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Standard Test Method for
Accelerated Leach Test for Diffusive Releases from Solidified Waste and a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Waste Forms[1]

This standard is issued under the fixed designation C 1308; 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 method for accelerating the leach rate of solidified waste and determining if the release is diffusion-controlled. This test method is applicable to any material that does not degrade, deform, or change leaching mechanism during the test.

1.1.1 If diffusion is the dominant leaching mechanism, then results of this test can be used to model long-term releases from waste forms. Diffusion can be confirmed as the leaching mechanism through the use of a computerized mathematical model for diffusion from the finite cylinder (

1.1 This test method provides procedures for measuring the leach rates of elements from a solidified matrix material, determining if the releases are controlled by mass diffusion, computing values of diffusion constants based on models, and verifying projected long-term diffusive releases. This test method is applicable to any material that does not degrade or deform during the test.

1.1.1 If mass diffusion is the dominant step in the leaching mechanism, then the results of this test can be used to calculate diffusion coefficients using mathematical diffusion models. A computer program developed for that purpose is available as a companion to this test method (Note 1).

1.1.2 The leaching mechanism should be verified as diffusion-controlled by a means other than analysis of the leach test data. For this purpose, analysis of post-leaching concentration profiles within the solid waste form is recommended.

1.1.2 It should be verified that leaching is controlled by diffusion by a means other than analysis of the leach test solution data. Analysis of concentration profiles of species of interest near the surface of the solid waste form after the test is recommended for this purpose.

1.1.3 Potential effects of partitioning on the test results can be identified through modeling, although further testing and analyses are required to determine the cause of partitioning (for example, if it occurs during production of the material or as a result of leaching).

1.2 The method is a modification of other semi-dynamic tests such as the IAEA test (1)[2] and the ANS 16.1 Leach Test wherein elevated temperatures are used to accelerate diffusive release to an extent that would only be reached after very long times at lower temperatures. This approach provides a mechanistic basis for calculating diffusive releases at repository-relevant temperatures over long times, provided that the leaching mechanism does not change with temperature.

1.2.1 Tests can be conducted at elevated temperatures to accelerate diffusive release and provide a mechanistic basis for calculating diffusive releases that would occur at lower temperatures over long times. Tests conducted at high temperatures allow the temperature dependence of the diffusion coefficient to be determined. They also demonstrate that the diffusion mechanism is rate-limiting through the measured extent of diffusive release.

1.2.2 Releases at any temperature can be projected up to the highest cumulative fractional release value that has been measured for that material (at any temperature), provided that the mechanism does not change. The mechanism is considered to remain unchanged over a range of temperatures if the diffusion coefficients show Arrhenius behavior over that range.

NOTE 1—The computer program and the models are briefly described in 1—A computer program in which the test results are evaluated using three diffusion models is briefly described in Annex A1 and in the Accelerated Leach Test Method and User's Guide for the "ALT" Computer Program (12). The data are fit with model equations for diffusion from a semi-infinite solid, diffusion from a finite cylinder, and diffusion with partitioning of the species

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[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.07 on Waste Materials.
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[2] Annual Book of ASTM Standards, Vol 14.02.
[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

of interest to determine effective diffusion coefficients and quantify the goodness of fit. The User's Guide contains several typographical errors; these are identified in Annex A1.

1.2

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E632 Standard Practice for Developing Accelerated Tests to Aid Prediction of the Service Life of Building Components and Materials [3]

C 1220 Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste

D 1193 Standard Specification for Reagent Water

2.2 *ANSI/ANS Standard:*

ANSI 16.1 Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure [4]

3. Terminology

3.1 *cumulative fraction leached* —the sum of the fractions of a species leached during all sampling intervals calculated as CFL=$\Sigma a_n / A_O$ and assuming no radioactive decay, where a_n is the quantity of a species in the leachate during any interval and A_O is the quantity of that species originally present in the sample.

