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

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

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

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

~~1.5~~

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](#)

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, 2004; 2010. Published July 2004; 2010. Originally approved in 1992. Last previous edition approved in 1998; 2004 as C1220 - 98 (2004). DOI: 10.1520/C1220-98R04.10.1520/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.

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

D1125 Test Methods for Electrical Conductivity and Resistivity of Water

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods—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~~ 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~~—the closeness of agreement between the accepted reference value and individual results (Practice E177).

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

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

3.1.4 ~~bias of a measurement process, n~~—a generic concept related to 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 (Practice E177). ~~measured.~~

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

3.1.5.1 ~~Discussion~~—The response of glass, ceramics, or cermets under other conditions is outside the scope of this test method. ~~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~~—a system that precludes the transport of matter into or out of the system. ~~closed system, n~~—a system utilizing a test container that is impervious to material transport.

3.1.7 ~~high-purity water~~—ASTM Type I or Type II water with a maximum total matter content of 0.1 g/m³, a minimum electrical resistivity of 16.67 MΩ·cm at 25°C, and no detectable soluble silica (see Specification D1193 and Terminology D1129). ~~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 ~~ion selective electrode (ISE)~~—a device for measuring F⁻. ~~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~~—a solution used, or intended for use, in leaching. ~~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~~—the solution resulting from a leach test. ~~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~~—the action of removing soluble constituents from a solid into a solution. ~~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 specimens~~ specimen, *n*—specimens that are physically one coherent piece, as opposed to powdered specimens that consist of many small pieces of irregular configuration. ~~MA monolithic specimens~~ may consist of several individual phases, but they must be bound in a stable coherent configuration.

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

3.1.14 ~~open system~~—a system that permits the transport of matter into or out of the system, e.g., O₂ and/or CO₂. ~~open system, n~~—a system utilizing a test container through which material transport is possible, for example, O₂ and/or CO₂, diffusion into or out of the system. ~~diffusion, or both.~~

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

3.1.16 ~~precision of a measurement process~~—a generic concept related to the closeness of agreement between test results obtained under prescribed like conditions from the measurement process being evaluated (Practice E177). In this test method, precision will be measured by either standard deviation or relative standard deviation. ~~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 SW846A, 3rd Ed., Revision 1, U.S. Environmental Protection Agency, Washington, DC, December 1987.

3.2 Abbreviations:

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

3.2.2 ~~ICP—inductively coupled plasma. ISE—ion selective electrode.~~

3.2.3 ~~PFA—perfluoroalkoxy. PTFE—polytetrafluoroethylene.~~

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

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

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

4. Summary of Test Method

4.1 ~~Specimens of known volume and geometric surface area are immersed in the reference leachants without agitation for defined time periods at defined temperatures. The S/V ratio is held constant within 0.5 of 10.0 m⁻¹. Three reference temperatures, 40°, 70°, and 90°C, and a number of specific time periods are identified in a series of test matrices established to meet objectives that include evaluation of waste forms for comparative purposes. In the test method, three reference leachants are used: high-purity water and two solutions (silicate/bicarbonate and brine) that approximate fluids that the waste form may encounter in a geologic repository. In addition to the reference leachants, others may be used. The test is for application to simulated waste forms and to radioactive specimens.~~

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

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

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

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 is intended principally to distinguish differences in the leaching behavior of candidate monolithic, inorganic, radioactive waste forms under low S/V ratio conditions. The test method can be used to produce altered solid specimens to study the reaction of monolithic waste forms during static exposure to solutions. Data from this test may form part of the larger body of data that is necessary in the logical approach to long-term prediction of waste form behavior, as described in Practice~~

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, solution concentrations and characterization and altered surfaces may be used in the testing of geochemical modelling codes. This test method excludes study of powdered or organic materials. This test method can be used as either a “characterization” or “accelerated” test under the protocol of Practice C1174, mentioned above.~~

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

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

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

the scope of this test method. Preparation of deaerated leachants is covered in 7.2.2. 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.

