



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

This standard is issued under the fixed designation C1220; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a measure of the chemical durability of a simulated or radioactive monolithic waste form, such as a glass, ceramic, cement (grout), or cermet, in a test solution at temperatures $<100^{\circ}\text{C}$ under low specimen surface-area-to-leachant volume (S/V) ratio conditions.

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

1.3 The test is conducted under static conditions in a constant solution volume and at a constant temperature. The reactivity of the test specimen is determined from the amounts of components released and accumulated in the solution over the test duration. A wide range of test conditions can be used to study material behavior, including various leachant composition, specimen surface area-to-leachant volume ratios, temperatures, and test durations.

1.4 Three leachant compositions and four reference test matrices of test conditions are recommended to characterize materials behavior and facilitate interlaboratory comparisons of tests results.

1.5 Specimen surfaces may become altered during this test. Although not part of the test method, it is recommended that these altered surface regions be examined to characterize chemical and physical changes due to the reaction of waste forms during static exposure to solutions.

1.6 This test method is not recommended for evaluating metallic materials, the degradation of which includes oxidation reactions that are not controlled by this test method.

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

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

1.9 *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

C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy

C1174 Practice for Prediction of the Long-Term Behavior of Materials, Including Waste Forms, Used in Engineered Barrier Systems (EBS) for Geological Disposal of High-Level Radioactive Waste

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

2.2 EPA Document:³

SW-846A Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

3. Terminology

3.1 Definitions:

3.1.1 *accumulated dose, n*—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, n*—the closeness of agreement between an individual result and an accepted reference value.

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.

Current edition approved June 1, 2010. Published July 2010. Originally approved in 1992. Last previous edition approved in 2004 as C1220 – 98 (2004). DOI: 10.1520/C1220-10.

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.

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

3.1.4 *bias of a measurement process, n*—a consistent or systematic difference between a set of test results obtained from the process when measuring a property and the accepted reference value of the property being measured.

3.1.5 *chemical durability, n—in leach tests*, the resistance of a material to alteration, dissolution and release of its constituents, under the specific conditions of the test.

3.1.6 *closed system, n*—a system utilizing a test container that is impervious to material transport.

3.1.7 *dissolution, n*—the result of reactions in which chemical bonds are broken and species are released from a solid material and become solvated in the test solution.

3.1.8 *high-purity water, n*—purified water conforming with the requirements given in Specification **D1193** for Type I or Type II water.

3.1.9 *leachant, n—in leach tests*, general term for the initial solution with which a solid is contacted and into which the solid dissolves or is leached.

3.1.10 *leachate, n—in leach tests*, general term for the solution resulting from a test in which a solid is contacted by a solution and leaches or dissolves.

3.1.11 *leaching, v*—the preferential loss of soluble components from a solid material into a solution leaving a residual phase that is depleted in those components, but structurally unchanged.

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

3.1.13 *nuclear waste form, n*—solid material in which radioactive wastes have been immobilized.

3.1.14 *open system, n*—a system utilizing a test container through which material transport is possible, for example, O₂ or CO₂ diffusion, or both.

3.1.15 *precision of a measurement process, n*—the expected dispersion of values obtained using a measurement process under prescribed conditions, usually represented as a standard deviation or relative standard deviation.

3.2 Abbreviations:

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

3.2.2 *ISE*—ion selective electrode.

3.2.3 *PTFE*—polytetrafluoroethylene.

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

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

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

4. Summary of Test Method

4.1 A specimen of known geometric surface area (S) is immersed in a known volume of leachant (V) in a test vessel

that is sealed and placed in an oven (or other controlled-temperature device) set at a defined temperature for a defined time period without agitation. After the prescribed time interval, the vessel is removed from the oven and allowed to cool before being opened. Aliquants of the leachate solution are removed and analyzed for pH and various dissolved and colloidal components that were released from the specimen during the test. The concentrations of dissolved soluble components are used to determine the extent of reaction. A separate test is conducted to provide data for each test condition (duration, temperature, S/V ratio, leachant composition, etc.). Although it is not a part of the test method, it is recommended that the reacted test specimens be examined for changes in the composition and structures of the near-surface regions for correlations with the solution results and to study the reaction mechanism.

5. Significance and Use

5.1 This test method can be used to provide a measure of the reactivity of a material in a dilute solution in which the test response is dominated by the dissolution or leaching of the test specimen. It can be used to compare the dissolution or leaching behaviors of candidate radioactive waste forms and to study the reactions during static exposure to dilute solutions in which solution feed-back effects can be maintained negligible, depending on the test conditions.

5.2 The test is suitable for application to natural minerals, simulated waste form materials, and radioactive waste form material specimens.

5.3 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 **C1174**. In particular, measured solution concentrations and characterizations of altered surfaces may be used in the validation of geochemical modeling codes.

5.4 This test method excludes the use of crushed or powdered specimens and organic materials.

5.5 Several reference test parameter values and reference leachant solutions are specified to facilitate the comparison of results of tests conducted with different materials and at different laboratories. However, other test parameter values and leachant solution compositions can be used to characterize the specimen reactivity.

5.5.1 Tests can be conducted with different leachant compositions to simulate groundwaters, buffer the leachate pH as the specimen dissolves, or measure the common ion effect of particular solutes.

5.5.2 Tests can be conducted to measure the effects of various test parameter values on the specimen response, including time, temperature, and S/V ratio. Tests conducted for different durations and at various temperatures provide insight into the reaction kinetics. Tests conducted at different S/V ratio provide insight into chemical affinity (solution feed-back effects) and the approach to saturation.

5.6 Either aerated or deaerated solutions may be used in this test method except when testing highly radioactive specimens. Deaerated solutions are mandatory in tests conducted with

highly radioactive specimens to minimize the effects of nitrogen radiolysis. Preparation of deaerated leachants is addressed in 7.2.2.

5.7 Control of the oxygen fugacity is not part of this test method. Such control and measurement may be required for specific uses of test data but are beyond the scope of this test method.

5.8 Tests can be conducted using vessels compatible with the test specimen, leachant, and test environment. Corrosion resistant materials shall be used for tests with corrosive brines. Radiation-resistant materials shall be used for tests in radiation fields wherein the accumulated absorbed dose will exceed 10^4 rad (see Note 1).

NOTE 1—Additional requirements to the test method apply when using a highly radioactive waste form specimen, as indicated in the procedure.

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 leachant volume hanging from a PTFE Teflon monofilament attached to the vessel lid or set on a coarsely woven support screen.

6.2 *Oven*—The test oven must be capable of controlling the temperature of the test vessels to within 1°C of the test temperature.

6.2.1 When radioactive specimens are used, take into account self-heating when selecting the oven temperature to achieve the desired leaching temperature. Identify zones within the chamber where vessels can be located that are constant within 1°C of the target temperature using at least ten points of temperature measurement.

6.2.2 A temperature recorder or other monitoring device must be provided to ensure that the desired temperature is maintained for the duration of the test. Brief fluctuations from the desired temperature (for example, 5 min.) are allowed when specimens are placed in or removed from the test oven, when

thermocouples are checked, etc. The cumulative time that the oven temperature fluctuates more than 1°C from the target temperature must be kept to a minimum. The thermal mass of the vessel and leachate are expected to moderate the variance in the specimen temperature, but the oven should remain closed as much as possible.

6.2.3 The locations of test vessels should be mapped to facilitate their retrieval when the tests are terminated. Placements should minimize the need to disturb neighboring vessels when retrieving vessels.

6.3 *Test Vessel and Specimen Support*—Steel, titanium, fused silica, or polytetrafluoroethylene (PTFE) Teflon vessels and specimen supports (Fig. 2) can be used. Vessels shall be selected to be compatible with the test specimen material, leachant, and the radiation field.

6.3.1 When testing is performed in radiation fields expected to yield an absorbed dose of less than 10^4 rad, PTFE vessels shall be qualified for use (see 6.4). PTFE vessels shall not be used if the integrated dose to any PTFE component from all radiation (alpha, beta, or gamma) is predicted to exceed 10^4 rad (100 Gy). Doses below 10^4 rad have been shown to not damage PTFE.⁴ The total absorbed dose of each PTFE test vessel may not exceed 10^4 rad (100 Gy) during the lifetime of the vessel. For this reason, a record of the absorbed dose received must be maintained for every vessel that is reused. The use of PTFE test vessels may result in the release of F^- from the vessel to the solution. The primary reason for limiting the integrated dose to PTFE vessels and specimen supports to 10^4 rad and requiring that the PTFE vessels be qualified for use is to ensure that excessive fluoride releases do not occur. For PTFE vessels that meet the qualification requirements of this test method (see 6.4), the amount of release at radiation levels $<10^4$ rad have not been demonstrated to have an effect on leaching behavior.⁴ Nevertheless, analysis for F^- concentration is a requirement for all tests in which PTFE vessels or components are used. Teflon vessels are pervious to carbon dioxide, which could affect the solution pH, and some water loss may occur. The use of Teflon vessels is not recommended for test durations beyond 91 days.

6.3.2 If the integrated dose to the test vessel and specimen support is expected to exceed 10^4 rad, Type 304L stainless steel or fused silica vessels and specimen supports can be used except when brine leachants are used. Fused silica vessels and components must be used in tests with highly radioactive waste forms in brine leachants because of the corrosion of stainless steel by the brine. Stainless steel and fused silica vessels are impervious to carbon dioxide and water loss is usually negligible.

6.3.3 Vessels made of the same material shall be used throughout a test matrix to allow interactions between the vessel and the leachate to be evaluated and taken into account, for example, the release of silicon from fused silica.