3.2 *diffusion coefficient (diffusivity)* —based on Fick's Laws for diffusion, the diffusion coefficient is the ratio of the rate of transfer of a diffusing substance through the unit area of a section to the concentration gradient measured normal to the section.

3.3 *effective diffusion coefficient (effective diffusivity)*—the diffusion coefficient that results from diffusion as it is modified by other processes (for example, adsorption) or physical constraints (for example, tortuosity and constrictivity).

3.4 *finite cylinder (finite medium)*

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

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

3.5 *incremental fraction leached* —the fraction leached of a species of interest during a single sampling interval calculated as IFL=a_n / A_O and assuming no radioactive decay.

3.6 *leachant*—the liquid that contacts the specimen during a leach test or contacts a waste form in the disposal environment.

3.7 *leachate*—the leachant after contacting the specimen or the waste form.

3.8 *leaching*—the process (or processes) by which mass transport from a solid to a liquid takes place.

3.9 *leaching interval*

3.1.5 *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.

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

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

3.1.8 *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.

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

3.10 *leaching mechanism*—the process that controls the rate of mass transport out of a specimen during leaching.

3.11 *reference leach test*—a leach test conducted under defined conditions, the results of which are used as a standard against which the results of other leach tests (for example, accelerated) are compared. In this test method a reference leach test is one that is conducted at 20°C.

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[3] 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* , Vol 11.01. volume information, refer to the standard's Document Summary page on the ASTM website.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.12*semi-dynamic leach test*—a leach test method that exposes the specimen to fresh leachant on a periodic schedule.

3.13*semi-infinite medium*—a body used in diffusion theory whose outer boundary is effectively at an infinite distance from the inner region.

3.14*source term*— the original concentration, prior to leaching, of a species of interest in a specimen or a waste form.

3.15*surface area*— for purposes of this test method, surface area is defined as the geometric surface area of a specimen calculated from macroscopic measurements of its dimensions.

3.16*volume*—for purposes of this test method, volume is defined as the volume of a specimen calculated from macroscopic measurements of its dimensions.

3.17*waste form*—a stable, solid body composed of the waste and a solidification agent.

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

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

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

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

3.1.14 *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.

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

3.1.16 *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.

3.1.17 *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.

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

4. Summary of Test Method

4.1This test method is a semi-dynamic leach test, that is, the leachant is sampled and replaced periodically. It is based on earlier semi-dynamic tests such as the IAEA test (

4.1 This test method is a semi-dynamic leach test in which a cylindrical specimen is immersed in a leachant that is completely replaced after specified intervals. The concentration of an element of interest in the recovered test solution is measured after each exchange; this is referred to as the *incremental fraction leached (IFL)*. The accumulated amount of the species of interest in the intervals prior to and including the interval of interest is analyzed to determine if the release from the solid can be described using a mass diffusion model. The amount accumulated through a particular test duration is referred to as the *cumulative fraction leached (CFL)*.

4.2 Tests at a single temperature are adequate to compare the leaching behaviors of different materials.

4.3 The results of tests at repository-relevant temperatures can be extrapolated to long times if data from tests run at elevated temperatures and data from tests run at the reference temperature (20°C) can be modeled using a diffusion model and the diffusion coefficients show Arrhenius behavior.

4.3.1 Elevated temperatures are used to accelerate the release of a species of interest and collect enough data to show that the release is controlled by diffusion and determine the value of the diffusion coefficient.

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

4.3.3 By generating data over a range of temperatures, an Arrhenius plot can be produced to interpolate values of the diffusion coefficient 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.

4.3.4 A computer program that plots the experimental data and a regression curve calculated using a finite cylinder model (**2**) and the ANS 16.1 Leach Test.[4] Elevated temperatures, large volumes of leachant, frequent leachant changes, and small specimen size are used to obtain accelerated releases. This is a short-term test, requiring sampling on each of eleven or more successive days. Tests must be performed to demonstrate that the leaching mechanism does not change at the elevated test temperature. This may be done by performing leach tests at a minimum of three temperatures to ascertain that the mechanisms and structural controls of leaching do not change with increasing temperature. If the test is to be used only for making comparisons among specimens, then testing at a single temperature is adequate.

