abrasive saw or an alternative technology to a standard surface finish (200, 320, 600 grit or other surface finish). The surface finish *will affect* the normalized release rates obtained in this method and is a user-specified variable.

8.4.1 *Specimen Cutting*—When specimens are prepared from a larger sample, use a saw or core drill with a 200-grit, diamond-impregnated cutting surface.⁷ Use water as the cutting fluid. The size or shape of the specimen is not critical, but a surface area of about 400 mm² is recommended. The following technique is recommended:

8.4.1.1 Read the operator's manual provided with the saw. Become familiar with basic wafer blade sectioning technique.

8.4.1.2 Fill the lubricant pan with water or other appropriate cutting fluid to a level that will immerse the blade approximately ¼ in. The specimen basket will not be used with a 5-in. diameter blade.

8.4.1.3 Ensure that the blade is mounted securely using the 2½-in. diameter recessed flanges provided with the saw.

8.4.1.4 Blades must be dressed before first use and each time before starting a cut. Dress new blades before first use for a total of seven cuts through a dressing stick. During subsequent dressing operations, a single cut through the dressing stick is sufficient. It is recommended that the dressing chuck be used for holding the dressing stick during this operation to help prevent broken blades. The blades must be flat to within approximately ⅛ in. with no nicks or dents.

8.4.1.5 Secure the sample firmly in the chuck to prevent inaccurate cuts and damage to the blade resulting from movement during sectioning.

8.4.1.6 Position the counterbalance weight after the chuck and the sample are mounted on the arm so that the arm is balanced.

8.4.1.7 Set the blade speed on 7 during the entire cut for a 5-in. blade and on 8.5 for a 4-in. blade.

8.4.1.8 The ratio of applied load to width of sample should be 225 to 300 g/in. of sample based on the maximum width of the piece being cut.

NOTE 3—Caution: Do not use more than 300-g weight on the arm as blade distortion may occur.

8.4.1.9 Do not turn the saw off during the cutting process, and keep the sample in contact with the blade. When approaching the end of a cut, support the piece being cut from the bulk material in order to prevent it from breaking off.

8.4.1.10 Discard the lubricant fluid after every 2 h (and after the cut has been completed if the cut takes longer than 2 h). Clean the pan and replace with fresh water.

8.4.1.11 On a new blade, the width of the diamond matrix at the rim is approximately ⅝-in. When this width becomes less than ⅓-in., the blade should be replaced.

8.4.2 *Specimen Cleaning*—Clean the specimens using the following procedure:

8.4.2.1 Subject specimens to 5-min ultrasonic wash in high-purity water or other appropriate fluid.

8.4.2.2 Subject specimens to three 5-min ultrasonic washes in fresh absolute ethanol or other appropriate fluid.

8.4.2.3 Dry to constant mass and record mass. Use a drying technique that has been demonstrated to be applicable to the specific waste form being tested. One hour at 110°C is sufficient for most nonporous waste forms. Porous waste forms may require higher temperatures and longer times.

8.4.2.4 When performing experiments using deaerated solutions, additionally wash the specimen under an argon atmosphere using deaerated high-purity water and store the specimen under argon until placing in the test vessel (see Note 1).

8.4.3 Calculate the geometric surface area from overall dimensions. Although a single specimen is preferred, up to four pieces may be included as one specimen if the S/V ratio as defined in 9.4.3 is achieved.

8.5 *Specimen Handling*—All handling of specimens after preparation and cleaning must be done with tongs, tweezers, or lint- and dust-free plastic or rubber gloves.

9. Procedure

9.1 *Quality Assurance Requirements*—This procedure must conform to all applicable quality assurance requirements of the laboratory performing the test.

9.2 The procedure for the individual tests, including those for blanks, is given in 9.4. The procedure consists of immersing test specimens in leachant solutions under conditions that vary depending on the purpose of the test. Test matrices designed for the different test purposes are defined in 9.5. Select at least one matrix from 9.5. The overall number of tests that must be run, including those for blanks, will depend on the test matrix selected.

9.3 When highly radioactive specimens are being tested in stainless steel or fused silica vessels, the specimens must be tested under deaerated conditions to minimize radiolysis of nitrogen. Such testing will provide a logical link between the testing done under nonradioactive conditions and the radioactive conditions. A test program may call for testing of both radioactive (using stainless steel or fused silica vessels) and nonradioactive (using PTFE vessels) specimens of a particular waste glass composition. If this type of program is undertaken, the test matrix should include additional tests of nonradioactive specimens that are conducted under the same conditions used for the radioactive samples (for example, stainless steel or fused silica vessel, deaerated leachant). Sufficient numbers of these additional tests should be performed to provide the link between the data from the radioactive and nonradioactive materials. (See Note 1.)

9.4 *Leaching an Individual Specimen*—This procedure also applies to blanks, except that the specimen is omitted. The volume of leachant used in the blanks should be approximately the average of the leachant volumes for which it serves as a blank. When using deaerated solutions, carry out the procedure

⁷ The Buehler Isomet Low Speed Saw with standard accessories, Buehler part number 11-1180 with Arbor Diamond Wafering Blades, low diamond concentration (200 grit), 5-in. diameter × 0.015-in. thickness, part number 11-4255, or 4-in. diameter × 0.012-in. thickness, part number 11-4254, Buehler dressing Sticks, part number 11-1190, and Buehler Isomet dressing chuck, part number 11-1196, available from Buehler, 41 Waukegan Rd., Lake Bluff, IL 60044, (or equivalent) are recommended.

in an argon atmosphere. Also follow precautions noted in 7.3.2 when leaching with brine under high-radiation fields (see Note 1).