6.3.4 The vessels must have sufficient volume to accommodate the leachant, specimen, and specimen support. Test vessel volumes will generally be between 20 mL and 1 L. The vessels

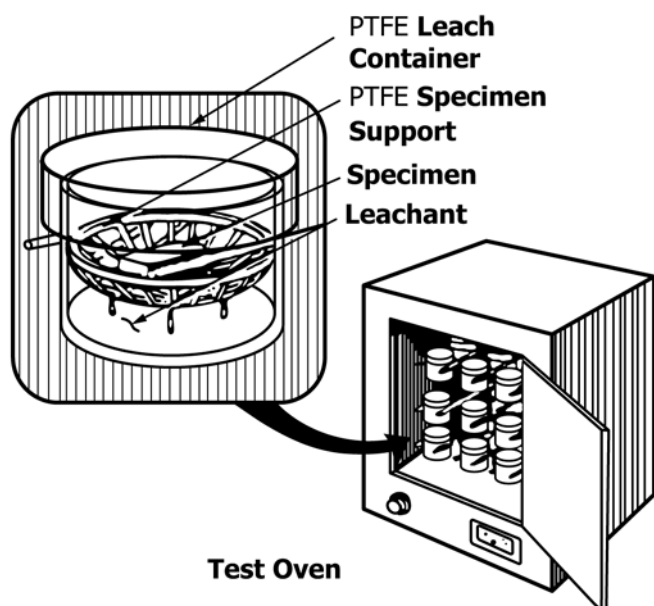


FIG. 1 Example Apparatus for Static Leach Test Method

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

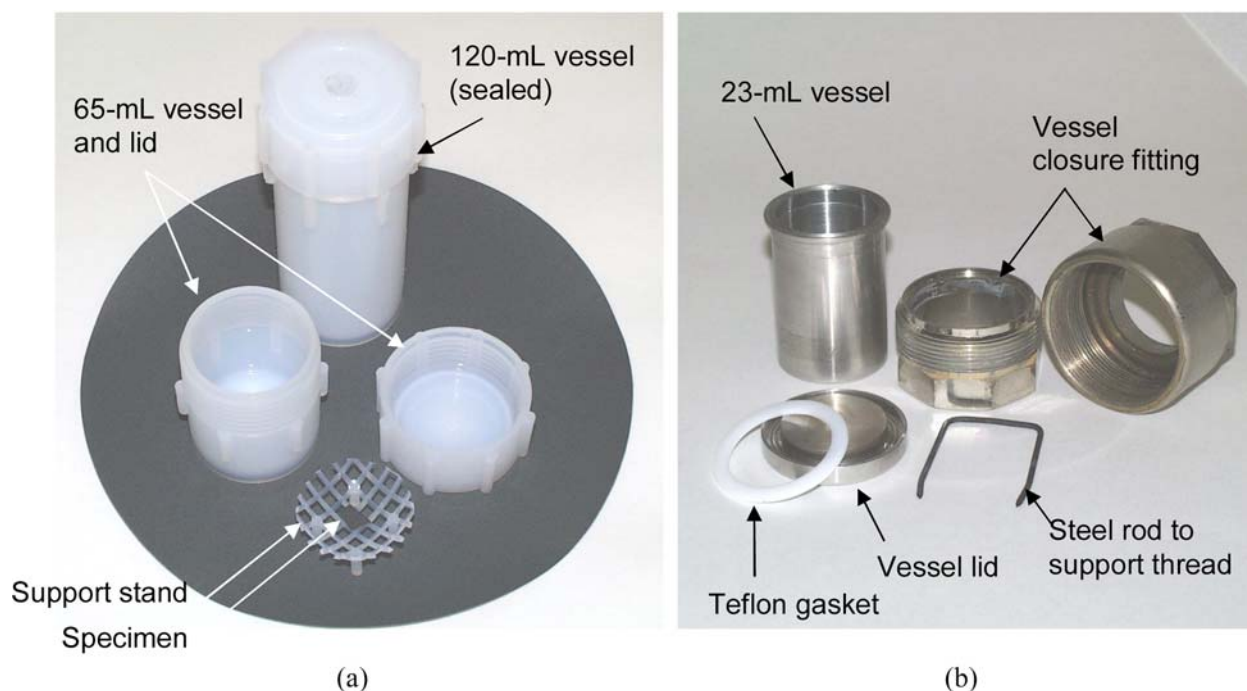


FIG. 2 Photograph of (a) PTFE Teflon Vessel and Support and (b) Type 304L Steel Test Vessel, Support, and Closure Fitting

shall have a tightly fitting lid and be sufficiently impervious to water to limit the loss during the test to less than 10 % of the initial volume (mass) of leachant.

6.3.5 The specimen support shall be constructed of the same material as the vessel or of an equally inert material and designed to hold the specimen near the centroid of the leachate volume throughout the test, but must not contact more than 5 % of the specimen surface area.

6.3.6 Vessel identification and the cleaning history of each vessel must be maintained during testing if the vessels are reused.

6.3.7 A unique identifying number should be permanently marked on each leach vessel and lid. This number is used to identify tests in the oven and to track the cleaning and use history of each vessel and lid.

6.3.8 It is usually convenient to clean several vessels and lids at the same time. This facilitates tracing any inconsistent test responses to improper cleaning of a batch of vessels or to a problem vessel. Each batch of cleaned vessels shall be identified using a unique batch number. It is recommended that a log book of the leach vessel number and date of cleaning 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.

6.4 *Qualification of PTFE Teflon Test Vessels and Supports*—Variations in manufacturing practice may cause particular lots of PTFE Teflon to release unacceptable amounts of fluoride 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. In addition, the fluoride level must always be checked for tests and blanks conducted in PTFE Teflon vessels. It is recommended that the vessels used in a test

series be from the same lot. Measurement of pH shall also be determined in these qualification tests, as well as in the analyses of test leachates. The test matrices in Section 9 require the use of blanks, which will provide additional evidence that excessive F^- release from the vessel has not occurred during testing.

6.4.1 To qualify a lot of PTFE vessels and supports, clean three randomly selected vessels from the lot as described in 6.5.

6.4.1.1 Fill each vessel (with the support in place) to about 85 % capacity with demineralized water and seal the vessel.

6.4.1.2 Place each vessel in a 90°C oven and leave undisturbed for 28 days.

6.4.1.3 After 28 days, let the vessel cool then open and withdraw aliquants for pH and F^- concentration measurements.

6.4.1.4 Measure the pH and F^- concentrations in each aliquant.

6.4.1.5 If the pH is in the range of 5.0 to 7.0 and the F^- is below 0.5 $\mu\text{g}/\text{ml}$, the lot of PTFE is acceptable for use. If the pH is not within the range of 5.0 to 7.0 or the F^- concentration is not below 0.5 $\mu\text{g}/\text{ml}$, repeat the cleaning procedure in 6.5 until both values are within the acceptable range for all three vessels.

6.4.1.6 Clean the remaining vessels with the same number of repeated steps required for the three vessels.

6.5 *Cleaning PTFE Vessels and Supports*—New PTFE vessels and supports must be cleaned to reduce the amount of F^- released during testing. PTFE vessels can be reused after testing provided they are cleaned before reuse and were not used in tests with actinide-doped specimens. This is because actinides are difficult to remove from PTFE and may not be sufficiently removed by leachate acidification and the vessel/

specimen support structure cleaning procedure. As these dopants may be present in very low concentrations in the leachates of subsequent tests, contamination due to leaching from the vessel walls could be significant. Clean new PTFE Teflon vessels and supports following steps 6.5.1 – 6.5.22.

6.5.1 Heat new PTFE test vessels and supports, but not fine monofilament used to suspend test specimens, in a $200 \pm 10^\circ\text{C}$ oven for one week prior to cleaning.

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

6.5.3 Fill vessels approximately 90 % full with 5 wt % NaOH solution and tighten lids.

6.5.4 Place PTFE vessels rated to 0.5 MPa or higher in an oven preheated to $110 \pm 10^\circ\text{C}$. Place PTFE vessels not rated to 0.5 MPa in an oven preheated to $95 \pm 2^\circ\text{C}$.

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

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

6.5.7 Remove lids carefully and dispose of NaOH solution.

6.5.8 Rinse vessels and lids in fresh high-purity water two times.

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

6.5.10 Remove vessels and lids and discard water.

6.5.11 Repeat steps 6.5.8 – 6.5.10.

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

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

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

6.5.15 Remove vessels and allow to cool to room temperature.

6.5.16 Take an aliquant of liquid from each vessel and measure pH.

6.5.17 If the pH is below 5, repeat steps 6.5.1 – 6.5.16 until the pH is above 5.

6.5.18 If the pH is above 7, repeat steps 6.5.8 – 6.5.16.

6.5.19 If the pH is between 5.0 and 7.0 take an aliquant and measure the F^- concentration.

6.5.20 If the F^- concentration is $>0.5 \mu\text{g/mL}$, repeat steps 6.5.8 – 6.5.19.

6.5.21 If the F^- is still $>0.5 \mu\text{g/mL}$ after performing steps 6.5.8 – 6.5.19 twice, repeat steps 6.5.1 – 6.5.19.

6.5.22 If the F^- concentration is $<0.5 \mu\text{g/mL}$, a vessel is acceptable for use.

6.6 *Cleaning of New PTFE Gaskets*—Clean new PTFE Teflon gaskets to be used with stainless steel vessel following steps 6.6.1 – 6.6.6.

6.6.1 Handle the gaskets only with clean tongs, forceps, or gloves.

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

6.6.3 Clean each gasket with high-purity water at ambient temperature for approximately 3 min.

6.6.4 Bake each gasket in an oven at $200 \pm 10^\circ\text{C}$ for a minimum of 4 h.

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

6.6.6 Dry gaskets at $90 \pm 10^\circ\text{C}$ for a minimum of 16 h and store in a clean environment until needed.

6.7 *Clean Used PTFE Teflon Vessels and Supports*—Clean used PTFE vessels and supports following steps 6.7.1 – 6.7.11.

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

6.7.2 Soak vessels and supports for 1 h in 0.16 M HNO_3 (1 wt % HNO_3) at $90 \pm 10^\circ\text{C}$.

6.7.3 Remove vessels and supports and discard acid soak solution.

6.7.4 Rinse vessels and supports again as specified in step 6.7.1.

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

6.7.6 Remove vessels and supports and allow to dry.

6.7.7 Fill the vessels with supports in place approximately 90 % full with fresh high-purity water. 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 in each vessel.

6.7.8 Take an aliquant of the water from at least two vessels from each vessel batch and measure the F^- concentration.

6.7.9 Repeat steps 6.7.4 – 6.7.8 until the pH is in the range of 5.0 to 7.0 and the F^- concentration is $<0.5 \mu\text{g/mL}$.

6.7.10 If the pH and fluoride requirements cannot be achieved after three repetitions of steps 6.7.4 – 6.7.8, then repeat the cleaning procedure starting at step 6.7.1.

6.7.11 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.

6.8 *Clean New Fused Silica Vessels*—Clean fused silica vessels following steps 6.8.1 – 6.8.10.

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

6.8.2 Soak vessels and supports for 1 h in 0.16 M HNO_3 (1 wt % HNO_3) at $90 \pm 10^\circ\text{C}$.

6.8.3 Rinse again as specified in 6.8.1.

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

6.8.5 Remove vessels and discard water. Allow vessels to dry.

6.8.6 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 $90 \pm 2^\circ\text{C}$.

6.8.7 Take an aliquant of the water from each vessel and measure the pH.

6.8.8 Repeat steps 6.8.4 – 6.8.7 until the pH is in the range of 5.0 to 7.0.

6.8.9 If the pH requirement cannot be achieved by three repetitions of steps 6.8.4 – 6.8.7, then repeat the cleaning procedure starting at step 6.8.1.

6.8.10 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.

6.9 *Clean New Stainless Steel Vessels*—Clean new stainless steel vessels using steps 6.9.1 – 6.9.13.