4.2The results of this accelerated test can be extrapolated to long times if the data from tests run at elevated temperatures and the tests run at the reference temperature (20°C), and can be modeled by diffusion. A computer program that plots the experimental data and a curve calculated from an effective diffusion coefficient for diffusion from a finite cylinder (Fig. 1) is available from ASTM. If the data from the accelerated tests, the reference test, and the modeled curve fit within defined criteria, the leaching mechanism is taken to be diffusion. In this case, the model can be used to project releases from full-scale waste forms and to long times. The accelerated test provides a measure of the maximum fractional release to which the modeled data can be extrapolated. By generating data over a specified temperature range, an Arrhenius plot can be produced allowing projections to be made at
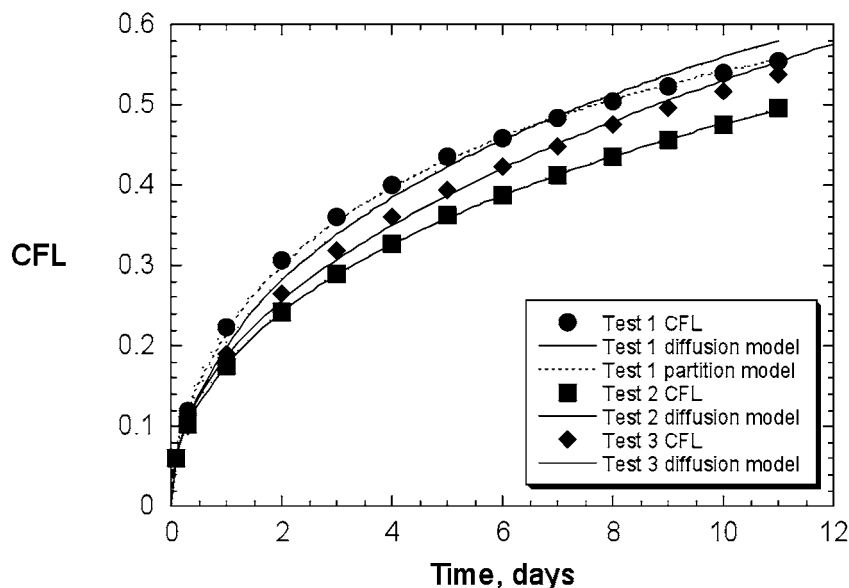
FIG. 1 Plotted Results of Test 1, Test 2, and Test 3 with
Model Fits

~~temperatures other than those tested. If the diffusion model cannot fit the data, other models (for example, diffusion plus partitioning and solubility limited leaching) contained in the computer program can be used to indicate the leaching mechanism that controls releases. No extrapolations are allowed with these models. If no model fits the data, then an alternative graphical comparison of the data is recommended. A linear plot of modeled CFL plotted against experimental CFL verifies that the accelerated data is comparable to the reference data, showing that the accelerated test is appropriate. With this technique, no extrapolation of data can be made.~~ is available from ASTM (see Note 1). The program provides the value of the effective diffusion coefficient, the modeled *IFL* and *CFL* values, and a measure of the goodness of fit of the model.

4.4 If the data from the accelerated tests, the reference test, and the fit of the modeled curve agree within defined criteria, the leaching mechanism can be taken to be diffusion-controlled and a diffusion model can be used to calculate releases from full-scale waste forms for long times.

4.4.1 The accelerated leach test provides the maximum cumulative fractional release to which the modeled data can be extrapolated. 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.

4.4.2 Because the cumulative fraction leached is a function of the specimen surface area-to-volume ratio, the results of tests with the small-scale specimens used in the ALT directly represent leaching from large-scale waste forms having the same aspect ratio.