9.4.1 Determine the pH of the leachant on an aliquot of the leachant and discard this aliquot.

9.4.2 If a deaerated leachant is used, determine the dissolved oxygen content of the leachant on an aliquot of the leachant and discard this aliquot (this oxygen determination will be compared to a similar measurement made subsequent to the leach test to determine whether excessive oxygen contamination has occurred during the course of the test).

9.4.3 Determine the volume of the leachant to be used based on the measured geometric surface area of the specimen. The S/V ratio must be within 0.5 of 10.0 m⁻¹.

9.4.4 Place the leachant into a clean test vessel. Support the specimen near the centroid of the leachant volume. When using a PTFE basket or mesh specimen support, avoid trapping bubbles in the mesh that could hinder the leaching of the specimen. Completely submerge the specimen in the leachant. Weigh the test vessel with contents; then within 30 min, place it into the preheated test oven in the zone where the temperature is within 1°C of the target test temperature (see 6.7).

9.4.5 The testing period starts when the test vessel is placed into the test oven. Record that date and time (d:h:min).

9.4.6 If the test vessel has a screw-top lid, retighten the leach-vessel lids at 1 h and 24 h after the start of the test to ensure a good seal. If PTFE vessel is used, the lid should be re-tightened daily until no further slack is present. This may take three or more days. At each of these time periods, swirl the solution in the vessel gently or tap gently, or both, to dislodge any bubbles. Do not change the placement of the samples, however. Thereafter, avoid movement of the test vessel that could disturb a surface film, if such a film forms on the test specimen.

9.4.7 Experience has shown that a slow water loss through the PTFE test vessel walls is inevitable (approximately 0.01 mL/day)⁸. To detect faulty sealing or an off-standard test vessel, check the mass of the test vessel and contents of a long-term test at least every three months. If the volume of leachant in the test vessel, as determined by weight measurement, becomes less than 90 % of the original volume, repeat the test starting at 9.4.1. The sample should be replaced at this juncture. If the water loss is greater than 5 % and less than 10 %, open the test vessel and add an amount of deionized water equal in mass to the measured loss from the test vessel. Water additions are limited to a total of 15 % per year, and no additions may be made any closer to the end of the test than three months. Make the addition in an argon atmosphere when using deaerated solutions (See Note 1). Do not cool the vessel and contents. Record the date and time (d:h:min) and the amount of water added. Return the vessel to the test oven.

9.4.8 Control the testing period to within 2 % of the target test durations in 9.5. Record the date and time (d:h:min) at which the test ended. At the conclusion of the testing period, remove the test vessel from the test oven and weigh the test

vessel with leachant and specimen to determine leachant loss. If the amount of leachant is less than 90 % of the original leachant, repeat the test starting at 9.4.1. If the volume is greater than 90 % of the original volume, remove the specimen from the hot leachate but leave any specimen support in place; replace the vessel lid immediately; and allow the leachate to cool.

9.4.9 Rinse the specimen in high-purity water for approximately 5 s. Dry the monolithic specimen to constant weight and record the weight. Weigh and analyze any material that sloughs off the surface during rinsing and handling and report the results with the leach test data.

9.4.10 Measure the pH of an aliquot of the cooled leachate. Discard the aliquot. If deaerated leachant has been used, this aliquot must remain under an argon atmosphere until after its pH has been measured.

9.4.11 Remove aliquots for any special analyses, such as for anions or colloids. If a deaerated leachant has been used, an aliquot should be removed and used for an oxygen content determination immediately and under an argon atmosphere. This measurement will be used to determine whether excessive oxygen contamination has occurred during the course of the test. If excessive contamination has occurred, the test must be repeated. Excessive oxygen contamination is arbitrarily defined as a leachate oxygen content that is greater than a factor of ten higher than the oxygen content of the initial leachant.

9.4.12 Inspect the leachate and vessel for residual solids either precipitated from solution or sloughed from the specimen and record observations. If solids are not present, proceed to 9.4.15 without filtering. If solids are present, filter the leachates through a clean membrane filter with pore size of 0.45 µm. Prepare filtering equipment by cleaning with three rinses of 6 M HNO₃, each equal to 20 % of the equipment volume, and three rinses of high purity water followed by three rinses of fresh leachant. Separated solids must be weighed, analyzed, and reported with the leach test data. At a minimum, the composition of the solids must be determined.

9.4.13 In some cases it may be desirable to determine the amount of sorption on the walls of the test vessel. If separate sorption determinations are to be made, skip 9.4.14 and proceed through 9.4.16 and 9.4.17 for most sorbed materials or 9.4.16-9.4.18 for sorbed elements that include actinides.

9.4.14 If sorption determinations are not made, return the clarified leachate to the test vessel, and proceed through only 9.4.15 and not 9.4.16-9.4.18.