6.9.1 Degrease new Type 304L stainless steel vessels and lids without gaskets and ultrasonicate in 95 % ethanol for approximately 5 min to remove any residual grease or oil left from machining operations:

6.9.2 Rinse vessels and lids three times in high-purity water.

6.9.3 Submerge vessels and lids in 0.16 M HNO₃ (1 wt % HNO₃) for 1 h at 90 ± 10°C.

6.9.4 Remove vessels and lids and discard acid soak solution.

6.9.5 Rinse vessels and lids three times with high-purity water at ambient temperature.

6.9.6 Remove vessels and lids and discard water. Allow vessels and lids to dry.

6.9.7 Submerge the vessels and lids in fresh high-purity water for 1 h at 90 ± 10°C.

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

6.9.9 Fill the vessel 80 to 90 % full with high-purity water. Close the lid and leave in a 90 ± 2°C oven for a minimum of 16 h.

6.9.10 Remove the vessels from the oven and let cool to room temperature. Take aliquant of the water and measure the pH.

6.9.11 If the pH is not in the range of 5.0 to 7.0, repeat steps 6.9.5 – 6.9.10

6.9.12 If the pH is not in the range of 5.0 to 7.0 after 3 repetitions of steps 6.9.5 – 6.9.10, repeat the cleaning steps starting at step 6.9.1.

6.9.13 Dry the vessels in a 90 ± 10°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.

6.10 *Cleaning Used Stainless Steel and Used Fused Silica Vessels*—When stainless steel or fused silica vessels are used in tests 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 in the water is below the detectable level using the analytical method to be employed for concentration measurement of the leachate. Stainless steel vessels are also checked for Si contamination before reuse. Clean used stainless steel vessels using steps 6.10.1 – 6.10.11.

6.10.1 Rinse the vessel and lid with high-purity water.

6.10.2 Fill the vessel 80 to 90 % full with 0.16 M HNO₃ (1 wt % HNO₃). Seal the vessel and place in an oven at 90 ± 2°C to digest for a minimum of 16 h to dissolve radionuclides adhering to the interior of the vessel.

6.10.3 Check the resulting solution for radioactivity. Repeat step 6.10.2 until the radioactivity of the solution is reduced to below the background levels.

6.10.4 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.

6.10.5 Fill the vessel 80 to 90 % full with fresh high-purity water. Close the lid using a new, cleaned gasket (see step 6.6) and place in oven at 90 ± 2°C for at least 24 h.

6.10.6 Remove vessels from oven, then take an aliquant of the water and measure the pH.

6.10.7 Take another aliquant and measure the radioactivity.

6.10.8 For stainless steel vessels, take an aliquant to measure the Si content of the solution.

6.10.9 If the pH is not in the range of 5.0 to 7.0, the measured radioactivity is not at the background level, or Si >1 ppm is detected for stainless steel vessels, repeat steps 6.10.2 – 6.10.8.

6.10.10 If three repetitions of steps 6.10.2 – 6.10.8 do not result in a pH within the range of 5.0 to 7.0, radioactivity below detection, and Si <1 ppm for stainless steel vessels, then repeat the cleaning starting at step 6.10.1.

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

6.11 *Cleaning Solution Bottles*—Solution bottles that will be used to contain analytical samples should be cleaned before use. Clean solution bottles using steps 6.11.1 – 6.11.3.

6.11.1 Rinse bottles three times with a dilute nitric acid solution (~2 wt %). For each rinse, fill bottle to about 10 % bottle volume, place cap on bottle, and shake to rinse all surfaces. Dispose of solution.

6.11.2 Rinse bottles three times with reagent grade (demineralized) water. For each rinse, fill bottle to about 25 % bottle volume, place cap on bottle, and shake to rinse all surfaces. Dispose of rinse water.

6.11.3 Dry bottle and cap in oven, then place cap on bottle and store until use.

6.12 *Mass Measurement*—Material masses shall be determined with balances that provide the following accuracies, depending on the materials being weighed:

TABLE 1 Required Accuracy for Mass Determinations

Leachant and vessels	within 0.25 % of the leachant mass
Chemical reagents used to prepare leachant	within 1 % of the reagent mass
Test specimens	within 0.5 mg

6.13 *Volume Measurement*—Measure leachant volumes gravimetrically or with pipettes, burettes, or flasks calibrated as described in Table 1.

6.14 *Solution Analysis*—Measure solute concentrations using equipment standardized with standards traceable to NIST, preferably, or other recognized organizations, such as EPA or USGS.

6.14.1 Determine and report precision and bias for analyses. Although analytical results should normally be accurate within 10 % when checked by individual measurements on reference solutions, this may not be possible when concentrations in the solution are near detection limits. The detection limits for each analysis must accompany the reported result.

6.14.2 Various analytical techniques can be used to determine the solute concentrations in leachates, including inductively coupled plasma spectroscopy (see Practice C1109 or EPA SW-846A, or both), direct current plasma spectroscopy,

TABLE 2 Required Calibration Schedule

Measurement	Device	Frequency Check and Methods
Temperature	thermocouple or thermometer	6 months NIST standard or ice/boiling water
Voltage	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 before use with commercial or NIST buffer solutions, and at intervals of 60 min during measurements
pH	pH meter	See Test Methods D1293 and 6.11 for guidance
Volume	volumetric flasks	use certified flasks or before use by measuring the mass of pure water contained
Activity	pipettes counting techniques	before use by measuring the mass of pure water transferred twice a day, before and after counting NIST or NIST-traceable standard isotope source for radionuclide of interest

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 small amounts may be of interest, radiochemistry/radiation counting may be needed or desirable.

6.14.3 Analyzing blanks and simulated leachates with test solutions helps ensure that high-quality data are obtained.

6.15 *pH Measurement*—Measure the pH to an accuracy of 0.1 unit using an electrode and meter calibrated with commercial buffers or buffers obtained from NIST. Follow Test Method [D1293](#), Method A to make this measurement.

6.15.1 When measuring the pH of a deaerated solution, conduct the measurement under an argon atmosphere.

6.15.2 When measuring the pH of a brine solution, the measured value will be affected by a significant liquid junction potential that is sensitive to the ionic strength and the activity coefficient of hydrogen that will differ significantly from unity. These effects lead to large uncertainties in the measured values. They are discussed in more detail in [Appendix X1](#).

6.16 *Calibration and Standards*—Calibrate all instruments used in these tests prior to use and check periodically to minimize possible errors due to drift. [Table 2](#) shows the methods and the minimum frequency of calibration for the various devices used. Use standardized 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.⁵ It is recommended that the assays of each chemical be assessed to

determine if impurities, once the leachant is prepared, will exceed detection limits of the 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.

7.1.1 Although any leachant can be used in tests, recipes for a reference brine leachant and a reference silicate leachant are provided to facilitate comparisons of test results from different laboratories. The reference brine and silicate leachant compositions are not intended to represent any particular ground waters.

7.1.2 Blank tests must be conducted with all leachants to measure the stability of the leachant under the test conditions and interactions with the vessel and support.

7.1.3 The density of all leachants should be measured to permit the addition of leachant to the individual leach tests by weight rather than by volume.

7.2 *Demineralized Water*—The water referred to in this procedure is air-saturated reagent water Type I or II conforming to Specification [D1193](#), with a total impurity level, including organics, of less than 0.1 mg/L.

7.2.1 Radiolysis of gases dissolved in water can become an important factor in tests with radioactive materials. Therefore, deaerated leachants should be used in tests with radioactive specimens.

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

7.2.3 The density of pure air-free water at 23°C is 0.9976 g/cm³.

7.3 *Preparation of Reference Brine Leachant*—Prepare the reference brine by dissolving 48.2 g KCl, 90.0 g NaCl, and 116.0 g MgCl₂ (247.9 g MgCl₂·6H₂O) in enough demineralized 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

⁵ *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 *Analar 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.

make 1.00 L of solution. Analyze the leachant to verify the composition and to determine impurity concentrations. Discard the leachant if the concentration of any constituent is in error by more than 10 % from the recipe concentration, which is 35.4 g Na⁺/L, 54.9 mg K⁺/L, 29.6 mg Mg²⁺/L, and 164 mg Cl⁻/L (neglecting the Na and Cl added as NaOH and HCl).

7.3.1 The density of the brine leachant should be measured to permit the addition of aliquants of brine leachant to the individual leach tests by weight rather than by volume.

7.3.2 **Caution**—When using brine in radiation fields, hydrogen gas will be generated and may pressurize the test vessel. Take the appropriate precautions when making such studies. Use appropriate pressure-rated 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.

7.4 *Preparation of Reference Silicate Water Leachant*—Prepare the silicate water leachant by dissolving 0.179 g NaHCO₃ and 0.096 g SiO₂·2H₂O in enough demineralized water to make about 900 mL of solution. Adjust the pH to within 0.1 unit of 7.5 with 0.01 M HCl. Add water to make 1.00 L of solution.

7.4.1 Analyze an aliquant of the leachant to verify the composition (27.1 mg Si/L) and to determine impurity concentrations. Discard the leachant if the concentration of any constituent is in error by more than 10 %.

7.4.2 The density of the silicate water leachant should be measured to permit the addition of aliquants of leachant to the individual leach tests by weight rather than by volume.

7.5 *Repository Water Leachants*—When actual ground waters or leachants that are representative of specific repository waters are used, the rigor with which the data characterizing the water 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, results of its chemical composition analysis, the presence of colloidal material, etc. of any precipitates that may have been formed in the blanks during the test.

7.5.1 Analyze an aliquant of the leachant to measure the composition.

7.5.2 The density of the ground water should be measured to permit the addition of aliquants to the individual leach tests by weight rather than by volume.

7.6 *Other Leachants*—Other leachant solutions are useful when using the test to study the waste form degradation mechanism and measuring model parameters. For example, pH buffer solutions can be used to measure the effect of pH on the test response and leachants with added components can be used to measure common ion (solution feed-back) effects.

7.7 *Leachant Storage*—Use polyethylene or polypropylene bottles with tight-fitting lids to store the leachants. Clean bottles before use following the cleaning steps given in 6.11.

7.7.1 Use the leachant immediately or store in a sealed vessel until beginning the test. If the leachant is not used within one month after it is prepared, report the storage time and

verify the composition by analysis before use in tests. Natural groundwaters should be stored in a dark location to prevent the growth of algae.

7.7.2 Store deaerated solutions in a tightly sealed container with an argon atmosphere above the liquid level and for no longer than one week.

8. Test Specimen Preparation

8.1 Test specimens may be either fabricated individually or cut from larger samples of the waste-form material.

8.1.1 When cutting a specimen from a larger sample, avoid the use of wax or adhesives to hold the sample being cut. If adhesive materials must be used, none of the surfaces of the resulting test specimen shall be surfaces to which the adhesive was applied.

8.1.2 Sawing and cutting test specimens for tests to be conducted in deaerated solutions need not be done in an argon atmosphere. However, a final specimen cleaning step must be carried out under an argon atmosphere using deaerated deionized water.

8.1.3 The surfaces of individually fabricated test specimens may not be representative of the bulk material due to formation of a surface skin, and the responses of such specimens may not be representative of the bulk material. If separate specimens are cast for each test, the user should determine the effect of the as-cast surface by comparing results of tests conducted with an as-cast specimen and a cut specimen without the outer layer has been removed.

