ASTM INTERNATIONAL

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Standard Test Method for
Accelerated Leach Test for Measuring Contaminant Releases From Solidified Waste[1]

This standard is issued under the fixed designation C1308; 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 procedure for measuring the release rates of elements from a solidified matrix material under conditions that mitigate solution feedback effects. Results can be analyzed by using different models to determine if the elemental releases are controlled by mass transport though the matrix (that is, by diffusion), by a surface dissolution process, or by a combination of processes. This test method is applicable to any material that does not deform during the test.

1.1.1 If mass diffusion is the dominant process in the release mechanism, then the results of this test can be used to derive diffusion coefficients for use in diffusion-based mathematical models.

1.1.2 If surface dissolution is the dominant process, then the results of this test can be used to derive the kinetic dissolution rate in the absence of reaction affinity effects for use in dissolution-based mathematical models.

1.1.3 If release is controlled by coupled or combined dissolution and mass transport processes, then the results of this test can be used to derive effective coefficient values for a mechanistic or empirical model.

1.2 Tests at elevated temperatures are used to accelerate the release process to determine the temperature range over which the release mechanism does not change and to generate results that can be used for calculating releases at lower temperatures over long times, provided that the release mechanism does not change with temperature.

1.2.1 Tests conducted at high temperatures can be used to determine the temperature dependence of model coefficients.

1.2.2 The mechanism is considered to remain unchanged over a range of temperatures if the model coefficients show Arrhenius behavior over that range.

1.2.3 Releases at any temperature within that range can be projected in time up to the highest cumulative fractional release value that has been measured for that material.

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

1.4 *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.*

1.5 *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]

C1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste

3. Terminology

3.1 *Definitions:*

3.1.1 *cumulative fraction leached*—the sum of the fractions of a species leached during all sampling intervals prior to and including the present interval divided by the amount of that species present in the test specimen before the test.

3.1.2 *diffusion coefficient (diffusivity)*—an intrinsic property of a species that relates *(1)* its concentration gradient to its flux in a given medium (Fick's first law), *(2)* its spatial rate of change in the direction of the concentration gradient to the time rate of change in its concentration in a given medium (Fick's second law), or *(3)* its mean square displacement to time in a given medium (the Einstein equation).

3.1.3 *dissolution*—the transfer of species from the solid test specimen into solution.

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

3.1.4 *effective diffusion coefficient (D$_e$)*—the diffusion coefficient as modified by other processes (for example, adsorption) or physical constraints (for example, tortuosity and constrictivity).

3.1.5 *finite cylinder (finite medium)*—a bounded body for which Fick's diffusion equation can be solved.

3.1.6 *incremental fraction leached*—the amount of a species leached during a single sampling interval divided by the amount of that species in the test specimen before the test.

3.1.7 *leachant*—the initial solution with which a solid is contacted and into which the solid dissolves or is leached.

3.1.8 *leachate*—the final solution resulting from a test in which a solid is contacted by a solution and leaches or dissolves.

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

3.1.10 *leaching interval*—the length of time during which a given volume of leachant is in contact with a specimen.

3.1.11 *leaching mechanism*—the set of processes that controls the rate of mass transport of a species out of a specimen during leaching.

3.1.12 *matrix material*—the solid material used to immobilize the waste or species of interest.

3.1.13 *partitioning*—the distribution of a contaminant between phases that may result in different release behaviors.

3.1.14 *reference leach test*—a leach test conducted with the material of interest under defined conditions, the results of which are used as a standard against which the results of other leach tests with that material are compared. In this test method, a reference leach test is one that is conducted at 20 °C using demineralized water.

3.1.15 *semi-dynamic leach test*—a leach test method in which the specimen is exposed to fresh leachant on a periodic schedule.

3.1.16 *semi-infinite medium*—a body having a single planar surface and extending infinitely in the directions parallel to the surface and in one direction normal to the surface.

3.1.17 *source term*—in this test method, the concentration of a species of interest in a specimen prior to leaching.

3.1.18 *specimen surface area*—for purposes of this test method, the geometric surface area of a monolithic specimen that is calculated from macroscopic measurements of its dimensions by assuming a simple geometric shape, such as a right circular cylinder.

3.1.19 *specimen volume*—for purposes of this test method, the volume of a monolithic specimen calculated from macroscopic measurements of its dimensions by assuming a simple geometric shape, such as a right circular cylinder.