9.4.15 If the amount of material sorbed on the walls of the test vessel is not to be a separate determination, add concentrated nitric acid (concentrated hydrochloric acid for brine leachates) equal to 1 % of the present leachate volume. Submit the acidified leachate for analysis as soon as possible after a 12 to 24-h digestion at 90°C. Submit the leachate in their original vessels, if practicable. Alternatively, transfer the leachate to a new polyethylene or polypropylene bottle that has been cleaned by rinsing with three volumes of 6 M HNO₃, each equal to 20 % of the bottle volume, and three rinses with high-purity water. Analyze the leachate and include determinations of waste components, such as fission-product elements. The analysis shall include a determination of F⁻ concentration

⁸ To eliminate or reduce water leakage, a PTFE vessel rated at 0.5 MPa can be used, such as that supplied by Savillex, Minnetonka, MN.

if the test has been conducted in a PTFE vessel. If an actinide-doped specimen was tested, proceed to 9.4.18 after completing 9.4.15.

9.4.16 If sorption measurements are to be done, prepare new polypropylene bottles that have been cleaned by rinsing with three volumes of 6 M HNO₃, each equal to 20 % of the bottle volume, and three rinses with high-purity water. Transfer the leachates from 9.4.12 to a new bottle. Add concentrated nitric acid (hydrochloric acid for brine leachates) equal to 1 % of the present leachate volume. Submit the acidified leachate for analysis as soon as possible. The analysis shall include a determination of F⁻ concentration if the test has been conducted in a PTFE vessel.

9.4.17 Most solids sorbed on the wall of the test vessel can be removed by either nitric or hydrochloric acid. First, however, assure that no residual leachate remains in the empty test vessel by rinsing the vessel with high-purity water. Then place an amount of 1 % nitric acid (1 % hydrochloric acid for brine leachates) equal to the volume of the original leachate in the empty test vessel. Allow the vessel to stand at least 12 h at 90°C and submit the acid strip solution for analysis. The analysis shall include a determination of F⁻ concentrations if the test has been conducted in a PTFE vessel.

9.4.18 Some actinide elements plate out on the walls of the test vessel. To remove these requires the use of an additional aggressive acid combination. If PTFE vessels are used, a 1 M HF-6 M HNO₃ acid combination equal to the volume of the original leachant should be used and the vessel allowed to stand for 1 to 2 h at 90°C. If 304L stainless steel or fused silica vessels are used, a 1 % HF-1 % HNO₃ strip solution should be used, and the vessel should be allowed to stand at least 12 h at 90°C. Check the walls of the vessel using the appropriate counting technique to determine the effectiveness of the acid treatment. Submit the plate-out strip solution for actinide analysis.

9.4.19 In certain cases, it may be useful to examine the post-leach test surface of leaching specimens. If this is to be done, it is recommended that this examination include SEM-EDX analysis of both the leached surface and a cross section of the leached surface (to estimate elemental depth profiles).

9.5 *Test Matrices*—The test method is divided into a series of matrices so that it can be used for different purposes. Matrix A comprises the simplest test matrix; each succeeding method includes additional test conditions, that is, becomes more comprehensive. To the degree possible, all waste form specimens and blanks from a test matrix should be placed in the same test oven. If several test matrices with one or more waste forms are started simultaneously and are used with the same batch of leachant, common blanks may be used for each leachant and specific time period. If 304L stainless steel test vessels are used, it is recommended that each test and blank be performed in triplicate (instead of, for example, only the 28-day, 91-day, and longer duration tests of Matrix D). This repetition is necessary because high chloride levels, which have a significant effect on leach test results, have occasionally been observed in tests conducted in stainless steel vessels. If such tests are conducted in triplicate, the researcher is reasonably assured of having at least one or two reliable data points

for each test condition. The matrices and their purposes are described in 9.5.1-9.5.5.

9.5.1 *Matrix A: 7 Days, 90°C*—This matrix is for waste-form-development screening tests in an individual laboratory.

9.5.1.1 Test temperature is 90°C.

9.5.1.2 Leach three separate specimens of each waste form for 7 days in water.

9.5.1.3 Prepare and test one blank for 7 days for each group of specimens tested simultaneously in the same environmental test chamber.

9.5.2 *Matrix B: 28 Days, 90°C*—This matrix can be used for an initial ranking of the leach resistance of waste forms.

9.5.2.1 Test temperature is 90°C.

9.5.2.2 The leachant is water, silicate water, or brine.

9.5.2.3 Use the following matrix giving the number of specimens:

Test period, days	Number of specimens
3	1
7	1
14	1
28	3

9.5.2.4 Prepare and test two blanks of each leachant for 28 days under identical test conditions but excluding a specimen.

9.5.3 *Matrix C: 28 Days, 40°, 70°, 90°C*—This matrix can be used for a preliminary determination of the effect of temperature on leaching.

9.5.3.1 The required test temperature is 90°C. Also use 40° or 70°C, or both.

9.5.3.2 The procedure is otherwise identical to Matrix B (9.5.2).

9.5.4 *Matrix D: Long-Term, 90°C*—This matrix is used to measure the kinetics of the approach to the steady-state, saturated boundary condition in a closed leaching system at the reference temperature of 90°C.