8.2 *Characterization of Source Material*—The source material is the sample from which test specimens are extracted. If possible document the fabrication method and fabrication conditions for the source material and provide information on how test specimens were selected. The researcher must include information on the chemical and radiochemical (if applicable) composition and compositional variations of the source material. 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 determine the distribution of alpha-emitting isotopes. The researcher may also wish to characterize the source material with optical microscopy, XRD, SEM-EDX, TEM, or other analytical methods to document microcracking, phase identification, relative concentrations of phases, and homogeneity between test specimens.

8.2.1 For specimens in which an as-fabricated surface is to be leached, analysis of a surface cross-section by SEM-EDX or other applicable surface spectroscopy techniques is also required to determine whether the surface composition differs from the bulk composition. If differences do exist, the possible effects on the test results should be discussed with the test results.

8.2.2 When the waste form source material is a heterogeneous or multiphase material, it may not be possible to produce test specimens exposing equal amounts of all phases to the leachants in a test series. In this case, the test operator must document the differences between the test specimens that are

used to ensure that the test specimens contain a representative distribution of the different phases. This should be documented with optical microscopy, SEM-EDX, or other applicable techniques. Individual particles of component phases may be large enough relative to the dimensions of the test specimen to estimate the exposed area.

8.3 Test Specimens—The test specimen is preferably prepared as a regularly shaped monolith, either a tablet (with six flat surfaces) or a disk such that the surface area exposed to the leachant can be easily calculated from the geometry and measured dimensions.

8.3.1 All surfaces must be prepared in a consistent manner using either an abrasive saw or an alternative technology to provide a reproducible surface finish (200, 320, 600 grit or other surface finish). The surface finish will affect the test response of many materials.

8.3.1.1 Samples having a tabular shape allow for the best control of the surface finish.

8.3.1.2 The preferred surface finish will depend on the material being tested.

8.4 Specimen Cutting—When specimens are prepared from a larger sample, use a saw to prepare tabular specimen or a core drill and saw to prepare disk samples.

8.4.1 The saw blade should be appropriate for the material being cut. For example, a 200-grit diamond-impregnated cutting surface is appropriate for most glasses.⁶

8.4.2 Use water as the cutting fluid unless the material is known or suspected to contain a water-soluble phase, in which case absolute ethanol or another polar fluid (for example, kerosene or cyclohexane) should be used as the cutting fluid. Experiments can be conducted to measure the extent to which the sample dissolves in the cutting fluid.

8.4.3 The size or exact shape of the specimen is not critical, provided the geometric surface area can be determined, but a surface area of about 400 mm² is recommended. This size is easily handled during sample preparation and requires 40 mL leachant for a standard test conducted at a S/V ratio of 10 m⁻¹.

8.4.4 A metal-bonded diamond core drill with an inner diameter of about 1 cm can be used to remove a core sample from the source material.⁷ The diameter of the core can be polished by carefully chucking it in a low-speed drill. Specimen disks can then be cut from the core.

8.4.5 Tabular specimens can be produced by cutting a bar from the source material and polishing the four sides. Specimen tablets can then be cut from the bar and the faces polished. The recommended technique using a Buehler low-speed saw is provided in [Appendix X2](#).

8.5 Specimen Polishing—The saw-cut specimens are polished using successively finer grit paper with water (or absolute

alcohol) lubrication. Saw-cut specimens will have a surface finish similar to 200-grit, depending on the hardness of the material, the condition of the wafering blade, and the cutting speed. The use of a mechanical polishing wheel is recommended to provide uniform surface finish. Typically, specimens can be polished successively with 240-grit, 320-grit, 400-grit, and 600-grit paper (and with finer grits) to produce a uniform and reproducible surface finish. Jigs can be constructed to hold specimens during polishing and automatic polishers can be used.

8.5.1 Place polishing paper of desired grit on polishing wheel and lubricate well with water (or absolute alcohol). Gently press sample face to be polished near center of paper and start wheel at low speed. Maintaining sample orientation, slowly rotate the sample around the paper in direction counter to wheel rotation moving towards the outside edge of wheel and then back towards the center. This motion subjects the sample to polishing in all directions. The force needed to hold the sample will vary with the hardness of the material, the grit size of the polishing paper, and the speed of the wheel. Because it is likely that hand-held samples will be pulled loose during polishing (especially when using coarse grit paper), drains in the tray beneath the wheel should be covered with a screen to prevent the loss of samples. The speed of the polishing wheel should be kept low enough to avoid binding and pulling the specimen, but high enough to effectively polish it.

8.5.2 Rinse the specimen with water (or absolute ethanol) and inspect the scratches on the specimen for uniformity in depth and directional randomness.

8.5.3 Repeat steps [8.5.1](#) and [8.5.2](#) with a finer grit size paper. The polishing times required to achieve a uniform surface usually increase with finer grit paper. For example, 10 seconds may be adequate for achieving a 240-grit finish whereas several minutes may be required to attain a 600-grit finish.

8.5.4 For typical glasses, a 600-grit finish will have a mirror finish to the naked eye with shallow scratches visible using a jeweler's loupe. Failure to remove surface scratches within a reasonable polishing time may require brief repolishing with a coarser grit.

8.5.5 Care should be taken to avoid rounding the faces at the edges during polishing.

8.6 Specimen Cleaning—Clean the specimens using the following procedure:

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

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

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

8.6.4 When performing experiments using deaerated solutions, additionally wash the specimen three times using deaerated high-purity water under an argon atmosphere and store the specimen under argon until it is placed in the test vessel.

⁶ 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.

⁷ Suitable diamond core drills are available for Starlite Industries, Inc., 1111 Lancaster Ave., Rosemont, PA 19010, Part #102135, ½ inch.

8.7 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.

8.8 Specimen Dimensions and Surface Area—The dimensions of each specimen shall be measured with a calibrated device (for example, digital calipers) to the nearest 0.01 cm. The measured dimensions are used to calculate the geometrical surface area, which is used in calculations to be performed with the test results. The porosity of a test specimen is not taken into account within this procedure, but the effects can be assessed by the user based on additional measurements that are performed in addition to this procedure.

8.8.1 For disk-shaped specimens, at least two measurements of the diameter and two measurements of the height shall be made at diametrically opposite locations. The geometric surface area and volume are calculated by modeling the specimen as a right circular cylinder and using the arithmetic averages of the measured diameters and heights.

8.8.2 For tabular-shaped specimens (that is, specimen with six flat faces and unequal edge lengths), the lengths of each edge and the length of one diagonal shall be measured.

8.8.3 The surface area is calculated based on the measured dimensions and the geometric shape of the test specimen.

8.8.4 If the specimen face has a regular geometric shape, calculate the area from the measured dimensions.

8.8.5 If it does not have a regular shape, the surface area can be determined as described in [Appendix X3](#).

8.8.6 The calculated surface area can be modified to take into account deviations in the specimen shape from an ideal right circular cylinder or tablet based on additional measurements and geometric calculations. For example, the specimen may have a nicked edge.

8.8.7 Although the use of a single monolithic specimen in each test is preferred, up to four monoliths can be used to provide the desired surface area, if necessary, provided that each can be placed in the test vessel without contacting another specimen.

8.8.8 The uncertainty in the surface area of the specimens contributes to the uncertainty in the normalized mass loss calculated from the test response and should be quantified, for example, by using the propagation of errors method or, preferably, the method developed by the International Committee for Weights and Measures (CIPM) and promulgated by NIST, see [Appendix X4](#).

8.8.9 Weigh each test specimen to an accuracy of 0.5 mg.

9. Test Matrices

9.1 A series of several tests, referred to as a matrix of tests, is usually conducted with a material of interest to address a particular issue.

9.1.1 Tests are often conducted for a series of durations to determine the kinetics, at several temperatures to determine the activation energy, at several S/V ratios to determine chemical affinity effects, etc.

9.1.2 The matrix identifies the sets of test parameters to be used, the number of replicate tests, and the series blank tests to be conducted.

9.1.3 If Type 304L stainless steel test vessels are used, it is recommended that key tests and blanks be performed in duplicate because high chloride levels, which may have a significant effect on test results, have occasionally been observed in tests conducted using stainless steel vessels. By conducting tests in duplicate, the researcher is reasonably assured of having at least one reliable data point for each test condition.

9.2 Blank tests conducted without a test specimen are required in most matrices.

9.2.1 Blank tests serve to monitor interactions between the leachant and the test vessel and changes in the leachant composition under the test conditions.

9.2.2 The results of blank tests are used to provide background concentrations of solutes to adjust the results of tests with specimens.

9.2.3 If several test matrices with one or more waste forms using the same batch of leachant are started simultaneously in the same oven, the same blank tests may be used for each matrix.

9.3 Reference Test Matrices—Four reference test matrices with specific test conditions are provided to facilitate the direct comparisons of tests conducted with different materials and at different laboratories. The reference test matrices are summarized in [Table 3](#) and discussed below. These represent the series of tests recommended to support the determination and comparisons of waste form behavior, including replicate tests conducted to determine intralaboratory precision. The test period must be controlled to within 2 % of the target test duration for direct comparisons of test results. Additional tests can be conducted to supplement each matrix and meet the user's needs. The matrices and their purposes are described in steps [9.3.1](#) – [9.3.4](#).

TABLE 3 Reference Test Matrices

Reference Matrix	A	B	C	D
Leachant	Demineralized Water	Demineralized Water, Brine, or Silicate Water	Demineralized Water, Brine, or Silicate Water	pH Imposing Solutions
S/V ratio, m ⁻¹	10	10	10	2
Temperature, °C	90	90	20, 40, 70, 90	20, 40, 70, 90
Test Durations, d	7(3)	28(3)	3, 7, 14, 28, 56, 91, 182, and 364, then 6-month intervals	0.67, 1(3), 1.67, 2, 3, 4, 5, 6, 7, 10, and 14(2)
Blank Durations, d	none	28(2)	28, 91, 364	7 and 14

9.3.1 *Matrix A: 7-day Tests at 90°C*—This matrix is intended for screening tests used during waste form development, where the specific objective could be measuring the qualitative effects of composition variations, waste loading, processing parameters, etc., on the chemical durability of the waste form.

9.3.1.1 The test temperature is 90°C.

9.3.1.2 The leachant is demineralized water.

9.3.1.3 The test specimen surface finish is 600 grit.

9.3.1.4 The S/V ratio is 10 m⁻¹.

9.3.1.5 The test duration is 7 days.

9.3.1.6 Triplicate tests are conducted to address the potential effects of inhomogeneities in the test specimens.

9.3.1.7 No blank test is required.

9.3.2 *Matrix B: 28-day Tests at 90°C*—This matrix can be used to compare the test responses of different waste forms and tests conducted with a material at different laboratories. The data set provides a measure of the dissolution rate with possible solution feedback effects.