3.1.20 *waste form*—the waste material and any encapsulating or stabilizing matrix in which it is incorporated.

4. Summary of Test Method

4.1 This test method is a semi-dynamic leach test in which a specimen of known surface area and volume is immersed in a leachant of known volume that is completely replaced after specified one-day intervals. The concentration of an element of interest in the test solution recovered after each interval is measured. The fraction of a species of interest that is released into solution during each test interval divided by the initial amount of that species in the specimen is referred to as the incremental fraction leached (*IFL*). The accumulated amount of the species of interest released during the intervals prior to and including the interval of interest is referred to as the cumulative fraction leached (*CFL*). Changes in the *IFL* and *CFL* values after each interval are analyzed to determine if the release from the solid can be described using an analytical model.

4.2 The results of tests at a single common temperature may be used to compare the release behaviors of different species from the same or different materials.

4.2.1 The derived model parameter values can be used to calculate releases from waste forms with shapes different than that of the test specimen.

4.2.2 The release model optimized by regression to test results can be used to calculate releases from full-scale waste forms for long times.

4.3 Elevated temperatures are used to accelerate the release of a species of interest, show that release is controlled by the same process over a temperature range, and determine the temperature dependence of the model coefficient values.

4.3.1 Tests must be performed at a minimum of three temperatures to verify that the leaching mechanism does not change over that temperature range.

4.3.2 By conducting tests over a range of temperatures, an Arrhenius plot can be produced to interpolate values of the test parameters within the temperature range that was tested. Values cannot be extrapolated to temperatures that are higher or lower than the temperature range spanned by the tests.

4.4 The *CFL* values of tests conducted at low temperatures can be extrapolated to long times if data from tests run at two or more elevated temperatures and at the reference temperature (20 °C) can all be represented by the same model and if the model coefficient values show Arrhenius behavior.

4.4.1 The results of tests at repository-relevant temperatures can be extrapolated to long times by utilizing results from tests at elevated temperatures if data from tests run at elevated temperatures and data from tests run at the reference temperature (20 °C) can be represented using the same model and the model coefficient values show Arrhenius behavior.

4.4.2 The maximum cumulative fractional release measured represents the maximum extent of reaction for which the consistency of the mechanism has been verified for that material.

4.5 The release model coefficients can be used to calculate releases from waste forms with other shapes and sizes.

4.6 The possibility of a solubility limit to the release of the species of interest shall be addressed. This can be done by evaluating the *IFL* values: constant *IFL* values may indicate either linear release kinetics or solubility limits. The results of additional tests with shorter or longer intervals can be used to discriminate between these possibilities.

4.7 If the data cannot be fit with a contaminant release model within the defined criterion, then graphical comparisons of the data are recommended for added insight: For example, a plot of the *CFL* values from a test conducted at an elevated temperature against the *CFL* values from a test conducted at the reference temperature can be used to verify that the data sets are consistent and that the tests at elevated temperatures appropriately accelerate the release, even though the release kinetics cannot be represented by an analytical model.

5. Significance and Use

5.1 This test method can be used to measure the release of a component from a solidified waste form into water at the reference temperature near 20 °C and at elevated temperatures that accelerate the rate and extent of leaching relative to the values measured at the reference temperature. Results of this test method can be used to quantify an intrinsic property of a material, but should not be presumed to represent releases in specific disposal environments. Tests can be conducted under conditions that represent a specific disposal environment (for example, by using a representative groundwater) to determine effective parameter values for those conditions.

5.2 This test method can be used to:

5.2.1 Compare releases of waste components from various types of solidification agents and formulations.

5.2.2 Determine the model parameter values quantifying the release of contaminants from a waste or waste form at a specific temperature.

5.2.3 Promote greater extents of reaction than can be achieved under expected service conditions within a laboratory time frame to provide greater confidence in modeled contaminant releases.

5.2.4 Determine the temperature dependence of contaminant release.

5.3 Fitting the experimental results with a mechanistic model allows release behaviors to be extrapolated to long times and to full-scale waste forms under the following constraints:

5.3.1 The same model must be used to represent the results of tests conducted at elevated temperatures and at the reference temperature because the mechanism must be the same.

5.3.2 Projections of releases over long times require that the waste form matrix remain stable, which may be demonstrated by the physical robustness of specimens recovered from tests conducted at elevated temperatures.

5.3.3 Extrapolations in time at any temperature within the range tested are limited to values that correspond to the maximum *CFL* value that was measured.