4.4.3 The effective diffusion coefficient can be used to calculate diffusive releases from waste forms with other shapes.

4.5 If the diffusion model does not fit the data within defined criteria, no extrapolation can be made in time or specimen size. However, other models can be applied to the data to evaluate the leaching process.

4.5.1 A model including diffusion with partitioning of the species of interest between phases having different release behaviors is included in the computer program **(2)** .

4.5.2 The possibility of a solubility-limit to the release of the species of interest is addressed in the computer program **(2)**.

4.6 If the data cannot be fit with a diffusion model within the defined criterion, then graphical comparisons of the data are recommended for added insight: For example, a plot of the cumulative fraction leached (*CFL*) from ALT conducted at an elevated temperature against the *CFL* from ALT conducted at the reference temperature can be used to verify that the accelerated data are consistent with the reference data and that the accelerated test appropriately accelerates the release, even though the release is not diffusion-limited.

5. Significance and Use

~~5.1 This test method measures mass transport from a cylindrical solidified waste form into water under conditions that accelerate leaching. Test parameters, such as the volume of leachant and the frequency at which the leachant is changed, have been optimized to eliminate experimental effects (for example, saturation effects that can complicate modeling of the net forward diffusion rate).~~

5.1 This test method can be used to measure the release of a component from a cylindrical solidified waste form into water at the reference temperature of 20°C and at elevated temperatures that accelerate the rate and extent of leaching relative to the values measured at 20°C.

5.2 This test method can be used to:

~~5.2.1 Provide diffusion coefficients for waste forms;~~

5.2.2 ~~Obtain higher fraction releases than can be achieved with expected service conditions for greater confidence in waste form properties;~~

5.2.3 ~~Obtain measurable leachate concentrations from materials that have very low release rates under service conditions; and~~

5.2.4 ~~Compare releases from various types of solidification agents and formulations.~~

5.3 ~~Modeling the experimental observations obtained in the test allows the extrapolation of leaching results to long times and to full-scale waste forms under the following constraints:~~

5.3.1 ~~Results of this test method cannot be taken to apply to releases in specific disposal environments unless tests are conducted to determine the leaching mechanism under those conditions.~~

5.3.2 ~~Projections of releases require the long-term stability of the waste form, which may or may not be indicated adequately by short-term tests.~~

5.3.3 ~~Extrapolations are limited to the maximum CFL obtained with the accelerated test.~~

5.3.4 ~~The leaching mechanisms observed in tests conducted at elevated temperatures must be the same as the mechanisms observed in the tests run at the reference temperature.~~

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

5.2.2 Determine the diffusion coefficients for the release of waste components from waste forms 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 diffusive releases.

5.2.4 Determine the temperature dependence of diffusive release.

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

5.3.1 Results of this test method address an intrinsic property of a material and 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 an effective diffusion coefficient for those conditions.

5.3.2 Projections of releases over long times requires that the waste form matrix remain stable, which may be demonstrated by the behavior of the specimen in ALTs at elevated temperatures.

5.3.3 Extrapolations in time and scale are limited to values that correspond to the maximum *CFL* value obtained in an accelerated test.

5.3.4 The mechanism must be the same at all temperatures used in the extrapolation. The same model that describes the results of tests conducted at elevated temperatures must also describe the results of tests run at the reference temperature of 20°C.

6. Apparatus

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

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

7. Reagents and Materials

7.1 *Leachant*—~~The leachant shall be distilled or deionized water meeting or exceeding standards for types II or III reagent water as specified in Specification D1193~~—The leachant can be selected with regard to the material being tested and the information that is desired. Demineralized water, synthetic or actual groundwaters, or chemical solutions can be used. The effects of the leachant solution on the species of interest (that is, the species for which the diffusion coefficient is to be measured) and the solid must be considered. For example, the leachant should not degrade the host solid. In general, the leachant should be devoid of the species of interest to minimize solution feedback and solubility limit effects. If the leachant does contain a non-negligible amount of the species of interest, blank tests should be conducted to provide background concentrations to calculate the amounts released from the solid by using the concentrations measured in the tests. If demineralized water is used, it must meet or exceed the standards for types II or III reagent water specified in Specification D 1193.