9.3.2.1 The test temperature is 90°C.

9.3.2.2 The leachant is demineralized water, reference silicate water, or reference brine.

9.3.2.3 The test specimen surface finish is 600 grit.

9.3.2.4 The S/V ratio is 10 m⁻¹.

9.3.2.5 The duration for tests and blank tests is 28 days.

9.3.2.6 Triplicate tests are conducted for 28 days to address potential inhomogeneities in the test specimens and provide a measure of precision for test execution.

9.3.2.7 Duplicate blank tests are conducted for background adjustment.

9.3.3 *Matrix C: Long Term Tests*—This matrix is used to measure the effect of solution feed-back and the approach to solution saturation in a closed leaching system. Tests conducted at different temperatures can be used to determine the temperature dependence, but quantitation of the temperature effect requires determination of the rate at each temperature rather than comparing the results of tests conducted for the same duration.

9.3.3.1 The test temperature is 90°C, with optional tests at 20, 40 and 70°C.

9.3.3.2 The leachant is water, reference silicate water, or reference brine.

9.3.3.3 The surface finish is 600 grit.

9.3.3.4 The S/V ratio is 10 m⁻¹.

9.3.3.5 Blank tests are conducted for 28, 91, and 364 days and every six months thereafter (to the culmination of test series extending beyond 364 days) in each leachant and under each test condition.

9.3.4 *Matrix D: Short Term Tests*—This matrix can be used to measure the dissolution rate of a material at a particular temperature and solution pH. For materials that dissolve by an affinity-controlled mechanism, the effect of solution feed-back can be determined from tests conducted for more that a few

days.⁸ The dissolution rates determined at each temperature can be compared to evaluate the temperature dependence of the rate.

9.3.4.1 The test temperature is 90°C. Additional tests at 20°C, 40°C, and 70°C are recommended for measuring the temperature dependence of the dissolution rate. Tests at 20°C require the use of a cooling device (for example, a water bath), but are recommended for estimating rates at near the ambient temperatures of many disposal sites.

9.3.4.2 The leachant is a solution that imposes a particular pH value. Commercial pH buffer solutions or buffer solutions prepared in the laboratory may be used. These solutions have little buffering capacity, but are effective for short-term tests at low S/V ratios in which the changes in the solution composition are small. The pH values of most test solutions will be higher at room temperature than at the elevated test temperature, for example, 1 pH unit higher at room temperature than at 90°C. Many commercial buffer solutions are certified up to about 60°C. pH values can be measured at elevated temperatures, but this is not required. The detailed solution composition can be used to calculate the pH at the test temperature with geochemical computer codes, but such analyses are beyond the scope of this method. Instead, the method only requires that the buffer composition used to impose the pH be reported with the test results. The releases from the specimen of those elements present in the pH imposing solutions (excluding contaminants) cannot be used to calculate the dissolution rate.

9.3.4.3 The surface finish is 600 grit.

9.3.4.4 The S/V ratio is 2 m⁻¹. A lower S/V ratio may be required to minimize solution feed-back under some test conditions, such as for tests with an alkali silicate glass conducted at 90°C and pH 12.

9.3.4.5 The test durations are 16 h, 1 d, 40 h, 2 d, 3 d, 4 d, 5 d, 7 d, 10 d, and 14 d. The 16 and 40 hour durations permit a test to be initiated at 5 pm and terminated at 9 am the following morning. Test initiations can be staggered to avoid the need to terminate a test during the weekend. Longer durations may be needed to produce measurable releases in tests at low test temperatures. Triplicate tests are conducted for 1 day and duplicate tests for 14 days because these are important for establishing the effect of the as-prepared surface and determining the saturation concentration for affinity-controlled release, respectively.

9.3.4.6 Blank tests are conducted for 7 and 14 days to provide background concentrations and indicate any degradation of the leachant due to heating or interactions with the test vessel. Background concentrations are also provided by the leachant composition.

⁸ Ebert, W. L., *Comparison of the Results of Short-Term Static Tests and Single-Pass Flow-Through Tests with LRM Glass*, Argonne National Laboratory report ANL-06/51 (2006).

10. Procedure

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

10.2 The procedure consists of immersing a test specimen in a leachant solution at a known S/V ratio selected by the user, placing the sealed vessel in an oven set at the temperature selected by the user, and leaving it undisturbed for the desired test duration. After the specified time interval, the vessel is removed from the oven, allowed to cool, and then opened. Aliquants of the leachate are taken for analyses to measure the solution pH, the concentrations of components released from the test specimen, and other optional analyses.

10.2.1 The release of soluble components from the test specimen to solution is the primary means for assessing the extent of specimen degradation.

10.2.2 The reacted specimen can also be examined to study the degradation mechanism and may provide an additional measure the extent of reaction.

10.2.3 Reference test matrices are identified in 9.3 to facilitate comparisons of test results for different materials and different laboratories.

10.2.4 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. A test program may call for testing of both a radioactive specimen and a non-radioactive surrogate material.

10.2.5 This procedure also applies to blank tests, except that the specimen is omitted.

10.3 *Characterize the Leachant Prior to Testing:*

10.3.1 Determine the pH of an aliquant of the leachant and then discard that aliquant.

10.3.2 Measure the composition of the leachant.

10.3.3 If a deaerated leachant is used, determine the dissolved oxygen content of an aliquant of the leachant and then discard that aliquant. 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.

10.4 Determine the volume of leachant to be used in each test based on the geometric surface area of the specimen and the desired S/V ratio.

10.4.1 The volume of the leachant, support, and specimen should not exceed 90 % of the vessel volume to provide adequate free volume for expansion at the test temperature.

10.4.2 If the leachant is to be added by mass, calculate the mass of leachant to be used by multiplying the desired volume by the measured density of the leachant.

10.4.3 Confirm that enough leachant is available for all of the tests to be conducted in the series (including blank tests).

10.5 *Assemble Test Vessel*—The test is assembled by placing the support stand (if used), test specimen, and leachant in the test vessel, sealing the vessel, and placing it in an oven (or other controlled temperature device) set to the appropriate temperature.

10.5.1 If the test specimen is to be set on a support stand, place the support stand and specimen in the vessel before

adding the leachant. Weigh the combined vessel, support, and specimen, including the vessel lid and any closure fittings. Add leachant to the vessel slowly to avoid trapping air bubbles that could prevent leachant from contacting the specimen. Tap the vessel lightly to dislodge any bubbles that do form. Completely immerse the specimen in the appropriate mass of leachant. Place the lid on the vessel and the closure fittings (if any). Weigh the assembled vessel.

10.5.2 If the test specimen is to be hung by a thread or wire in the leachant, adjust the length of the thread or wire so that the specimen will be supported near the centroid of the leachant volume before adding leachant. Weigh the vessel without the leachant, including the lid and closure fittings (if any). Place the appropriate mass of leachant into the clean test vessel. Lower the specimen into the leachant slowly to avoid trapping air bubbles that could prevent leachant from contacting the specimen. Tap the vessel lightly to dislodge any bubbles that do form. Completely submerge the specimen in the leachant. Weigh the assembled vessel.

10.5.3 The volume of leachant used in a blank test should be approximately the same as the leachant volumes in the tests with specimens for which it serves as a blank.

10.5.4 When using deaerated solutions, carry out the procedure in an argon atmosphere.

10.5.5 When using brine leachants, follow precautions noted in 7.3.2 when leaching under high-radiation fields.

10.6 *Test Initiation*—Place assembled test vessel into the preheated oven (or controlled temperature device) in a zone where the temperature has been determined to be within 1°C of the target test temperature (see 6.2). It is recommended that the position of the vessel in the oven (or controlled temperature device) be mapped to help locate the vessel when monitoring for mass loss and when the test is terminated.

10.6.1 For tests conducted at 40°C or above, the testing period starts when the test vessel is placed into the test oven. This must be within 30 min of assembly.

10.6.2 For test conducted at or below 40°C, the testing period starts when the leachant first contacts the test specimen.

10.6.3 Record the date and time that the test was initiated (d:h:min).

10.6.4 If the test vessel has a screw-top lid, retighten the vessel lids at times about 1 h and 24 h after the start of the test to ensure a good seal. If a PTFE vessel is used, the lid should be re-tightened daily until no further slack is detected. This may take three or more days. At each of these time periods, swirl the solution in the vessel gently or tap the vessel gently, or both, to dislodge any bubbles that are seen. Do not disturb the placement of the specimens however. Thereafter, avoid movements of the test vessel that could disturb the test specimen.

10.6.5 Experience has shown that a slow water loss through the PTFE test vessel is inevitable and occurs at a rate of approximately 0.01 mL/day.⁹ To detect faulty vessel seal or an off-standard test vessel, check the mass of an assembled test vessel used in a long-term test at least every three months.

⁹ To eliminate or reduce water leakage, a PTFE vessel rated at 0.5 MPa can be used, such as that supplied by Saville, Minnetonka, MN.

10.6.6 If the mass of a test vessel decreases to below 85 % of the initial mass, the test is considered to have failed and the test results should not be used quantitatively.

10.6.7 If the mass of a test vessel decreases to between 90 % and 85 % of the initial mass, terminate the test as soon as possible. The test results can be used quantitatively.

10.7 *Test Termination*—At the conclusion of the scheduled testing period (or if water loss requires early termination), remove the test vessel from the test oven.

10.7.1 For tests conducted under one of the reference matrices given in 9.3 for comparisons to other materials, the testing period must be controlled to within 2 % of the target test duration.

10.7.2 Record the date and time (d:h:min) at which the vessel was removed from the oven. This is defined as the end of the test duration.

10.7.3 Weigh the test vessel. Subtract this mass from the vessel mass when the vessel was assembled (step 10.5) to determine leachant loss.

10.7.3.1 If the final vessel mass is greater than 90 % of the initial vessel mass, allow the vessel to cool to near room temperature.

10.7.3.2 If the final vessel mass is less than 90 % of the initial vessel mass, the test results are not suitable for quantitative comparisons and the test must be repeated.

10.7.4 Open the vessel carefully to avoid disturbing the test specimen.

10.7.4.1 If the test specimen is tied to the vessel lid, remove lid carefully and place the lid on a clean holder (for example, an empty test vessel).

10.7.4.2 If the test specimen is on a support stand, remove aliquants for solution analyses (step 10.7.5) prior to removing the test specimen.

10.7.5 Transfer aliquants of the leachate for analyses using a pipette to clean tared bottles labeled to identify the test and analysis to be performed.

10.7.5.1 Take an aliquant to measure the leachate pH (required).

10.7.5.2 Take an aliquant to measure the concentrations of major components of the test specimen (required).

10.7.5.3 If deaerated leachant was used, an aliquant must be taken for oxygen analysis. This aliquant must be stored in an inert environment.

10.7.5.4 An aliquant may be taken for anion analysis (optional).

10.7.5.5 An aliquant may be taken for colloid analysis (optional). Note that colloids may form as the leachate cools from the test temperature.

10.7.5.6 An aliquant may be taken for radionuclide analysis (optional).

10.7.5.7 Aliquants may be taken for other analysis (optional).

10.7.6 Properly store or dispose remaining leachate.

10.8 Remove the test specimen from the wire suspending it from the vessel lid or from the specimen support. Rinse the specimen gently in high-purity water for approximately 5 s, then place specimen in clean container.