6. Apparatus

6.1 A forced-air environmental chamber or a circulating water bath capable of controlling leachant temperatures to within 1 °C of the target test temperature shall be used.

6.2 *Balance*—The balance shall be accurate to 0.1 % of the minimum test load.

7. Reagents and Materials

7.1 *Leachant*—The leachant can be selected with regard to the material being tested and the information that is desired.

ASTM Type I water, synthetic or actual groundwater, or chemical solutions can be used to evaluate release in the absence of external effects, represent a particular disposal environment, or measure specific solution feed-back effects, respectively. If the leachant contains a non-negligible amount of the contaminant of interest, blank tests should be conducted to provide background concentrations to calculate the amounts released from the solid during each test interval.

7.2 *Leaching Containers*—Leaching containers shall be made of a material that does not react with the leachant, leachate, or specimen. It is particularly important to select materials that are not susceptible to plate-out of species of interest from solution. High density polyethylene and polytetrafluoroethylene (PTFE) are suitable container materials. The top of the container shall fit tightly to minimize evaporation. The mass of the vessel must be checked before sampling to verify that evaporative losses are less than 1 % of the leachant mass over every test interval. This is especially important for tests at elevated temperatures.

7.3 *Specimen Supports*—Supports for the specimens shall be made of a material that does not react with the leachant, leachate, or test specimen and is not susceptible to plate-out. High density polyethylene and PTFE have been found to be suitable support materials. The method of support should not impede leaching by contacting more than 1 % of the surface area of the specimen. Moreover, the support should not interfere with the removal and replacement of the leachate.

7.3.1 It is often convenient to suspend the waste form from the cover of the leaching container using monofilament string.

7.3.2 Alternatively, samples can be placed on perforated or mesh stands.

7.4 *Solution Containers*—Containers used to store aliquots of leachate prior to analysis must allow for adequate preservation of the leachate and not be susceptible to plate-out of radionuclides.

7.5 *Stirrers*—Stirrers may be used to homogenize the leachate solution prior to removing aliquots for analysis. This is especially important for tests conducted at the reference and low temperatures. The specimen shall not be contacted during stirring.

7.6 *Filtration Equipment*—All visible particulates shall be removed from the leachate either manually or by using a filter medium that is capable of removing particulates that are 0.45 μm in diameter or larger. Disposable syringe filters are recommended, but preliminary tests must be conducted to determine if the filter and the filtration apparatus retain a significant amount of the species of interest (for example, due to sorption). It may be necessary to pre-condition each filter with a sacrificial volume of the leachate solution to saturate sorption sites in the filter.

8. Specimens

8.1 To the extent possible, the test material should be prepared using the same techniques as those used to produce full-scale waste forms. For example, the curing conditions used to prepare laboratory-scale specimens should match those used for actual waste forms as closely as possible, especially the

temperatures experienced by the large waste forms. The formulation, composition, processing conditions, history, and other pertinent information regarding the material being tested that could affect performance shall be recorded. Particular attention should be paid to the distributions of contaminants of interest in the material being tested so that representative specimens are used in the tests. This may include visual assessments and compositional or radiological analyses.

8.2 Accurate determination of the amount of the species of interest in the specimen at the start of the leach test shall be made and recorded. This may be based on analysis of the source material from which the specimens are prepared or analysis of the individual specimens.

8.3 Test specimens shall be physically, chemically, and radiologically representative of the full-scale solidified waste form.

8.3.1 Test specimens can be cut from a larger sample or cast individually. Specimens shall be large enough to represent the homogeneity and contaminant content of the full-scale waste form and have a geometric shape for which the surface area and volume can be determined from measured dimensions.

8.3.2 Specimen sizes should be selected based on the expected leaching behavior and test conditions. Smaller specimens can be prepared for durable materials than for less-durable materials.

8.3.3 Right circular cylindrical specimens with a diameter-to-height ratio between 1-to-1 and 1-to-2 are suitable. A convenient size is 2.5 cm diameter by 2.5 cm height.

8.3.4 Cuboid and wafer specimens are also suitable and shall have a minimum edge dimension of 0.2 cm for durable materials.

8.4 If a specimen is prepared in a mold, any excess material should be removed from the specimen prior to weighing it. These could include casting burrs, parting lines, riser points, tags, etc. that formed during the molding process and release agents.

