Designation: C1220 − 21

Standard Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste[1]

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 °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 °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. Other units of measurement are included for reference only, with the following exceptions:

1.8.1 Grit size used in this standard can be converted to the corresponding μm values using the current revision of Guide E3.

1.8.2 Appendix X2 describes the usage of a model of saw for which components and instruments are imperial unit based; imperial units are used in this section.

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

1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials
C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy
C1174 Guide for Evaluation of Long-Term Behavior of Materials 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
E3 Guide for Preparation of Metallographic Specimens

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 *EPA Standard:*[3]
SW-846A Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

3. Terminology

3.1 Refer to Terminology C859 for additional terminology that may not be listed below.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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.

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

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

3.2.4 *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.

3.2.5 *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.

3.2.6 *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.

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

3.2.8 *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.

3.3 *Abbreviations:*

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

3.3.2 *ISE*—ion selective electrode.

3.3.3 *PTFE*—polytetrafluoroethylene.

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

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

3.3.6 *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. Aliquots 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.

[3] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

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 100 Gy (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 polytetrafluoroethylene (PTFE) 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.

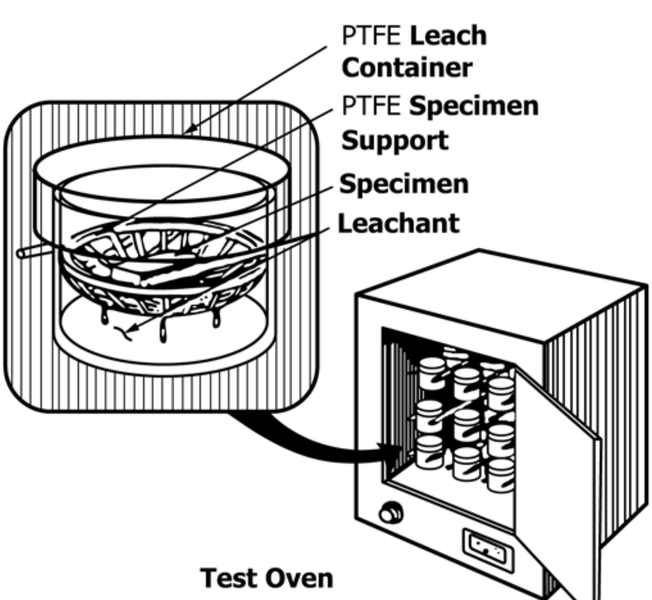

FIG. 1 Example Apparatus for Static Leach Test Method

6.3 *Test Vessel and Specimen Support*—Steel, titanium, fused silica, or PTFE 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 100 Gy (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 100 Gy. Doses below 100 Gy have been shown to not damage PTFE.[4] The total absorbed dose of each PTFE test vessel may not exceed 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 100 Gy 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 <100 Gy 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. PTFE vessels are pervious to carbon dioxide, which could affect the solution pH, and some water loss may occur. The use of PTFE 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 100 Gy, 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.

[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.