10.8.1 Dry the monolithic specimen to constant weight (to nearest 0.5 mg) and record the weight.

10.8.2 Weigh any material that sloughs off the surface during rinsing and handling and include the results with the test data.

10.8.3 It may be useful to examine the surfaces of some test specimen after testing. 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 measure elemental depth profiles).

10.9 Measure the pH of the cooled leachate within one hour. Discard the aliquant.

10.9.1 If a deaerated leachant was used, the oxygen content of the aliquant should be analyzed under an inert (argon) atmosphere immediately. This measurement will be used to determine whether oxygen contamination occurred during the test by comparison with the oxygen content measured in the leachant prior to the test. If excessive contamination has occurred, the test must be repeated. Excessive oxygen contamination is arbitrarily defined in this method as a leachate oxygen content that is greater than a factor of ten higher than the oxygen content of the initial leachant.

10.9.2 Inspect the leachate and vessel for residual solids either precipitated from solution or sloughed from the specimen and record observations.

10.9.2.1 If no solids are observed, proceed to 10.10 without filtering.

10.9.2.2 If solids are observed, the leachate should be filtered prior to analysis.

10.9.2.3 Prepare a clean membrane filter having a pore size no greater than 0.45 μm by passing in series three rinses of 6 M HNO_3 , each equal to 20 % of the equipment volume, three rinses of high purity water, and three rinses of fresh leachant.

10.9.2.4 Pass an aliquant of the leachate to be analyzed through the filter and collect filtrate in a clean, tared, and labeled solution bottle.

10.9.2.5 If desired, the filtrate solution may be sampled and passed sequentially through finer filters to measure the distribution of suspended and colloid-sized materials.

10.9.2.6 The user must determine the role of the solids. Pieces of reacted materials that have sloughed off the sample may have had no effect on the solution composition or may have exposed additional surface area that reacted during the test (that is, increase the S/V ratio of the test). Colloids and phases that precipitate from the solution should be included in the calculation of the amount of material reacted.

10.10 *Acid Strip of Test Vessel*—Components released from the test specimen that adhered to the interior surfaces of the test vessel shall be dissolved for analysis by filling the vessel with a 1 wt% nitric acid solution and heating for several hours. Dissolution of actinide elements from the vessel may require several strips with 1 wt% nitric acid. Alternatively, the interior of the vessel may be analyzed directly for residual radioactivity if the contaminant of interest is a γ -emitter.

10.10.1 Remove all leachate from the test vessel and rinse the test vessel and sample support three times with demineralized water.

10.10.2 Place sample support in vessel and allow to dry.

10.10.3 Add an amount of 1 wt% nitric acid solution equal to the amount of leachant that was used in the test plus about 3 mL to immerse the surfaces that had been contacted by the leachate during the test. However, the solution must not occupy more than 90 % of the vessel volume.

10.10.4 Determine the mass or volume of added nitric acid.

10.10.5 Place lid on vessel and replace closure fixture (if necessary).

10.10.6 Place sealed vessel in 90°C oven for 12 to 24-h.

10.10.7 Remove vessel from oven and let cool to room temperature.

10.10.8 Open the vessel and transfer aliquants appropriate for the planned analyses of the acid strip solution to new polyethylene or polypropylene bottles.

10.10.8.1 The acid strip solutions should be analyzed for matrix elements of the test specimen.

10.10.8.2 The acid strip solutions should be analyzed for radionuclides, if present in the test specimen.

10.10.8.3 The acid strip solutions from tests conducted in a PTFE vessel shall include a determination of F^- concentration.

10.10.8.4 Properly dispose of the remaining acid strip solution.

10.10.9 Repeat the acid strip procedure (beginning with step 10.10.1) to verify all adhering species have been removed.

10.11 *Prepare Test Solutions for Analysis*—The compositions of the leachates must be analyzed for every test. Some tests may also require analysis of filtered leachate and one or more solutions from acid strips of the test vessel. A single analysis of the leachant is adequate for tests in which it is used within a 1-month period. It is recommended that the leachant be analyzed within one month prior to use in testing. It is also recommended that the leachant be analyzed periodically with leachates.

10.11.1 Aliquant for pH measurement: Transfer required amount of solution to clean container with a lid. The container size will depend on electrode to be used. For example, the use of a microtip electrode will only require about 0.5 mL of leachate, which can be contained in a small capped vial.

10.11.1.1 It is not necessary to measure the aliquant mass for pH analysis.

10.11.1.2 The pH should be measured within 1 hour of terminating the test to minimize the effect of CO_2 uptake.

10.11.2 Aliquant taken for F^- analysis for tests conducted in a PTFE vessel: Transfer the required amount of solution to clean container with a lid.

10.11.2.1 It is usually not necessary to measure the aliquant mass for F^- analysis.

10.11.3 Aliquant for cation analysis: Acidifying the solution to about pH 1 ensures metals will remain dissolved.

10.11.3.1 Measure the mass of a clean solution bottle.

10.11.3.2 Transfer an aliquant of the leachant, leachate, solution from a blank test, filtered leachate, or acid strip solution to the bottle.

10.11.3.3 Measure the combined mass of the bottle and transferred solution.

10.11.3.4 (Optional) Add reagent water (demineralized water) to dilute the solution or provide the required amount of solution for analysis, or both.

10.11.3.5 Add an amount of ultrapure concentrated nitric acid equal to 1 % of the solution mass.

(a) Use hydrochloric acid instead of nitric acid for brine leachates and blank solutions.

(b) Do not add acid to acid strip solutions.

10.11.3.6 Measure the combined mass of the bottle, transferred solution, demineralized water, and acid.

10.11.3.7 Submit the acidified solution for analysis as soon after test termination as possible.

10.11.4 Aliquants for other analyses: Transfer aliquants to appropriate labeled containers.

11. Calculations

11.1 *Use of Blanks*—The blank test data are used to track the stability of the leachant under the test conditions and to adjust the leachate concentrations by subtracting background concentrations for species in the leachant, vessel contamination, analytical contamination, etc.

11.1.1 The application of blank test results is restricted to tests conducted in the same leachant and at the same temperature.

11.1.2 Blank data can be used as background concentrations for tests conducted with the same leachant and under the same conditions but for different durations.

11.1.3 Acceptable differences in the durations of blank tests and tests with a specimen depend on the purpose of the test. Differences up to 6 months are permitted for the long-term tests in Matrix D, whereas differences of 7 days or less are permitted in the short-term tests in Matrix C.

11.2 *Use of Blanks as Background Concentrations*—Adjust the leachate concentration of a species by subtracting the concentration of that species measured in an appropriate blank test. Refer to the result as the background-adjusted concentration.

11.2.1 In the reference matrices, a blank test is conducted for the longest test duration so the difference between concentrations in this blank test and the leachant indicates the cumulative effects of heating and vessel interactions. When other blank tests are conducted at intermediate durations, the results can be analyzed to distinguish trends, such as increases or decreases in element concentrations, from a single occurrence of contamination.

11.2.2 In cases where a blank test was conducted for the same duration, concentrations measured in that blank test can be used to adjust measured concentrations in the test.

11.2.3 For cases where a blank was not obtained after the same test duration, the correction can be made by using the average of blank data that bracket the test, single value for blank of similar test duration, or an interpolated value based on the trend of several blank tests.

11.2.4 The investigator must report all individual blank concentrations.

11.3 *Mass Released*—The mass of element i released from the test specimen j is calculated from the measured concentrations of i in the leachate and in the blank and the final solution volumes.

11.3.1 The final solution volume can be calculated from the initial mass of leachant, the mass of water lost during a test, and the density of water by using Eq 1:

$$V_{fj} = \left(V_{ij} - \frac{d_j}{\rho_w} \right) \quad (1)$$

where:

V_{fj} = final volume of leachate in test vessel containing specimen j
 V_{ij} = initial volume of leachant in test vessel containing specimen j
 d_j = total vessel mass loss during test with specimen j
 ρ_w = density of water

11.3.1.1 Note that pure water is lost due to evaporation of the leachate during the test, and the entire mass loss of the sealed vessel is attributed to the loss of water.

11.3.1.2 An increase in the total vessel mass is possible due to weighing uncertainty. Mass gains less than twice the uncertainty in the measured mass should be ignored as experimental uncertainty. Mass gains in excess of twice the uncertainty in the measured mass should be investigated as potential miscalibration of the balance, misidentification of the vessel, or other error.

11.3.2 The mass released can be calculated from the measured concentrations and final leachate volumes in tests and blank tests by using Eq 2:

$$m_{ij} = (C_{ij} \times V_{fj}) - \sum_{k=1}^a V_{Bk} \times \frac{B_{ik}}{a} \quad (2)$$

where:

m_{ij} = mass of element i released from the test specimen j , in g
 C_{ij} = concentration of element i measured in leachate from specimen j , in g/L
 V_{fj} = final volume of leachate in test vessel containing specimen j , in L
 B_{ik} = concentration of element i measured in blank test k , in g
 V_{Bk} = final volume of solution in blank test k , in L
 a = number of blank solutions used for background adjustment

11.4 Calculate the normalized elemental mass loss based on the release of element i , NL_i , using Eq 3:

$$NL_{ij} = \frac{m_{ij}}{f_i \times S_j} \quad (3)$$

where:

NL_{ij} = normalized elemental mass loss from specimen j based on element i , in g/m²
 m_{ij} = mass of element i in the leachate from specimen j , in g
 f_i = mass fraction of element i in specimen j
 S_j = surface area of specimen j , in m²

11.4.1 Differences in the normalized elemental mass loss values calculated based on different elements may indicate non-stoichiometric release from the test specimen or the retention of some elements in alteration phases.

11.4.2 When radioactivity is measured rather than concentration of solute, the normalized elemental mass loss can be calculated by using Eq 4:

$$NL_{ij} = \frac{a_{ij} \times W_j^o}{A_{ij}^o \times S_j} \quad (4)$$

where:

NL_{ij} = normalized elemental mass loss from specimen j based on element i , in g/m²
 a_{ij} = total activity of isotope i in the leachate from specimen j , in Ci
 A_{ij}^o = total activity of isotope i in specimen j , in Ci
 W_j^o = initial mass of the specimen j , in g
 S_j = surface area of specimen j , in m²

11.4.3 Calculate the change in mass of the test specimen m_j by subtracting the final mass measured in steps 10.8.1 and 10.8.2 from the initial mass measured in step 8.6.3. Calculate the normalized mass loss using Eq 5:

$$NL_j = \frac{m_j}{S_j} \quad (5)$$

where:

NL_j = normalized mass loss from specimen j , in g/m²
 m_j = mass loss of specimen j , in g
 S_j = surface area of specimen j , in m²

11.5 *Calculation from Stainless Steel or Fused Silica Test Vessels*—When stainless steel or fused silica test vessels are used, the leachate may contain elements released due to corrosion of the vessel. Test data for elements in the test specimen that are also components of the vessel (notably Si from fused silica vessels and Fe, Cr, and Ni from Type 304L stainless steel vessels) should be clearly identified as potentially biased by vessel contamination. Results for those elements should be reported in separate tables and plots.