9.5.4.1 The test temperature is 90°C.

9.5.4.2 The leachant is water, silicate water, or brine.

9.5.4.3 Use all or selected portions of the following matrix. (“Selected portions” means, for instance, that only one leachant may be used.) Also, the test may be terminated at 56 days or at any longer duration shown in the matrix. Suitable blanks must be carried through to the end of the test.

Test period, days	Number of specimens
7	1
14	1
28	1
56	3
91	1
182	3
364	1
Optional 12-month intervals	3

9.5.4.4 Prepare and test two blanks at 28, 91, and 364 days (and yearly thereafter to the culmination of the test when it extends beyond 364 days) in each leachant under identical test conditions but excluding a specimen.

9.5.5 *Matrix E: Long Term, 40°, 70°, 90°C*—This matrix is used to determine the effect of temperature on the kinetics of the approach to the steady-state, saturated boundary condition.

9.5.5.1 The required test temperature is 90°C. Also use 40° or 70°C, or both.

9.5.5.2 Matrix E is otherwise identical to Matrix D (9.5.4).

10. Calculation

10.1 *Use of Blanks*—The blank data is used to correct the mass leached from a specimen by subtracting blank concentration. In cases where blank data are available for the same test condition (temperature, time, leachant), correct the leachate concentration by subtracting the blank for the same test conditions. For cases where a blank was not obtained under the same test duration, the correction can be made by using blank data that bracket or are close to the test duration, for example, shorter times or longer times. Do not use blank data for different leachates or different temperatures to correct leachate concentration.

10.1.1 When tests are conducted according to the various matrices in 9.5, the following blank corrections are recommended. In Matrices B through E, use the original leachant analyses as blank for 3, 7, and 14-day data. In Matrices D and E, use 28-day blanks for 56-day data and 91-day blanks for 182-day data.

10.1.2 The blank serves as a control. If a large change in the blank concentration is observed, an out-of-control condition may exist, which could invalidate the data. The investigator must report all individual leachate concentrations and individual blank concentrations.

10.2 *Mass Leached*—Calculate the mass of element i , m_i , leached, using the following equation:

$$m_i = (C_{ij} - B_i) \cdot V_j \quad (1)$$

where:

C_{ij} = concentration of element i observed in 0.45- μm filtered leachate from specimen j , averaged over replicate aliquots,

B_i = average concentration of element i observed in 0.45- μm filtered leachate from blanks, averaged over replicate aliquots and replicate blanks, and

V_j = initial volume of leachate in test vessel containing specimen j .

10.2.1 An optional method for calculating m_i is given in 10.2.2. Use this method when a correction for possible volume loss of leachate is desired. Because the allowable volume loss is 10 % or less, the correction, using the method in 10.2.1, will be 10 % or less in most cases. Normally, a correction for volume loss will be unnecessary. However, in cases where the blank concentration is greater than approximately 45 % of the leachate concentration, this correction can be greater than 10 %. When the blank concentration is large relative to the leachate concentration, that is, greater than 45 %, the investigator should examine the data further to determine whether the volume losses of the leachate and blank are large enough to warrant a correction for volume loss. Always report the method used to correct for volume loss if a correction is performed.

10.2.2 *High Precision Procedure for Correction of Mass Leached Taking into Account Volume Losses of Leachate and Blank Solutions (Optional)*—Calculate the mass of element i , m_i , leached, using the following equation:

$$m_{ij} = C_{ij} \cdot (FV)_j - \sum_{k=1}^a \sum_{l=1}^b (FVB)_k \cdot B_{ikl} / a \cdot b \quad (2)$$

where:

C_{ij} = concentration of element i observed in 0.45- μm filtered leachate from specimen j , averaged over replicate aliquots,

$(FV)_j$ = final volume of leachate in test vessel containing specimen j ,

B_{ikl} = average concentration of element i observed in 0.45- μm filtered leachate from blanks, averaged over replicate aliquots and replicate blanks from the l th aliquot of the k th blank,

$(FVB)_k$ = final volume of solution in k th blank vessel,

a = number of blank vessels, usually two, and

b = number of aliquots for each blank vessel, usually two.

10.3 *Normalized Elemental Mass Loss*—Calculate the normalized elemental mass loss, $(NL)_i$, in g/m^2 removed from the specimen, using the following equation:

$$(NL)_i = m / (f_i \cdot SA) \quad (3)$$

where:

m_i = mass of element i in the 0.45- μm filtered leachate (g) (calculated as shown above),

f_i = mass fraction of element i in the unleached specimen, and

SA = specimen surface area, m^2 .

10.3.1 When using radioactive specimens, use the following equation:

$$(NL) = a_i / a_o \cdot W_o \cdot 1/SA \quad (4)$$

where:

a_i = total activity of isotope i in the 0.45- μm filtered leachate,

a_o = the original total activity of the isotope in the specimen, and

W_o = original mass of the specimen, g.

10.4 *Molarity*—Since the test is conducted in a closed system, the concentration of some constituents in the leachate will become saturation limited with time. This behavior is sometimes easily followed by expressing the results of the test in terms of molarity of the constituents in the leachate. Calculate the molarity, M_i (mol/L), of the constituents removed from the specimen using the following equation:

$$M_i = m_i / (A_i \cdot V) \quad (5)$$

where:

A_i = atomic weight of element i , and

V = volume of leachate, L.