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

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 10⁴ rad, is not significant. Fluoride, at the concentrations encountered in tests where the radiation dose is limited to less than 10⁴ rad and qualified PTFE is used, has not been demonstrated to have an effect on leaching behavior.⁴ The primary reason for limiting the integrated dose to PTFE vessels and specimen supports to 10⁴ rad and requiring that the PTFE vessels be qualified is to ensure that excessive fluoride releases do not occur (see Note 1). In order to monitor fluoride releases, which could have an influence on test results, analysis for F⁻ concentration is a test requirement.

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 leach volume, for example with a monofilament or by use of a coarsely woven support screen. The specimen surface-area-to-leachant-volume (S/V) ratio must be within 0.5 of 10.0 m⁻¹.

6.2 *Test Vessel Material*—The choice of material for construction of the test vessels will depend on the radiation field. When testing is performed in fields that yield an absorbed dose of less than 10⁴ rad, PTFE vessels are preferred. 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.

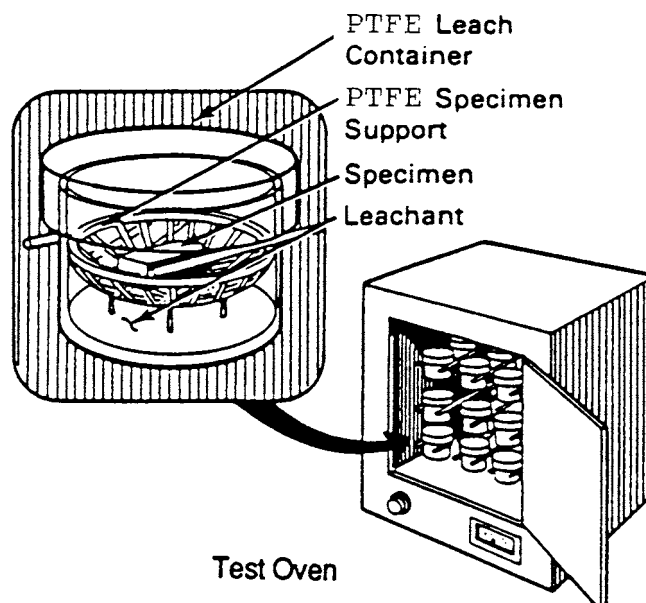


FIG. 1 Example Apparatus for Static Leach Test Method

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, use PTFE PFA vessels that have been qualified (see rad, PTFE vessels shall be qualified for use (see 6.4):

6.2.1 For fields where the absorbed dose exceeds 10^4 rad, 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, use fused silica or 304L stainless steel. If the vessel dose exceeds 10^4 rad (100 Gy). Doses below 10^4 rad and the brine leachant is employed, fused silica vessels are to be used (because of the corrosion of stainless steel by the brine). Use of the same vessel material throughout the test matrix will allow an evaluation of the contribution to the leachate by the vessel, for example, silicon from fused silica. (See Note 1.)

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

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

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

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

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

6.4.2 *Record of Vessel Cleaning History*—Each batch of cleaned vessels shall be labeled with a unique batch number. A log book of the leach vessel number and date of cleaning shall be kept. The date can be used as the batch number identifier if only one batch