7.2 *Leaching Containers*—Leaching containers shall be made of a material that does not react with the ~~leachant/leachate and the~~ leachant, leachate, or specimen. It is particularly important to select materials that ~~allow very little~~ are not susceptible to plate-out of ~~radionuclides~~ species of interest from solution. High density polyethylene has been found to be a suitable container material. The top of the container shall fit tightly to minimize evaporation. ~~This fit~~ The mass of the vessel must be checked ~~at test temperatures~~ before sampling to ~~ensure~~ verify that evaporative losses are less than 1 % of the leachant mass (or volume) over ~~24 hours.~~ every test interval.

7.3 *Specimen Supports*—~~Supports for the specimens shall be made of a material that does not react with the leachant/leachate or the specimen and does not allow plate-out onto the support. The method of support should not impede leaching by obstructing the surface area of the specimen by more than 1%. Moreover, it should not interfere with replacement of the leachate. It is often convenient to suspend the waste form from the cover of the leaching container using monofilament string.~~ —Supports for the specimens shall be made of a material that does not react with the leachant, leachate, or specimen and is not susceptible to plate-out. 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 *Sample Containers*—Containers to hold aliquots of leachate for storage prior to analysis should not allow any plate-out of radionuclides. The containers should be checked to ascertain that evaporation over long times is acceptably small. —Containers to hold aliquots of leachate for storage prior to analysis should not be susceptible to plate-out of radionuclides. The container must allow for adequate preservation of the leachate and specimen.

7.5 *Stirrers*—Stirrers that are long enough to reach the bottom of the leaching containers are required. Wood or plastic tongue depressors are adequate. —Stirrers are used to homogenize the leachate solution prior to removing aliquots for analysis.

7.6 *Filtration Equipment*—If particulates are present in the leachate, filtration is necessary. Tests must be conducted to ensure that the filter and the filtration apparatus do not adsorb the species of interest. The filter medium should be capable of removing particulates that are 0.45 µm in diameter. Disposable syringe filters are recommended. —If filtration of visible particulates in the leachate is required, the filter medium should be capable of removing particulates that are 0.45 µm in diameter or larger. Disposable syringe filters are recommended. Tests must be conducted to determine if the filter and the filtration apparatus adsorb a significant amount of the species of interest. 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 Right circular cylindrical specimens shall be used with a diameter-to-height ratio between 1:1 and 1:2. A convenient size is 2.5 cm diameter by 2.5 cm height. Smaller sizes should be avoided to preclude problems of mixing and producing nonhomogeneous samples.

8.2 Specimens shall be representative of the full-scale solidified waste form. Particular attention should be paid to ensuring that the laboratory specimen is homogeneous. The specimens should be prepared using the same techniques as those used to produce full-scale waste forms. Curing conditions, especially the temperatures experienced by the large waste forms, should be duplicated for laboratory-scale specimens. Care should be taken to ensure that surfaces of the laboratory specimens reflect the structure of surfaces of large waste forms. This refers to surfaces that are cast against container walls as opposed to free or cut surfaces.

8.3 A minimum of three specimens should be tested at any given temperature.

8.4 The dimensions, weight, composition, and curing history shall be recorded for each specimen. Accurate determination of the source term (A_0), that is, the amount of the species of interest in the specimen at the start of the leach test, shall be made and recorded.

8.1 Right circular cylindrical specimens shall be used with a diameter-to-height ratio between 1:1 and 1:2. This shape is used to facilitate modeling the test results. A convenient size is 2.5 cm diameter by 2.5 cm height. Smaller specimen sizes should not be used to avoid producing nonhomogeneous samples.