10.5 *Calculation for Tests Using Stainless Steel or Fused Silica Test Vessels*—When stainless steel or fused silica test vessels are used, the leachate will contain elements leached from the vessel. Leach data for elements that are components of the vessel (Si for fused silica vessels and Fe, Cr, and Ni for 304L stainless steel vessels) should be reported in tables and plots that are separate from those used to report data for elements that are only present in the glass waste form. These data should be clearly identified as being potentially biased by the presence of elements leached from the vessel.

11. Report

11.1 Report the following information:

11.1.1 Material tested and its identification number; test number (for example, ASTM C 1220, Matrix B); name of investigator; affiliation of investigator; and date report submitted.

11.1.2 *Test Conditions, Specimen Preparation, and Description*—Material tested, preparation, and composition including the following:

11.1.2.1 Detailed sample preparation with starting materials, manufacturing technique, and size, shape, and thermal history of the prepared sample, and unique material identification number;

11.1.2.2 *Chemical Composition*—Intended and as analyzed, and, if available, with quantitative information on analytical errors, and errors associated with non-homogeneity of specimens;

11.1.2.3 Microstructural examination with a description and short discussion of results from all solid-state techniques used;

11.1.2.4 Test equipment including test oven-type, model number, etc;

11.1.2.5 Temperature distribution and monitoring, including definition of zone in which target temperature is maintained (include sketch if necessary). Describe monitoring technique, equipment used, equipment model number, etc;

11.1.2.6 *Test Vessel*—Model number and manufacturer; method of supporting specimen in test vessel;

11.1.2.7 *Balances*—Model number and manufacturer;

11.1.2.8 Analytical techniques, including pH meter and probe (model numbers and manufacturers), solution and isotope analyses; brief description of technique and equipment used;

11.1.2.9 Other determinations, if any, for example, dissolved oxygen, bacteria, etc;

11.1.2.10 Calibrations and reference materials including list of equipment and method of calibration;

11.1.2.11 Reference simulated leachate with source, identification number, and analysis traceable to a certified standard;

11.1.2.12 Leachant preparation, including chemical list; description of equipment used to prepare deionized water with manufacturer and model number listed; identification of manufacturer, catalog number (and lot number, if available) of leachant makeup chemicals with a list of major impurities in makeup chemicals; and

11.1.2.13 Specimen preparation and description, including sampling procedure (use sketch if necessary to show location of test specimens in original large sample); description of equipment used to prepare specimens with manufacturer and model number of saw or core drill and saw blade or core drill bit and with grit size of saw blade or core drill bit listed; date of preparation and storage conditions; description of technique for measuring surface area; tabulation of the identification number, weight, dimensions, calculated surface areas, and corresponding volume of leachate used for each specimen.

11.1.3 *Leachant Analysis*—Include the following information: identification number of each leachant batch; date of makeup, date of analysis, and date leach testing began; and tabulation of analytical results for all elements, isotopes, ions, and organics analyzed in the leachants. The detection limits for each analysis must be given in parenthesis when that result is

less than a factor of 10 greater than the detection limit. When the result is below detection limits, it may be left blank or reported as not detected (ND), with detection limit in parenthesis. If the leachant is other than the reference leachants, report the chemical and isotopic composition, chemicals and isotopes used in preparation, date of preparation, and date of analysis. If the leachant is a natural aqueous solution, report the date, place, and stratigraphy from which it was obtained. Report the chemical composition, Eh (if measured), pH, and date of analysis.

11.1.4 *Leachate and Blank Analyses*—Raw data tabulation including specimen and blank identification; date and time of start and end of runs; starting and ending mass of leachants and percent mass loss; general observations with presence of solids, solution discoloration, etc. described (if solids are present, include method of removal, weight and results of analyses of solids); final pH leachates and blanks; final leachate and blank volumes; temperature of test; final weight and weight loss of specimen, where applicable; and tabulation of analytical results for all elements, isotopes, ions, and organics analyzed in leachates, acid strips, and blanks. Also report detection limits for the analyses, as described under leachant analyses above; treated data tabulation, including blank corrected and averaged leachate analyses (where applicable); and identify leached material, leachant, time, and temperature.

11.1.5 *Tabulated and Plotted Data*:

11.1.5.1 Normalized elemental mass losses, including: (1) tabulation and plot of normalized elemental mass losses versus time in days (include actual mass loss where applicable); (2) identify leached material, leachant, and temperature of analysis; (3) if the leachant is a natural aqueous solution, report the date, place, and stratigraphy from which it was obtained; (4) report the chemical composition, Eh (if measured), pH, and date of analysis; include error bars in the plots for the triplicated data points that extend one standard deviation above and below the average value. (The standard deviation of the normalized mass-loss values is computed by a propagation of errors method to include the uncertainty in both the concentration values and the mass fraction value f_i);

11.1.5.2 molarity, including the following information: (1) tabulation and plot of the molarity of all elements reported in the raw data tabulation versus time in days; (2) identification of leached material, leachant, and temperature; (3) error bars in the plots for the triplicated data points that extend one standard deviation above and below the average value.