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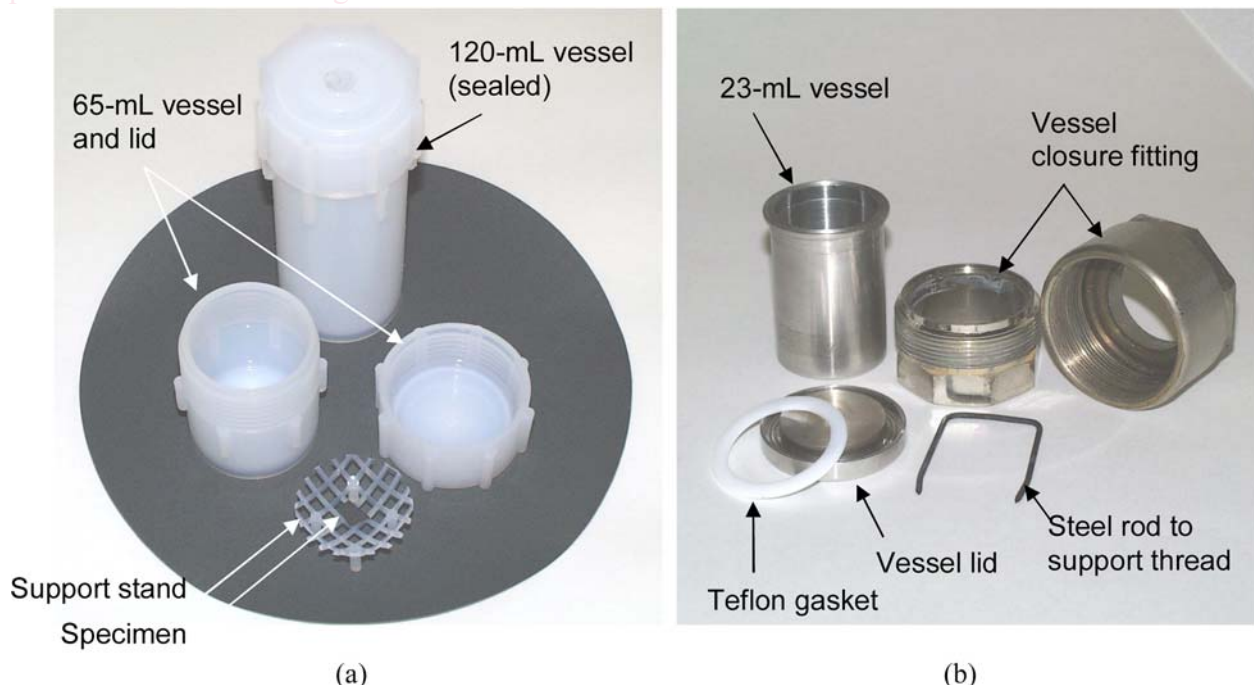


FIG. 2 Photograph of (a) PTFE Teflon Vessel and Support and (b) Type 304L Steel Test Vessel, Support, and Closure Fitting

has been cleaned on that date. Alternatively, a separate batch number can be assigned and recorded. In this manner, any inconsistent test responses might be traced to improper cleaning of a batch of vessels or to a problem vessel.

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

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

6.6 Preparation of Vessel—New PTFE vessels and supports must be cleaned to reduce the amount of F⁻ released during testing. This cleaning is done by a special procedure using a NaOH solution, described in 6.6.1. PTFE vessels can be reused after testing provided they are cleaned before reuse. However, reuse of PTFE test vessels and supports is not allowed in this test method when these components have been used in tests with actinide-doped specimens. This is because actinides are difficult to remove, and may not be sufficiently removed from the PTFE vessel walls by leachate acidification and the vessel/specimen support structure cleaning procedure. As these dopants may be present in very low concentrations in a leachate, contamination from the vessel walls could be potentially significant. New PTFE test vessels and supports, except fine monofilaments, shall be heated in a 200 ± 10°C oven for one week prior to cleaning. Both new and used PTFE vessels and PTFE specimen supports, except for fine filaments, must be cleaned according to the specified procedures. Stainless steel and fused silica vessels are cleaned according to different procedures, described in 6.6.3 rad have been shown to not damage PTFE.⁴ The total absorbed dose of each PTFE test vessel may not exceed 10⁴ 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 10⁴ 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 <10⁴ 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 10⁴ 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 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

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

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.

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

6.4.1.1 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 $\mu\text{g/ml}$, 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 $\mu\text{g/ml}$, 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.

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

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

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

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

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

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

6.6.1.6 Remove lids carefully and dispose of NaOH solution.

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

6.6.1.8 Repeat 6.6.1.7

6.5.1 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.

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

6.6.1.10 Repeat 6.6.1.7 and 6.6.1.9

6.5.12 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.