8.2 To the extent possible, the specimens 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.

8.3 Specimens shall be representative of the full-scale solidified waste form. Particular attention should be paid to ensuring that the species of interest is homogeneously distributed in the material being tested. Test specimens can be cut from a larger sample or cast individually.

8.4 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 are representative of the structure of surfaces 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 diffusion coefficients for materials with and without the skin.

8.5 A minimum of three replicate tests should be conducted at each temperature if results are to be used to predict long-term behavior.

8.6 The dimensions, weight, composition, curing history, and other pertinent information that could affect performance shall be recorded for each specimen.

8.7 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.

8.8 If a specimen is prepared in a mold, any excess material should be removed from the specimen prior to weighing it.

8.8.1 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.

9. Procedure

9.1 *Removal of Specimen from Mold* —After removing the specimen from its container or mold, any excess material should be removed prior to weighing the specimen. The mold should be rinsed in a volume of water equal to the volume of the specimen. If the quantity of the species of interest contained in this rinse water represents more than 0.5% of the total quantity in the specimen, the value should be subtracted from the source term (A_0

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9.1 The dimensions of each specimen shall be measured with a calibrated device (for example, digital calipers) to the nearest 0.01 cm. At least two measurements of the diameter shall be made at the top and bottom of the specimen and two measurements of the height at diametrically opposite locations. The geometric surface area and volume are calculated by modeling the specimen as a right circular cylinder and using the arithmetic averages of the measured diameters and heights.

9.1.1 The surface area and volume of the specimen are used to calculate the diffusion coefficient (see A1.3.2.1).

9.1.2 The uncertainty in the surface area and volume of the specimens contribute to the uncertainty in the diffusion constant and should be quantified, for example, by using the propagation of errors method or, preferably, that developed by the International Committee for Weights and Measured (CIPM) and promulgated by NIST **(3)** ; see Annex A2.

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

9.2 *Leachant Volume*— ~~The leachant volume used for each interval is 100×the surface area of the specimen as calculated below:~~ —The leachant volume is selected based on the specimen surface area and an estimate of the leach rate. The volume must be low enough that the solution concentrations that are generated during the test can be analyzed, but high enough that solution feedback effects on leaching are negligible (that is, so that the chemical gradient between the solid and solution remains nearly constant). The solution mass can be measured and used to calculate the volume if the solution density is known.

9.2.1 The solution volume is not used directly in the calculation of the diffusion constant, but is used to calculate the mass of the species of interest from the measured solution concentration.

9.2.2 The specimen surface area-to-solution volume must remain the same for all test intervals in an ALT to ensure that any impacts of solution feedback and solubility limitation are similar during each test interval.

9.2.3 The specimen size and solution 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.).

9.2.4 The effects of solution feedback and solubility limits can be identified (or mitigated) by conducting tests at different specimen surface area-to-leachant volume ratios. Solution feedback effects are expected to be more significant at higher temperatures and surface area-to-leachant volume ratios.

9.2.5 For example, to replicate the standard conditions in the Test Method C 1220 static leach test, the leachant volume (in cm^3) used for each interval must be $10\times$ the surface area of the specimen (in cm^2) as calculated below:

$$\frac{\text{Leachant volume (cm}^3)}{\text{Specimen surface area(cm}^2)}=100cm\pm2\% \tag{1}$$

$$\frac{\text{Specimen surface area (cm}^2)}{\text{Leachant volume (cm}^3)}=100cm\pm2\% \tag{1}$$
~~Specimen surface area(cm²)~~

~~This ratio requires a large volume of water, for example, a 2.5 cm×2.5 cm specimen has a surface area of approximately 30 cm²~~) ≡ 1 cm210 cm3 = 0.1 cm⁻1

9.2.5.1 This ratio requires a very 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 ~~, giving a leachant volume of 3000 mL. Specimens that are much larger than this will require volumes of water that need more sophisticated means of wastewater handling (such as peristaltic pumps for draining the containers), since large volumes are too unwieldy for pouring.~~

~~Note2—Some waste form materials, such as glass, have such low leach rates that a volume-to-surface area ratio of 100 cm is not necessary. In some cases, such a large volume of leachant can make analysis challenging, even for major constituents of the specimen. Under these circumstances, the volume-to-surface area ratio may be reduced to 10 cm.~~ would require 294.5 mL of solution for each of the 11 test durations. Specimens that are much larger than this and tests at lower surface area-to-leachant volume ratios will require volumes of water that need sophisticated means of wastewater handling (such as peristaltic pumps for draining the containers), since large volumes may be too unwieldy for pouring.