8.4.1 If the quantity of the species of interest in the specimen (that is, the source term) was determined before the specimen was removed from the mold, the amount of that species that remained in the mold (plus material removed as excess) shall be determined and the amount accounted to be in the specimen adjusted appropriately.

8.4.2 Many solids prepared by casting form a skin on the outer surface during preparation that has different characteristics than the bulk material. The effect of the skin must be determined and differentiated from the bulk property. This can be done by conducting separate tests using samples with surfaces that represent the surface structures of large waste forms, such as surfaces that are cast against container walls, and tests with samples having cut or polished surfaces that expose the bulk material to the leachant. The effect of the skin can be determined from differences in the derived model parameter values for materials with and without the skin.

8.5 The dimensions of each specimen shall be measured with a calibrated device (for example, digital calipers) to the nearest 0.01 cm. The surface area and volume of the specimen are used to calculate model parameter values from the test results.

8.5.1 For right cylinder specimens, the geometric surface area and volume are calculated by modeling the specimen as a right circular cylinder. Two measurements of the diameter shall be made at both the top and bottom of the specimen and two measurements of the height made at diametrically opposite locations to take non-parallel faces and out-of-roundness into account. Measurements shall not be made at regions with visible irregularities. The surface area is calculated by using the arithmetic averages of the measured diameters and heights.

8.5.2 For cuboid and wafer test specimens, one measurement of each edge shall be made. The geometric surface area and volume are calculated by modeling each face of the specimen as a parallelepiped.

8.6 The surface area and volume can be adjusted to take into account deviations in the specimen shape from an ideal right circular cylinder or parallelepiped based on additional measurements and geometric calculations.

8.7 The uncertainty in the surface area and volume of the specimens should be quantified, for example, by using the propagation of errors method or, preferably, that developed by the Joint Committees for Guides in Metrology (1)[3] and promulgated by NIST (2, 3); see Annex A1.

9. Procedure

9.1 The specimen surface area-to-leachant volume ratio must remain the same for all intervals in a test series to ensure that any impacts of solution feedback and solubility effects are similar during each test interval if model coefficient values are to be derived from the test results.

9.1.1 The specimen size and leachant volume must be selected by compromising the benefits of using a large specimen (ease of fabrication, uniformity of specimens, ease of sampling reacted materials, etc.) with the complications of large solution volumes (handling, analytical limitations, waste disposal, etc.).

9.1.2 For example, to replicate the standard conditions in the Test Method C1220 static leach test, the leachant volume (in cm^3) used for each interval must be 10× the surface area of the specimen (in cm^2) as calculated below:

$$\frac{\text{specimen surface area } (cm^2)}{\text{leachant volume } (cm^3)} \equiv \frac{1 \ cm^2}{10 \ cm^3} = 0.1 \ cm^{-1} \quad (1)$$

9.1.3 This ratio requires a large volume of water for specimens of even moderate size. For example, a 2.5 × 2.5 cm cylindrical specimen having a surface area of 29.45 cm^2 would require 294.5 mL of solution for each of the 11 test durations.

9.2 *Leachant Volume*—The leachant volume is selected based on the specimen surface area and an estimate of the release rates of the species of interest. The volume must be

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

small enough that the solution concentrations that are generated during the test can be analyzed, but should be large enough that solution feedback effects on the release rates are negligible (that is, so that the chemical gradients between the solid and solution are the same during each test interval). The leachant mass can be measured and used to calculate the volume if the leachant density is known.

9.2.1 Large volumes of leachant that maintain highly dilute solutions can make analysis challenging, even for major constituents of the specimen, and present unnecessary waste disposal costs. Under these circumstances, a higher specimen surface area-to-leachant volume ratio may be used.

9.2.2 The leach rates of some waste form materials may be low enough that a specimen surface area-to-leachant volume ratio higher than 0.1 cm^{-1} must be used to generate measurable solution concentrations. In contrast, the leach rates for some waste form materials may be high enough that a specimen surface area-to-leachant volume ratio lower than 0.1 cm^{-1} must be used to avoid solution saturation effects.

9.2.3 The amount of a species released may be measured directly or calculated from the measured solution concentration and volume of leachate present at the end of the test interval. The mass of the assembled vessel is measured before and after each test interval to determine the mass of water lost due to vessel leakage during the test interval and calculate the final leachate volume.