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

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

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

- 6.6.1.14 Remove vessels and allow to cool to room temperature.
- 6.6.1.15 Take an aliquot of liquid from each vessel and measure pH.
- 6.6.1.16 If pH is below 5, repeat 6.6.1.1-6.6.1.16 until pH is above 5.
- 6.6.1.17 If pH is above 7, repeat 6.6.1.7-6.6.1.17
- 6.5.19 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 µg/mL, repeat steps 6.5.8-6.5.19.
- 6.6.1.18 If pH is between 5.0 and 7.0 take a second aliquot and send for ISE F⁻.
- 6.6.1.19 If ISE F⁻ level is >0.5 µg/mL, repeat 6.6.1.7-6.6.1.19. If ISE F⁻ is still >0.5 µg/mL repeat 6.6.1.1-6.6.1.19
- 6.5.21 If the F⁻ is still >0.5 µg/mL after performing steps 6.5.8-6.5.19 twice, repeat steps 6.5.1-6.5.19.
- 6.6.1.20 If ISE F⁻ level is <0.5 µg/mL, a vessel is acceptable for use.
- ~~6.6.2 Used Vessels—Clean used PTFE vessels and supports (that is, vessels and supports that were cleaned according to 6.6.1.1-6.6.1.20 and then used for testing) according to 6.6.2.1-6.6.2.8~~
- 6.5.22 If the F⁻ concentration is <0.5 µg/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.
- ~~6.6.2.1~~
- 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 ± 10°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 ± 10°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.
- ~~6.6.2.2 Soak~~ 6.7.2 Soak vessels and supports for 1 h in 0.16 M HNO₃ (1 wt % HNO₃) at 90 ± 10°C.
- ~~6.6.2.3 Rinse again as specified in 6.6.2.1~~
- 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.
- ~~6.6.2.4~~
- 6.7.5 Soak vessels and supports for 1 h in high-purity water at 90 ± 10°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 ± 2°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 µg/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 ± 10°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 HNO₃ (1 wt % HNO₃) at 90 ± 10°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 ± 10°C.
- ~~6.6.2.5 Fill the vessels approximately 90% full with fresh high-purity water with support in place. Close the lids and hold for at least 16 h at 90 ± 2°C and then measure the pH of the water from each vessel. Take an aliquot of the water from at least two vessels from each vessel batch and submit for ISE F⁻.~~
- ~~6.6.2.6 Repeat 6.6.2.4 and 6.6.2.5 until the pH is in the range of 5.0 to 7.0 and the F⁻ is <0.5 µg/mL.~~
- ~~6.6.2.7 If the pH and fluoride requirements cannot be achieved by three repetitions of 6.6.2.4 and 6.6.2.5, then repeat the cleaning procedure starting at 6.6.2.1~~
- 6.8.5 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 ± 2°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.
- ~~6.6.2.8 Dry vessels and lids at 90 ± 10°C for a minimum of 16 h and store inside a clean environment until used.~~
- 6.6.3 Stainless Steel and Fused Silica Vessels—The procedures in 6.6.1 and 6.6.2 are specifically for PTFE vessels. When using

other inert vessels, such as fused silica or 304L stainless steel, variations of these procedures are appropriate.

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

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

6.6.3.3 Rinse three times in high-purity water.

6.6.3.4 Submerge in 0.16 M HNO₃

6.8.10 Dry vessels and lids at 90 ± 10°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.

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

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

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

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

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

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

6.9.4 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 aliquot 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.

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

6.6.3.12 Dry the vessels in a 90 ± 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.

6.6.4 *Cleaning of Used Stainless Steel and Fused Silica Vessels*—When stainless steel or fused silica vessels are reused subsequent to their use with radioactive specimens, residual contamination may be present. The vessels shall be cleaned before reuse using 0.16 M HNO₃

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.

6.6.4.16.10.1 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). Reseat/Seal the vessel and place in an oven at 90 ± 2°C to digest for a minimum of 16 h to acid-strip any dissolve radionuclides adhering to the interior of the vessel.

6.6.4.2Check/6.10.3 Check the acid-stripped resulting solution for radioactivity. Repeat step 6.6.4.16.10.2 until the radioactivity of the acid-strip solution is reduced to below the background levels.

6.6.4.3Remove/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.

6.6.4.4Fill/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 6.6.5.6.6) and place in oven at 90 ± 2°C for at least 24 h.

6.6.4.5Remove/6.10.6 Remove vessels from oven, then take an aliquot/aliquant of the water and measure the pH.

6.10.7 Take another aliquot/aliquant and measure the radioactivity.

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

~~6.6.4.6~~6.10.9 If the pH is not in the range of 5.0 to 7.0 ~~or 7.0~~, the measured radioactivity is not at the background level, or Si ≥ 1 ppm is detected for stainless steel vessels, repeat steps ~~6.6.4.3-6.6.4.5~~6.10.2-6.10.8.