12. Report

12.1 Key aspects of the test that should be reported include information regarding the material being tested, objectives of the test matrix, preparation of the test samples, preparation of the leachant, information regarding the test vessels, data from test execution and solution analyses, calculations performed with the test results, and estimated uncertainties. Because experience with the test specimen preparation and test performance are known to affect the test results, the name of the investigator and affiliation should be reported for every test. In addition, the dates over which the tests were conducted and solutions were analyzed should be reported, and when report was submitted.

12.2 *Material and Specimen Selection*—Description of source material being tested, including any unique identification number, phase composition, chemical composition, detailed sample preparation procedure and identification of starting materials, manufacturing technique, size, shape, thermal history, etc. of the prepared test specimens, and unique material identification number.

12.2.1 Discuss how test specimens were selected and obtained from source material. For example, provide a sketch to show locations of test specimens taken from source material.

12.2.2 If applicable, provide a description and short discussion of results from material characterizations of source material from all solid-state techniques used. For example, report microscopic examinations performed to detect and identify inclusion phases.

12.2.3 List the intended and as-analyzed chemical composition of the test specimens (source material). If available, provide quantitative information on homogeneity of specimens and analytical uncertainties.

12.3 *Test Matrix*—Provide the test matrix and discuss the objectives of each test series.

12.4 *Specimen Preparation*—Describe the procedures used to prepare test specimens and the equipment that was used, including descriptions of saw blade or core drill bit, grit sizes used to polish test specimens, date of preparation, and storage conditions.

12.4.1 Provide a description of the technique used to measure specimen dimensions and calculate specimen surface areas.

12.4.2 Identify the calipers used to measure specimen dimensions and the calibration record.

12.4.3 List the unique identification number, measured dimensions, calculated surface area, and measured mass of each test specimen. Give corresponding target volumes of leachant to attain the desired S/V ratio with each specimen.

12.5 *Leachant Preparation*—Identify the leachant used for every test.

12.5.1 If demineralized water is used, provide analysis or measured resistance (or conductivity) used to verify purity.

12.5.2 If the reference silicate water or brine is used, provide the as-batched and analyzed compositions, the measured pH value, and the measured (or assumed) density. Also provide the date the solution was made and storage conditions.

12.5.3 If a solution of reagents is used to impose a pH or provide a common ion, provide a list of the vendor and lot numbers for each chemical and the assayed impurities. Report the as-batched and analyzed compositions and the pH value measured at room temperature. Also provide the date the solution was made and the measured (or assumed) density of the solution.

12.5.4 If a ground water is used, provide information regarding the source and the analyzed composition and the pH value measured at room temperature. Also provide the measured (or assumed) density of the ground water solution.

12.6 *Test Apparatus*—Identify the equipment used to perform tests by manufacturer, model, and serial number.

12.6.1 Describe the oven or temperature control apparatus used in the test, how temperature was measured and monitored, thermometer or thermocouple identifiers, calibration records, etc.

12.6.2 Identify the type of test vessel used, including material, model number and manufacturer. Provide a summary of previous use of the vessel, its cleaning history, etc.

12.6.3 Identify all balances used in testing and their calibration histories.

12.6.4 Identify the method used to support test specimen in test vessel during the test.

12.6.5 Discuss any deviations from test procedure or inadvertent happenings and possible impact on results, for example, power outages, power fluctuations, etc. (for longer test, in particular).

12.7 *Analytical Methods*—Identify the analytical techniques and instruments that were used by manufacturer, model, and serial number.

12.7.1 Identify the pH meter and probe used and the buffer solutions used for calibration.

12.7.2 Identify the methods used for solution and isotope analyses, including the use of calibration and control samples. Provide estimates of the detection and quantitation limits for components used to assess the test response.

12.7.3 Identify all other analyses performed, if any. For example, dissolved oxygen measurement, identification of bacteria, measurement of anion concentrations, analyses of colloid content, serial filtration, etc.

12.8 *Test Execution Data*—Report all data related to performing the test, including test initiation and termination data, the oven temperature at test initiation and test termination, any deviations from the test temperature.

12.8.1 Report the initial and final masses of the test specimen and the assembled test vessel, and the calculated mass changes.

12.8.2 Report any anomalies associated with test termination. For example, if the test vessel was tipped over.

12.8.3 Discuss how mass loss was taken into account when interpreting the test results.

12.8.4 Report general observations regarding the leachate and test specimen. Note if suspended solids were observed in the leachate or solids in the bottom of the vessel, if the leachate was discolored, the presence of a film or fines on the surface of the leachate, etc.

12.8.5 If solids were observed, describe method used to remove solids from the leachate. Discuss how the solids were accounted for when interpreting the test results.

12.9 *Leachant Analysis*—Include the identification number of each batch of leachant, date of makeup, date of analysis, and tabulation of analytical results for all elements, isotopes, ions, and organics that were analyzed.

12.9.1 If the leachant is a natural aqueous solution, report the date, place, and stratigraphy from which it was obtained.

12.9.2 For all leachants, report the chemical composition, Eh (if measured), pH, and date of analysis.

12.9.3 The detection limits for each analysis must be reported. When the result for an analyte is below detection limits, it may be left blank or reported as not detected with the detection limit provided in parenthesis.

12.10 *Analyses of Test and Blank Solutions*—Analyses are performed for leachates, blank test, and acid strip solutions.

12.10.1 Report the measured pH value for leachates and blank test solutions.

12.10.2 Report the volume of the leachate and blank test solutions at test termination as calculated from the test assembly data and the final vessel mass and the solution density.

12.10.3 Provide the as-measured solution concentrations and the values adjusted for all dilutions that were made prior to analysis.

12.10.4 Identify background concentrations for each element based on the results of blank tests.

12.10.5 Tabulate analytical results for all elements, isotopes, ions, and organics analyzed in the leachates blank, and acid strip solutions.

12.10.6 Report detection limits for all analyses. When the result for an analyte is below detection limits, it may be left blank or reported as not detected with the detection limit provided in parenthesis.

12.10.7 Report background-corrected concentrations used for leachates at each test duration.

12.11 *Analyzed Data*—Data analyses include determination of background concentrations, computation of dissolved masses, and computation of normalized elemental mass loss values.

12.11.1 Report the normalized mass loss values calculated with the background-corrected concentrations of key waste form components or the activities of radioactive waste components for each test.

12.11.2 Clearly identify the test conditions for each test, including the leachant, S/V ratio, test duration, and temperature, and the background concentrations used to calculate the normalized elemental mass loss values for each component.

12.12 *Uncertainty*—The combined uncertainty in the results due to test execution and solution analyses should be addressed by using the propagation of errors method or, preferably, that developed by the International Committee for Weights and Measured (CIPM) and promulgated by NIST¹⁰ (see [Appendix X4](#)).

12.12.1 The standard deviation of the normalized mass-loss values is computed by a propagation of errors method to include the uncertainty in the concentration values, the mass fraction value f_i , and the surface area.

12.12.2 Replicate tests can be used to estimate the within-day and between-day components of variance, the control-chart limits (see [12.14](#)), and a relative bias estimate for the data (see [Section 13](#)).

12.13 *Post-Leach Specimen Analyses (Optional)*—Discuss the results of analyses performed to characterize reacted test specimens. In particular, discuss how the results of surface analyses correlate with the leachate analyses and the implications concerning leaching mechanisms. While surface analyses of the test specimens prior to testing are not required as part of the method, they would be useful for comparison to the reacted specimens to identify alteration.

12.14 *Control Charts (Optional)*—Control charts are useful for establishing the precision with which the test can be conducted when developing a waste form composition, establishing process control limits, and determining the capabilities of the testing and analytical laboratories. They are also useful for identifying elements that provide the best measures of the material response (for example, as normalized elemental mass loss) for tracking process control or monitoring waste form performance.

13. Precision and Bias

13.1 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. However, the within and between laboratory precisions achievable for 28-day tests has been measured in several interlaboratory (round-robin) studies.

13.2 The Materials Characterization Center (MCC) at Pacific Northwest Laboratory 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 test method is applied using several glasses, leachant solutions, and temperatures. The results of this study are reported by Johnston and Daniel.¹¹ (Results are summarized in [Appendix X5](#)). By comparison with results obtained since this round robin, it has been demonstrated that experience in applying the test method (or similar test methods) has a significant effect on both the between laboratory and within laboratory precision. The data from inexperienced laboratories that participated in this round robin is believed by the MCC to

¹⁰ NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Technical Note 1297 (1994).

¹¹ 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 4 Comparison of NIST and MCC Test Results

	NIST		MCC		% Relative Difference ^A	95 % Confidence Interval for % Relative Difference	$\frac{s(\text{MCC})}{s(\text{NIST})}$
	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 $(1 - X_{\text{MCC}}/X_{\text{NIST}}) \times 100$

have resulted in over-estimation of both the between laboratory and within laboratory standard deviations.

13.3 Another data set is available from the results of an interlaboratory round robin test initiated and supported by the Commission of European Communities (CEC), wherein the MCC-2 static leach test method¹¹ was applied to a simulated high-level radioactive waste glass, designated UK-209, 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 represent the expected precision of this static leach test method. The between laboratory precision for major glass components B and Si were 19 % relative standard deviation. The precision for Cs and Sr, which are present in the glass and leachates at lower concentrations, were 31 and 40 %, respectively. (Results are summarized in [Appendix X5](#).)

13.4 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.

13.4.1 The results are summarized in [Table 4](#). The differences between unknown true means for tests conducted at the

MCC and NIST are 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 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. The percent relative differences for the remaining elements range from –6.5 to 4.7, and the confidence intervals indicate that the true differences probably range from –11.3 to 7.4 %.

13.4.2 When compared with the between-laboratories percent relative standard deviation from the round robin (see [Appendix X5](#)), differences in this range are not unexpected.

13.4.3 The ratio of the sample standard deviations ranges from 0.62 to 1.43. A ratio would have to be less than 0.41 or greater than 2.4 to be statistically significant at the 95 % confidence level. Thus, there are no significant differences in the level of precision achieved at the two laboratories.

13.5 The precision of the test for test durations longer than 28 days has not been established.

14. Keywords

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

APPENDIXES

(Nonmandatory Information)

X1. pH MEASUREMENTS OF BRINE SOLUTIONS

X1.1 Measurement of the pH in concentrated brines using standard glass electrodes is complicated by two principal factors: (1) a significant liquid junction potential is developed and (2) significant differences exist between the hydrogen ion concentrations and the activities. Both effects usually result in the measured pH value being significantly lower than the actual value.