9.2.4 The effects of solution feedback and solubility limits on the release rates can be identified by conducting tests at different specimen surface area-to-leachant volume ratios or using different exchange intervals. Solution feedback effects are expected to be more significant in tests conducted at higher temperatures, higher surface area-to-leachant volume ratios, and using longer test intervals.

9.2.5 The user must verify that solution feed-back effects or solubility limits do not affect the results. Solution feedback effects (or solubility limits) are considered negligible if the same model coefficient values (for example, D_e), within experimental uncertainty, are determined for tests conducted under different test conditions (for example, specimen surface area-to-leachant volume ratios or test interval).

9.3 *Temperature*—Tests shall be conducted at a minimum of three temperatures to establish that the release rate increases systematically with higher temperatures. One test must be conducted at a temperature near 20 °C to be used as a reference. The other temperatures should be selected based on knowledge of the material being tested. For example, the recommended maximum temperature for cementitious materials is 50 °C, which is below the threshold of anomalous releases observed previously (**4**). Higher temperatures may be used in tests with materials known to be stable at those temperatures.

9.3.1 The reference test shall be conducted at a temperature between 19 and 25 °C and maintained within 1 °C of that temperature throughout the test duration. Flexibility in the reference temperature is provided in deference to the variation in ambient temperatures and its negligible effect on the test response.

9.3.2 The temperature of the controlled-temperature device shall be stabilized at or near the desired temperature prior to test initiation. The stabilized temperature establishes the target temperature for the test.

9.3.3 The controlled-temperature device must maintain a temperature within 1 °C of the target temperature throughout the test (except for short-term perturbations with the vessels are placed in the device or removed for sampling). The temperature shall be recorded both before the vessel is placed in the device at the beginning of each test interval and before it is removed at the end of each test interval.

9.3.4 The time required for the device to return to the set temperature after it is opened (for example, to emplace or remove a test vessel) should be noted, even though the vessel contents may not have attained that temperature. The time required to heat the specimen to relatively high test temperatures should be a small fraction of the test interval, but is expected to be consistent for all test intervals. The leachant can be preheated to the target temperature prior to the exchange to minimize this effect.

9.4 *Leachant Replacement*—This test method includes daily leachant replacements over a total test duration of eleven days. The times of day at which the vessel is removed from the controlled-temperature device, the specimen is removed from the leachate and placed in the fresh leachant, and the vessel is placed back into the controlled-temperature device should all be recorded to the nearest minute. The use of an electric clock or a watch is adequate.

9.4.1 Test vessels shall be removed from the controlled-temperature device for leachant replacement individually to minimize the temperature change of the solution prior to it being sampled.

9.4.2 The fresh leachant may be at room temperature or pre-heated to the test temperature prior to use to minimize the time required for the test to return to the test temperature. The leachant must be consistently pre-heated (or not pre-heated) prior to all replacements during the test series.

9.4.3 The mass of the assembled vessel shall be measured before the vessel is placed in the controlled-temperature device at the start of a test interval and when the vessel is removed at the end of the test interval. The difference in mass provides a measure of the loss of water due to evaporation during the test interval (see 7.2).

9.4.4 If the specimen is suspended from the top of the container, the most convenient method for changing the leachant is to lift off the cover (with the specimen still attached) and place it on a new vessel that contains the appropriate volume (or mass) of fresh leachant. The new leachant may be pre-heated to the test temperature (if practical). The new vessel shall be sealed, weighed, and placed into the temperature-controlled device as quickly as possible. The specimen should be exposed to air for as short a time as possible during leachant changes. It is not necessary to remove the small amount of adhering solution or rinse the sample during the transfer.

9.4.5 If the specimen is on a support stand at the bottom of the test container, a portion of the leachate can be decanted into a collection container and the sample removed from the test vessel with forceps and either set aside while the vessel is

emptied and fresh leachant added or placed immediately into another test vessel with leachant or pre-heated leachant (do not rinse the specimen). The specimen should be exposed to air for as short a time as possible when the solution is exchanged. The new test vessel shall be sealed, weighed, and then placed into the controlled-temperature device as quickly as practical.

9.4.6 If appropriate, two additional test intervals of 8 and 16 hours can be run immediately after the series of 1-day test intervals to discriminate between constant dissolution kinetics and solution saturation effects. These shall be excluded from the data set of 1-day interval tests used to derive model parameter values.

9.4.7 If appropriate, an abbreviated replacement schedule of four 1-day test intervals can be used for material screening purposes, but those data shall not be used to derive model parameter values.