11.1.6 *Post-Leach Specimen Analyses (Optional)*—Results and discussion of surface analyses such as SEM-EDX. In particular, discuss how surface analyses correlate with leachate analyses and the implications concerning leaching mechanisms. While pre-test SEM-EDX profiles are not required as part of the method, they would be useful for comparison.

11.1.7 *Precision and Bias*—Preparation of control chart, including establishment of the analytical capability of the laboratory for each element that is to be used for calculating normalized elemental mass losses; tabulation of data and averages of pairs run in the 28-day preliminary analysis of the reference leachate for each element that is to be used for calculating normalized elemental mass losses; estimation of the

within and between day components of variance and the control-chart limits and relative bias estimate for the data; example control charts that indicate upper and lower control limits for selected elements; reference simulated leachate data including tabulation and plot of the reference simulated leachate data obtained during the analysis of the test-matrix leachates on the control chart; reporting of any systematic pattern in the control chart that may indicate an out-of-control situation or cross-contamination; laboratory-measurement-control information, including maintenance of a file containing procedures used and data resulting from calibrations and laboratory quality-control practices for those measurement methods involved in gathering data for the test matrix; reporting the calibration procedure and frequency of calibration for the chemical analysis methods used; summary statement on data quality, including giving a concluding estimate of the overall precision and accuracy of the reported pH's and normalized elemental mass losses; factors such as sample inhomogeneity, variance in leachate analyses, and the author's previous experience with the test, which should be considered in arriving at the summary estimate.

11.1.8 Discussion, including deviations from test procedure; inadvertent happenings, for example, power outages, power fluctuations, etc. (for longer test, in particular); and significance of results.

12. Precision and Bias

12.1 When triplicate specimens with blanks are run for a specified time in one leachant, the following solutions will be available for analysis (leachates from single-specimen data points must be analyzed as soon as possible after 9.4.11):

Code	Solution Type	Number of Solutions per Leachant Used
B	Blank leachates	2
WF	Leachate from waste-form test specimens	3

12.2 In addition, samples of reference simulated leachate, SL, must accompany submittal of the solutions to the analytical laboratory. The solutions will be shaken, sampled, and analyzed in the following order: there will be three analyses of WF, then two analyses of the SL, then two analyses of B, for a total of seven analyses per leachant type for each applicable time period.

12.3 *Use of Reference Simulated Leachate*—The simulated leachate (SL) will be used to estimate the bias and within-laboratory precision of the analytical method used. The requirements for an SL are (1) that it contains selected elements that will be present in actual leachates, (2) that these elements be present in concentrations similar to those expected in the leachates, and (3) that the SL be chemically stable over the time period for which it will be used. Stability of the SL is more important than accuracy of composition. This is because the SL is not used to calibrate the analytical equipment. Instead it is submitted for analysis as an independent check of the analytical technique(s) used for analyzing leachate samples. SL analyses are used to determine whether the results obtained from the analyses of identical samples are constant, within analytical uncertainty. Data from the analyses of the SL will also be used in control charts to provide criteria for control of

bias and precision (see ASTM Manual 7⁹ for guidance concerning the use of control charts).

12.3.1 To establish the analytical capability of the laboratory, two aliquots will be drawn from the SL every other day over a 28-day period and analyzed separately. The results will be used to develop a control chart for each element that is to be used for calculating normalized elemental mass loss. During analysis of leachates from the test matrix, the analyses for each leachate type will be bracketed by analyses of SL. Instructions for preparation of control charts and details of the statistical analysis to be used will be included with the instructions for preparation of the SL.

12.4 *Waste-Form Replicates*—The three WF replicates for 28 days (in Matrix B, also at longer times in Matrices D and E) will be used to provide estimates of laboratory experimental procedure variability. Averages and standard deviation and relative standard deviation of normalized elemental mass loss and molarity will be tabulated.

12.5 *Laboratory-Measurement-Control Information*—Although a consideration burden of measurement-control procedures and analyses are built into the test method in the form of calibration requirements, chemicals used, and SL and B analyses, the degree of replication of specimens and analyses is minimal for demonstrating statistical control. In the case of marginal results, as demonstrated by the control criteria of 12.1, more detailed information will be required on the quality-control methods and resulting data used by the laboratory in question. The types of information that should be available at the laboratory would include procedures and recent data for the following:

12.5.1 Calibration of chemical and isotope analysis methods,

12.5.2 Calibration of weighing, volume, and surface-area measurement instruments, and

12.5.3 Routine blind specimens or other quality-control methods.

12.6 *Discussion of Deviations*—Discuss any deviations from the procedure and their expected effect on the results.

12.7 *Precision and Bias Estimates from Round-Robin Data:*

12.7.1 The within laboratory precision achievable by a single laboratory applying the static leach test method for 28-day tests is well documented in data analyzed by the Materials Characterization Center (MCC) at Pacific Northwest Laboratory. Also, the MCC conducted an extensive interlaboratory round robin test to estimate both the between laboratory and within laboratory, single operator precision that can be expected when the static leach test method is applied using any of several glasses, leachant solutions, and temperatures. The results of this study are reported by Johnston and Daniel (see Appendix X1).¹⁰ By comparison with results obtained since this round robin, it has been concluded that experience in applying the test method (or similar test methods) has a significant effect on both the between laboratory and within

⁹ ASTM Manual 7, *Presentation of Data and Control Chart Analysis*, 6th ed., ASTM International, 1990.