X1.1.1 A potential at the junction between the reference electrode filling solution and the sample solution (“liquid junction potential”) occurs 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. The ionic strength of these buffer solutions are much lower than those of concentrated brines so that a significantly different liquid junction potential occurs between the electrode and brine. This usually

results in a measured pH value that is one or more pH units lower than the actual value.

X1.1.2 Changes in ion activity coefficients as a function of ionic strength can also be important in pH measurements. (Activity is equal to the product of concentration and the activity coefficient.) The values of activity coefficients are usually unity in dilute solutions, but average cation and anion activity coefficients can be much larger than unity in solutions of high ionic strength (for example, greater than 10 for HCl in saturated magnesium chloride solutions). Because glass electrodes respond to ion activities, not concentrations, there can be a large effect of ionic strength on the measured pH value. Accounting for the activity coefficient on the measured pH is complicated by a lack of activity coefficient data for concentrated brines and a similar lack of theoretical models applicable to such solutions.

X1.2 Considerable caution must be exercised, therefore, when attempting to interpret brine pH measurements. The effects of the liquid junction and ion activity coefficients will

result in measured pH values being shifted significantly lower from the true pH (based on H^+ ion activities). However, for a given brine system, these contributions should be constant for a large range of pH values, such that trends in the pH will be unaffected. The bulk composition of the brine solution is not

likely to change significantly under most test conditions so that trends in the measured pH are expected to represent the trends in the actual pH values during testing. If significant changes do occur in the brine composition for a series of experiments, even differences in measured pH values may be suspect.

X2. PREPARING TEST SPECIMENS USING A LOW-SPEED SAW

X2.1 Read the operator's manual provided with the saw. Become familiar with basic wafer blade sectioning technique.

X2.1.1 Fill the lubricant pan with water or other appropriate cutting fluid to a level that will immerse the blade approximately 0.6 cm. The specimen basket will not be used with a 5-in. diameter blade.

X2.1.2 Ensure that the blade is mounted securely using the 2½-in. diameter recessed flanges provided with the saw.

X2.1.3 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 to remove cutting debris from the abrasive. 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 2 mm with no nicks or dents.

X2.1.4 Secure the sample firmly in the chuck to prevent inaccurate cuts and damage to the blade that could result from movement during sectioning.

X2.1.5 Position the counterbalance weight after the chuck and the sample are mounted on the arm so that the arm is balanced.

X2.1.6 Set the blade speed so as to cut the material without binding (too slow) or losing contact (too fast) with the material. Appropriate settings to cut most waste glasses are 7 for a 5-in. blade and 8.5 for a 4-in. blade.

X2.1.7 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. **Caution**—Do not use more than 300-g weight on the arm as blade distortion may occur.

X2.1.8 Keep the sample in contact with the blade and do not turn the saw off during the cutting process. Add lubricant to the pan as needed to lubricate the blade during the cutting process. When approaching the end of a cut, support the piece being cut from the bulk material in order to prevent it from breaking off. A small tag of material will remain on the chucked piece.

X2.1.9 Replace the lubricant fluid after every 2 h (and after the cut has been completed if the cut takes longer than 2 h).

X2.1.10 The width of the diamond matrix at the rim is approximately $\frac{5}{32}$ -in on a new blade. When width becomes less than $\frac{1}{32}$ in., the blade should be replaced.

X3. METHOD TO DETERMINE SURFACE AREA OF THE IRREGULAR FACE OF A TABLET TEST SPECIMENS

X3.1 Make an enlarged drawing of the specimen face on paper using the measured dimensions (this is why a diagonal measurement is needed in step 8.8.2). For example, if a particular specimen dimension is 9 mm, make the drawn dimension 9 in. As an example, consider a four-sided planar surface shown in Fig. X3.1(a) having corners a, b, c, and d. The lengths of sides ab, bc, cd, and ad and the diagonal ac have been measured.

X3.2 Generate an enlarged drawing of the surface. As an example, a sketch of the specimen in Fig. X3.1(a) is shown in Fig. X3.1(b). Draw the longest edge first, from A to B in Fig. X3.1(b). Use a compass to draw an arc proportional to the length of the diagonal AC from point A and an arc proportional to the length of side BC from point B. Where the arcs intersect locates point C. Use a compass to draw an arc from point C

proportional to the length of side CD and an arc from point A proportional to the length of side AD. Where the arcs intersect locates point D. Verify the accuracy of the drawing by measuring the length of side CD.

X3.3 Draw a square representing 10×10 mm using the same factor (that is, 10×10 in.) on the same type of paper used to sketch the sample face. Cut out the 10×10 square and the drawing of the sample face and weigh both. Calculate the area/mass ratio from the mass of the 10×10 square. Calculate the area of the sample face by multiplying the mass by the area/mass ratio determined by weighing the square.

X3.4 Increasing the expansion factor of the drawn specimens will reduce the uncertainty in the calculated surface areas.

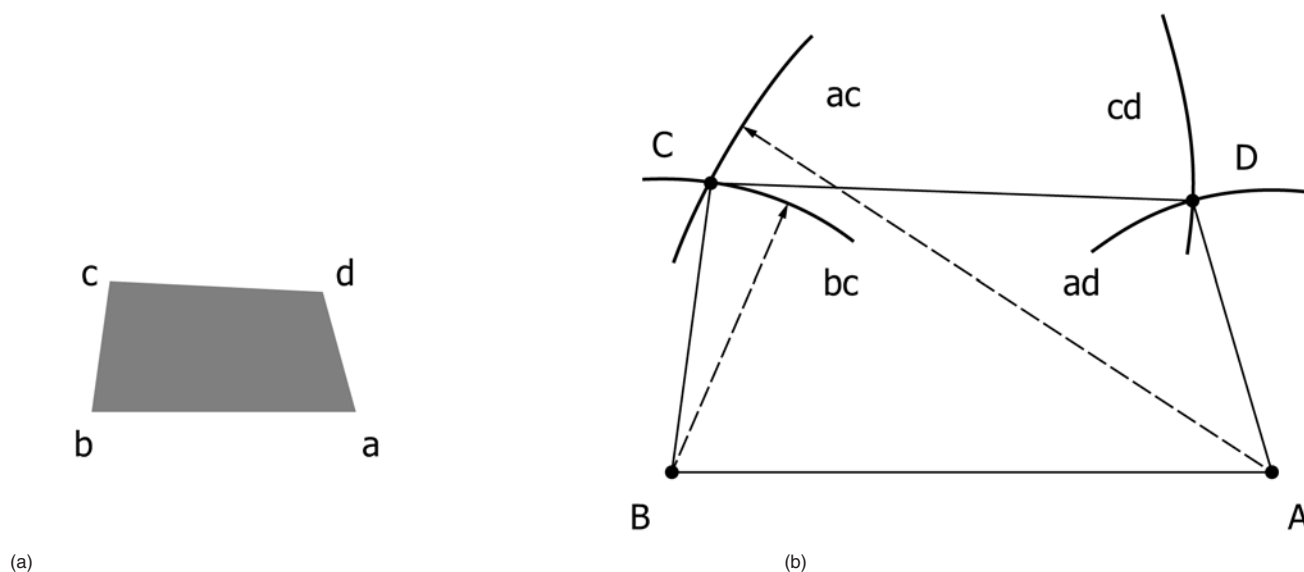


FIG. X3.1 Example of (a) Sample Face and (b) Enlarged Sketch of Sample Face

X4. UNCERTAINTY

X4.1 It is recommended that the contributions of the parameters used to calculate a value of interest (for example, S , NL) be combined to estimate the uncertainty in the calculated value following the recommendations in NIST Guidelines.¹⁰

X4.1.1 Each component used in a calculation contributes a standard uncertainty u_i to the result. The uncertainty in a parameter may be due to a single measurement or the propagation of several measurements.

X4.1.2 The parameters contributing to various values calculated within this standard are summarized in Table X4.1. The variables used to represent the parameters in this standard are included for convenience. Values of u_i are usually represented as a relative standard deviation and are assigned based on experimental variables.

X4.2 The estimated standard deviation of a result y is represented by the combined standard uncertainties of the

measurements that contribute to the result u_c . The combined standard uncertainty the measurand y , $u_c(y)$, is calculated by propagating the uncertainties using the root-sum-of-squares method.

X4.2.1 For example, the surface area of a 2.5×2.5 cm right cylinder is 29.45 cm^2 . If the diameter and height are measured to a precision of 0.01 cm , then the standard uncertainty in the surface area is 0.176 cm^2 , and the relative standard deviation is 0.596% .

X4.3 The combined standard uncertainty can be multiplied by a coverage factor, k , to represent an uncertainty range at a given confidence level. The value of k varies with the confidence level; for example, $k=2$ represents the 95.45 % confidence level. The product of k and $u_c(y)$ gives the expanded uncertainty U about the measurand y , such that any measured value Y is expected to lie within the range $y-U \leq Y \leq y+U$ at the

TABLE X4.1 Contributions to Uncertainties in Parameters and Calculated Values

Parameter	Variable	u_i	Main Contributors to Uncertainty
Source term in specimen	A°		Analytical uncertainty, homogeneity of source material
Specimen radius	R		Measured dimensions (half of measured diameter)
Specimen height	H		Measured dimensions
Specimen surface area	S		Measured dimensions (diameter and height); geometry
Volume of specimen	V		Measured dimensions (diameter and height); geometry
Concentration in sample	a_i		Analytical uncertainty
Volume in leachant	...		Density (if leachant volume determined by weight)
Mass of leachant	...		Measurement accuracy (if leachant volume determined by weight)
Volume or mass of counting sample	...		Measurement accuracy
Source term multiplication factor	...		Measurement accuracy, analytical uncertainty
Normalized elemental mass loss	NL		Measured concentration in leachate, volume leachate, surface area and mass analytical sample, source term
Time (interval or cumulative)	t		Precision of interval time; fraction of time at temperature
Temperature	T		Thermometer reading, thermocouple calibration

confidence level used to calculate U .

X4.4 The combined standard uncertainty does not include systematic uncertainties such as laboratory or operator bias. It may be possible to estimate systematic uncertainties based on

previous measurements in the same or other laboratory, experience with the behavior of similar materials, literature reports, and manufacturer's specifications.

X5. ADDITIONAL DATA ON THE PRECISION OF THE TEST METHOD FROM ROUND-ROBIN DATA

X5.1 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.

X5.2 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.¹¹

X5.3 The Commission of European Communities (CEC) initiated and supported an inter-laboratory round-robin test with the MCC-2 test method.¹² 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.

X5.4 For comparison purposes, Table X5.1 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/m²) 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 X5.1 are for normalized elemental mass loss values calculated using the of B, Cs, Si, and Sr concentrations measured in the leachates from the tests performed for 28 days at 90°C in deionized water.

X5.5 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

¹² 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 X5.1 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.

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.

X5.6 For each study and each element, **Table X5.1** displays the range of concentration values (in ppm) observed in the study and the average normalized elemental mass loss in g/m². 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).

X5.7 Comparison of the MCC round-robin results and the CEC round-robin results in **Table X5.1** 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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