9.5 *Leachate Sampling Procedure*—Immediately after opening the test vessel, stir the leachate thoroughly and remove sample aliquots quickly to minimize any artifacts that could occur during cooling (for example, precipitation). Alternatively, decant the leachate into one or more solution bottles. Several aliquots may be required at each sampling for use in the desired analyses.

9.5.1 If the specimen is suspended from the vessel lid, place the lid on the new vessel containing the appropriate amount of fresh water and initiate the next test interval before removing aliquots of the leachate from the old vessel for analysis.

9.5.2 If the specimen is placed on a stand at the bottom of the vessel, stir the leachate and remove aliquots of the leachate for analysis before initiating the next test interval. Alternatively, decant most of the solution into one or more clean containers and remove aliquots from that container.

9.5.3 The solution aliquots should be collected and preserved in ways appropriate for the analytical technique to be employed.

9.6 The solution should be inspected for the presence of particulates prior to collecting solution aliquots for analysis. If particulates are visible in the leachate, the quantity of the species of interest associated with them must be taken into account with regard to the fractions that are released and retained in the specimen during subsequent intervals. How they are treated depends on when they were generated, which can be inferred from the similarity to the material being tested. If the particles are a significant fraction of the specimen mass, the amounts of species available for release in subsequent test intervals should exclude the amounts estimated to be in the particles.

9.6.1 The particulates should be removed prior to analyzing the solution to exclude the amounts of the species of interest associated with the spalled material from the amount released.

9.6.2 If the particulates form by spalling from the specimen due to handling, they should be removed prior to analyzing the solution and the amounts of the species of interest associated with the spalled material should be excluded from the amounts released. Large particles can be removed manually and small particles can be removed by filtration.

9.6.3 If the particulates are suspected to have formed after the species of interest were released, such as by precipitation

from a saturated solution, two approaches can be used. One requires filtration of the leachate and subsequent analysis of both the filtrate and the particulate material on the filter. The other is to acidify the leachate to dissolve the particulates and thereby include the associated species of interest in the analyzed solution. One or both methods can be used depending on the information desired.

9.6.4 The test should be terminated if the test specimen becomes highly fractured or friable and the increase in surface area of the specimen and the particulates cannot be quantified.

9.7 *Acid Strip*—At least one vessel bottom shall be subjected to an acid strip at the end of a test interval to verify that the species of interest is not sorbing or plating onto the vessel. If the amount sorbed is not negligible, the vessel shall be acid-stripped after every sampling. The amount of the species of interest recovered in the acid strip shall be added to the amount determined to be in the leachate to represent the total amount released.

9.7.1 Discard any remaining leachate solution from the vessel and rinse the vessel with demineralized water to remove any remaining test solution.

9.7.2 Fill vessel with an amount of 2 volume % nitric acid that is equal to or greater than the amount of leachate that was removed.

9.7.3 Cap the container and agitate, then let settle for at least 30 min.

9.7.4 Collect a sample of the acid strip solution for analysis of the contaminant content.

9.7.5 Discard and properly dispose of the remaining acid strip solution.

9.8 *Analysis and Standards*—Analysis of the leachate for the species of interest shall be conducted by standard methods and using appropriate calibration standards. If appropriate, standards should be prepared to include concentrations of matrix elements similar to those in the test solutions. For radioactive materials, a series of reference solutions can be prepared by diluting an aliquot of the original waste solution (or waste) that was used to make the material being tested for comparative analysis. The analytical results for the test samples can then be compared directly to analytical results for these reference solutions to calculate fractional releases without the need for absolute standards, calibrated detector efficiencies, or decay corrections.

9.9 *Standard Test*—One or more tests with equivalent specimens shall be conducted at 20 °C for use as a standard for comparison with tests conducted at other temperatures and with other materials. Triplicate standard tests at 20 °C are required if the results will be used to project releases to long durations or larger waste forms.

9.10 *Blank Test*—Depending on the species of interest, a blank test with either no specimen or with a specimen that does not contain the species of interest is recommended to provide background solutions to detect contamination that may occur during the procedure or provide background levels for tests using leachants that contain measurable amounts of the species of interest.

10. Calculations

10.1 *Incremental Fraction Leached*—The incremental fraction of species *i* leached (*IFL*) during test interval *n* is calculated by using Eq 2:

$$IFL_n = \frac{a_n}{A_o} \qquad (2)$$

where:

a_n = the quantity of species *i* measured in the leachate recovered during the *n*th test interval, and

A_o = the quantity of species *i* in the specimen at the beginning of the test.