¹⁰ Johnston, J. W., and Daniel, J. L., Summary Report for the Interlaboratory Round-Robin on the MCC-1 Static Leach Test Method, PNL-4249, Materials Characterization Center, Pacific Northwest Laboratory, Richland, WA, 1982.

TABLE 2 Comparison of NIST and MCC Test Results

	NIST		MCC		% Relative Difference ^A	95 % Confidence Interval for % Relative Difference	$\frac{s(MCC)}{s(NIST)}$
	X	s	X	s			
B	1.3714	0.0255	1.2830	0.0303	-6.4	[-8.8,-4.1]	1.19
Ba	0.0077	0.0003	0.0072	0.0003	-6.5	[-11.1,-1.9]	1.37
Ca	0.1384	0.0094	0.1338	0.0058	-3.3	[-10.0, 3.3]	0.62
Cs	0.4311	0.0081	0.4135	0.0103	-4.1	[-6.6,-1.6]	1.27
Mo	0.5231	0.0093	0.5089	0.0107	-2.7	[-5.0,-0.5]	1.15
Na	4.4486	0.1697	4.3489	0.1776	-2.2	[-6.8, 2.3]	1.05
Si	5.4600	0.1017	5.7165	0.1453	4.7	[2.0,7.4]	1.43
Sr	0.0310	0.0010	0.0311	0.0010	0.3	[-3.5,4.1]	1.01
Zn	0.0245	0.0317	0.0317	0.0036	29.4	[9.8,48.9]	0.80

^A $(-X_{NIST} + X_{MCC}/X_{NIST}) \cdot 100$

laboratory precision. The data from inexperienced laboratories that participated in this round robin is believed by the MCC to have resulted in over-estimation of the between and within laboratory standard deviations.

12.7.2 A possibly more representative data set was obtained from the results of an interlaboratory round robin test initiated and supported by the Commission of European Communities (CEC), where the MCC-2 static leach test method was used. A simulated high-level radioactive waste glass, designated UK-209, was tested in deionized water at temperatures of 90, 110, 150, and 190°C for durations of 7, 14, and 28 days. Since at 90°C the MCC-2 test and this static leach test are the same, results from the CEC round robin are used to illustrate the expected precision of this static leach test method (see Appendix X1).

12.7.3 *Bias*—At this time, no certified test values are available to accommodate estimation of biases that might result from the test method itself or from the manner in which the individual steps of the test method are performed by a particular laboratory.

12.7.4 A comparative study was conducted, where the MCC applied the static leach test method and the NIST applied the same test method but with tighter laboratory controls and more extensive specimen preparation than are required by the test method. The objective was to determine whether the extra control and specimen preparation would significantly affect the test results or improve precision. The tests were run for 28 days at 90°C in deionized water. Each laboratory used seven replicate test specimens of a simulated waste glass provided by the NIST. The results are summarized in Table 2.

12.7.5 Each 95 % confidence interval, for the difference between unknown true means for the MCC and the NIST, is expressed as a percent of the NIST sample mean. Confidence intervals that do not include zero indicate that the difference

between the sample means is statistically significant, which implies that the unknown true difference is probably not zero. Six of the nine elements have confidence intervals that do not include zero. The largest percent relative difference is 29.4 % for Zn, and the confidence interval indicates that the true difference is probably between 10 % and 49 % of the NIST sample mean. For the remaining elements, the percent relative differences range from -6.5 to 4.7, and the confidence intervals indicate that the true differences probably range from -11.3 % to 7.4 %. When compared with the between-laboratories percent RSD from the round robin (see Appendix X1), differences in this range are not alarming.

12.7.6 The ratio of the sample standard deviations ranges from 0.62 to 1.43. In order to be statistically significant at the 0.05 level, a ratio would have to be less than 0.41 or greater than 2.4. Thus, there are no significant differences in the level of precision achieved by the two laboratories.

12.7.7 The precision of the test for test durations longer than 28 days has not been established. In particular, the possible effects of F⁻ release and pH increases that might result from variations in PTFE properties should be considered. The test user for test durations greater than 28 days shall particularly note the F⁻ concentration in blanks (a required measurement for the test method) and the pH of the blank after testing. In particular, when comparing results from different manufacturing lots of PTFE, these pH data should be compared and found within 0.5 pH units in order to make a straightforward comparison of leach results for the waste forms.

13. Keywords

13.1 ceramic leaching; cement leaching; glass leaching; leach test; nuclear waste; nuclear waste forms; nuclear waste materials; radioactive waste; simulated waste forms; static leach test; waste form durability leachate; waste forms

APPENDIX
(Nonmandatory Information)
X1. ADDITIONAL DATA ON THE PRECISION OF THE STATIC LEACH METHOD FROM ROUND-ROBIN DATA

X1.1 Because no authoritative reference values are available at present, it is not possible to determine whether application of the test method produces biased estimates of the true normalized elemental mass losses that would occur under the prescribed conditions of the test method. That is, it is not possible to determine whether the test method is inherently biased.