~~6.6.4.7~~6.10.10 If three repetitions of steps ~~6.6.4.3-6.6.4.5~~6.10.2-6.10.8 do not result in a pH within the range of 5.0 to 7.0, low radioactivity, radioactivity below detection, and Si < 1 ppm for stainless steel vessels, then repeat the cleaning starting at step ~~6.6.4.16~~6.10.1.

~~6.6.4.8~~ Dry vessels, lids, and gaskets at $90 \pm 2^\circ\text{C}$ for a minimum of 16 h and store in a clean environment until needed.

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

~~6.6.5.1~~ Handle the gaskets only with clean tongs.

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

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

~~6.6.5.4~~ Bake each gasket in an oven at $200 \pm 10^\circ\text{C}$ for a minimum of 4 h.

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

~~6.6.5.6~~ Dry gaskets at $90 \pm 10^\circ\text{C}$ for a minimum of 16 h and store in a clean environment until needed.

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

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

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

~~6.9~~

~~6.10.11~~ Dry vessels, lids, and gaskets at $90 \pm 2^\circ\text{C}$ 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:

~~6.13~~Volume Measurement—Measure leachant volumes gravimetrically or with pipettes, burettes, or flasks calibrated as described in Table 1 (see also ~~6.10~~) and accurate to within 1% or better.

~~6.10~~

~~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 % of the reference solution when checked by individual measurements on reference solutions, this may not be possible when concentrations in the solution approach 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 Test Methods Practice C1109 or EPA SW846a SW-846A, or both), direct current plasma spectroscopy, atomic absorption emission spectroscopy, and neutron activation. Selection of a specific technique depends on specific test objectives and the particular solutes of interest. For radioactive elements such as actinides and fission products, where low small amounts may be of interest, radiochemistry/radiation counting may be needed or desirable. Use of

~~6.14.3~~ Analyzing blanks and simulated leachates, as discussed in Section 10, help leachates with test solutions helps ensure that high-quality data are obtained.

~~6.11~~6.15 pH Measurement—Measure the pH to an accuracy of 0.1 unit using a calibrated meter. Use Test Methods—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 and commercial buffers to make this measurement. When measuring the pH of deaerated solutions, make the measurement under an argon atmosphere.

~~6.12~~pH Measurement in Brines:

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
pH	pH meter	before use with commercial or NIST buffer solutions, and at intervals of 60 min during measurements
Volume	volumetric flasks	See Test Methods D1293 and 6.11 for guidance 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

TABLE 1 Required Accuracy for Mass Determination Schedules

Measurement	Device	Frequency
Temperature	thermocouple or thermometer	6 months
Temperature	Chemical reagent	6 months used to prepare leachant
Temperature	electronics or temperature probe (without sensor)	6 months against a calibrated millivolt source
Length	within 1 % of micrometer	6 months the reagent mass
Test specimens	micrometer	6 months standard foils, gage blocks
Mass	balance	3 months standard foils, gage blocks
Chemical concentration	analytical method	3 months NIST standard masses
Chemical concentration	analytical method	3 months NIST standards, where possible, 2 times daily (routine), secondary standards
pH	pH meter	3 months NIST standards, where possible, 2 times daily (routine), secondary standards
pH	pH meter	each day of use with commercial buffer solutions at intervals of 30 to 60 min during measurements
Volume	pH meter volumetric flasks	each 0.5 mg guidance use certified flasks
Volume	volumetric flasks	3 months by measuring the mass of pure water contained
Activity	pipettes counting techniques	3 months by measuring the mass of pure water transferred twice a day, before and after counting NIST or NIST-traceable standard isotope source of interest

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

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

6.12.3 In addition, changes in ion activity coefficients as a function of ionic strength can be important in pH measurements. (Activity is equal to the product of concentration and the activity coefficient.) Activity coefficients are usually unity in dilute solutions. However, in solutions of high ionic strength, average cation-anion activity coefficients can be much larger than unity (for example, greater than 10 for HCl in saturated magnesium chloride solutions). Because glass electrodes respond to ion activities,