NOTE 1—The index for the species of interest is excluded for clarity.

10.1.1 In the case of a radionuclide having a half-life that is short relative to the time span between when a_n and A_o were measured, both terms must be corrected for radioactive decay to the beginning of the test.

10.1.2 If the value of a_n is calculated from the measured solution concentration using the leachate volume, the uncertainty in the measured concentration and the uncertainty in the leachate volumes must be taken into account (see Annex A1). The leachate volume can be calculated using the measured leachate mass and leachant density.

10.1.3 The average rate of release during any test interval can be calculated by dividing *IFL* by the duration of that interval. That rate can then be divided by the surface area of the specimen to obtain the average fraction released per area per time (flux). Flux values for different species in tests conducted with different materials and specimens having different surface areas can be compared directly and used to calculate releases from full-scale waste forms.

10.2 *Cumulative Fraction Leached*—The cumulative fraction of species *i* released through the *N*th interval (CFL_N) is calculated by using Eq 3:

$$CFL_N = \frac{\sum_{n=1}^{N} a_n}{A_o} = \sum_{n=1}^{N} IFL_n \qquad (3)$$

NOTE 2—The index for the interval is excluded for convenience hereafter.

10.2.1 Plotting the *CFL* value at each interval against the cumulative time provides a graphical comparison between data from various tests with each other and with modeling results. Two examples of this type of plot are shown in Fig. 1. Fig. 1(a) shows the linear releases of Si from a borosilicate glass into a leachant with 30 ppm $NaSiO_4$ in tests conducted at S/V ratios of 6, 25, and 150 m^{-1} and 90 °C that occur at different rates. Fig. 1(b) shows the non-linear release of Cr from a grout-based material in tests conducted at S/V ratios of 10 m^{-1} in demineralized water at 20, 70, and 90 °C. Regression fits are shown with the results as discussed in 10.3.

10.3 *Regression of Models*—Analytical models can be regressed to the *CFL* values calculated from the test data at each test duration to identify the process controlling the release and optimize model parameter values. If the optimized *CFL* values calculated with the model agree with the measured values within a designated "goodness of fit" (which is related to the uncertainty in the regression; see 10.4), then it can be concluded that that model represents the release mechanism and the regressed parameter values can be used to represent intrinsic material properties.

10.4 *Quantifying Uncertainty in Model*—The error in the fit of the model to the data is quantified as the sum of the squares of the residuals at each *CFL*. For a total of *N* measured *CFL* values, the sum of the squares of the residuals is defined as:

$$R^2 \text{ Sum} = \Sigma (CFL_{model} - CFL_{measured})^2 \qquad (4)$$

The uncertainty in the cumulative test duration (the abscissa value) is considered to be negligible.

10.5 *Effective Linear Dissolution Rate*—The results of this test method can be used to determine the kinetic dissolution rate under far-from-saturation conditions. For example, the linear fits in Fig. 1(a) indicate the same solution concentrations are generated during each test interval. However, higher concentrations generated in tests at higher S/V ratios are seen to attenuate the dissolution rate. This test method can be used to quantify the attenuating effects of different solution species.

10.6 *Effective Diffusion Coefficient*—The results of this test method can be used to determine the effective diffusion coefficient (D_e) of species of interest released by a diffusion-controlled process. For example, the fit in Fig. 1(b) indicates the solution concentrations generated during sequential test intervals decrease in proportion to the square root of the cumulative test duration. The curves show regressions of semi-infinite solid diffusion model.

10.6.1 If diffusion is shown to limit the contaminant release measured in a test, then a diffusion model can be used to calculate releases over long times at that temperature. The use of a diffusion model to extrapolate release behavior requires that the waste form remains intact and the leaching mechanism does not change with time. Demonstrating that the same mechanism is operative at 20 °C and at elevated temperatures provides confidence that the same process will control release over long times at intermediate temperatures, at least up to the extent represented by the maximum *CFL* value measured in a test.

10.7 *Relationship of Temperature to Contaminant Release*—The accelerated leach test uses elevated temperatures as the primary means of increasing the contaminant release rate relative to the reference temperature of 20 °C, which is expected to be the minimum test temperature. The maximum temperature may be limited by the thermal stability limit of the host solid. For example, some organic matrix materials become unstable at temperatures below 50 °C.