X1.2 The within-laboratory precision achievable by a single laboratory applying the test method is well documented in the MCC-D1, MCC-D2, and MCC-D5 data packages. Also, the MCC conducted an extensive inter-laboratory round-robin test to estimate both the between-laboratory and within-laboratory precision that can be expected when the test method is applied by almost any laboratory, using any of several glasses, leachant solutions, and temperatures and for a test period of 28 days. The results of this study are reported by Johnston and Daniel.¹⁰

X1.3 The Commission of European Communities (CEC) initiated and supported an inter-laboratory round-robin test with the MCC-2 test method.¹¹ Twelve laboratories from nine different countries participated. All twelve laboratories had previous experience in applying the method or similar test methods. A simulated high-level radioactive waste glass, designated UK-209, was tested in deionized water at temperatures of 90°, 110°, 150°, and 190°C for durations of 7, 14, and 28 days. Because at 90°C the MCC-2 and this test method are the same, the 90°C results from the CEC round-robin test are used

to illustrate the between-laboratory and within-laboratory precision that can be expected when the test method is used by a laboratory with experience in applying similar test methods.

X1.4 For comparison purposes, Table X1.1 displays a subset of the results from the MCC-D5 data package, the MCC round-robin study, and the CEC round-robin study. In each study, the blank-corrected concentrations were converted to normalized elemental mass losses (NL_i in g/m^2) that incorporate leachant volume loss, actual S/V ratio, and mass fractions computed from bulk analyses of the glass used in each study. The results displayed in Table X1.1 are for normalized elemental mass loss of B, Cs, Si, and Sr observed in the leachate from the tests performed for 28 days at 90°C in deionized water.

X1.5 The results from the MCC round-robin test and the CEC round-robin test were separately analyzed by application of analysis of variance methods to obtain estimates of the within-laboratory precision, between-laboratory precision, and total precision. Also, estimates of the within-laboratory precision for the MCC-D5 results were computed by analysis of variance methods. For each study, the within-laboratory precision is a measure of the ability (or inability) of a single laboratory to reproduce its own results with replicate specimens when applying the test method. The between-laboratories precision (relative standard deviation) is a measure of the variation in results among the participating laboratories in addition to the within-laboratory precision. The estimated total precision (relative standard deviation) includes both the within- and the between-laboratories components; thus, it provides an indication of the ability (or inability) of one laboratory to reproduce the results of another when they both apply the test method under prescribed similar test conditions.

¹¹ Konnecke, J. R., and Kirsch, J., EC Static High Temperature Leach Test Summary Report of a European Community Interlaboratory Round-Robin, BF-R-66.006-1, Battelle-Institute e.V. Frankfurt am Main, West Germany, 1985.

TABLE X1.1 Estimated Averages and Percent Relative Standard Deviations (% RSD) of Normalized Elemental Mass Loss from Tests Performed for 28 Days at 90°C in Deionized Water

Element	Concentration Range, ppm	Average NL g/m^2	Within Single Run and Laboratory % RSD	Between Laboratory % RSD	% RSD Total
MCC-D5 ^A					
B	4.1 to 4.5	12.2	3 ^B
Cs	0.73 to 1.1	8.23	3 ^B
Si	19.1 to 21.3	9.33	2 ^B
Sr	0.206 to 2.214	5.40	2 ^B
MCC Round-Robin Test ^C					
B	7.5 to 19.0	36.05	8	31	32
Cs	0.56 to 4.5	27.75	5	43	43
Si	28.7 to 91.0	23.91	7	34	34
Sr	0.025 to 0.137	1.85	16	60	62
CEC Round-Robin Test ^D					
B	2.24 to 4.9	10.52	4	19	19
Cs	0.44 to 1.2	9.54	11	31	33
Si	14.6 to 30.0	9.54	4	19	19
Sr	0.056 to 0.200	5.37	4	40	40

^A ARM-1 generic borosilicate waste glass, 1 lab/2 runs/3 tests each run.

^B Pooled estimate from two runs of test.

^C PNL-76-68 simulated high-level borosilicate waste glass, 25 labs/2 tests each lab.

^D UK-209 simulated high-level borosilicate glass, 7 labs/2 tests each lab.

X1.6 For each study and each element, Table X1.1 displays the range of concentration values (in ppm) observed in the study and the average normalized elemental mass loss in g/m^2 . Also displayed are the within-laboratory, between-laboratories, and total standard deviations expressed as a percent of the average and denoted by percent relative standard deviations (% RSD).

X1.7 Comparison of the MCC round-robin results and the CEC round-robin results in Table X1.1 supports the MCC

position that experience in applying this test method (or similar test methods) results in a definite improvement in both the between-laboratory and within-laboratories precision. Specifically, the % RSD estimates are smaller for the CEC round-robin test and the MCC-D5 results than for the MCC round-robin test. The exception is the within-laboratory % RSD for Cs, possibly because Cs is at lower concentrations in the CEC round-robin and MCC-D5 results than in the MCC round-robin results. Note, however, that the CEC within-laboratory % RSD estimates are comparable to those from the MCC-D5 results.

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