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

9.2.7 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 value of D_e, within experimental uncertainty, is obtained for tests conducted at different specimen surface area-to-leachant volume ratios.

9.3 *Temperature*— ~~For materials and formulations that have not been tested previously, leach tests shall be conducted at a minimum of three temperatures to establish that leaching increases systematically with higher temperatures. One temperature must be 20°C. The recommended maximum temperature is 50°C, which is below the threshold of anomalous releases observed so far~~ (—**For materials and formulations that have not been tested previously, leach tests shall be conducted at a minimum of three temperatures to establish that the leach rate increases systematically with higher temperatures. One temperature must be 20°C. The other temperatures should be selected based on knowledge of the material being tested. For example, the recommended maximum temperature is 50°C for cementatious materials, which is below the threshold of anomalous**

releases observed previously (3). ~~Temperatures above 50°C can be used if it is demonstrated that releases follow the trend observed at low temperatures, that is, that the leaching mechanism has not changed.~~ . Temperatures above 50°C can be used if it is demonstrated that the leaching mechanism does not change.

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

9.3.2 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 may not have attained that temperature. The time required to heat the specimen to relatively high test temperatures may be a significant fraction of the first two test intervals (2 and 5 hours).

9.4 *Leachant Replacement*—~~Leachant replacements shall take place at the time intervals shown in Table 1. The time at which the specimen is first placed in the leachant should be noted. The leachant shall be brought to the test temperature before the specimen is placed in it.~~

~~9.4.1If the specimen is suspended from the top of the container, the most convenient method of changing the leachant is to lift off the cover (with the specimen attached) and place it on a new container full of fresh leachant that is at the test temperature. This can be replaced in the temperature-controlled environment while the leachate in the other container is being sampled. During leachant changes, the specimen should be exposed to air for as short a time as possible.~~ —Leachant replacements shall take place at the following time intervals: 2 hours, 5 hours, 17 hours, and 24 hours, and then daily for the next 10 days, for a total test duration of 11 days. The times at which the specimen is removed from the leachate and placed in the fresh leachant should be noted to the nearest minute. The times at which the vessel is removed from and emplaced in the controlled-temperature devise should be noted to the nearest minute. The use of an electric clock or a watch is adequate.

9.4.1 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 container with the appropriate volume (or mass) of fresh leachant. The new leachant may be pre-heated to the test temperature (if practical). The new container can be sealed and placed into the temperature-controlled environment immediately. During leachant changes, the specimen should be exposed to air for as short a time as possible. Rinsing the sample prior to transfer is not necessary.

9.4.2 If the specimen is at the bottom of the test container, the leachate can be decanted into a collection container and the sample recovered with forceps and placed immediately into another test container with pre-heated leachant (is not necessary to rinse the specimen). The new test container can be sealed and placed into the controlled-temperature device.

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 leachate solution due to evaporation (see 7.2).

9.5 *Leachate Sampling*—Immediately after the specimen has been moved to fresh water, the old leachate should be stirred thoroughly and sampled to minimize any artifacts caused by cooling (for example, precipitation). These samples will be used to ~~determine quantities of the species of interest that have leached from the specimen during each interval and should be preserved~~

TABLE 1 ~~Repl~~Exa~~cement Interva~~ls for the ~~Accelerated~~LT Test ~~L~~ Rea~~chan~~sults

Time (days)	Test 1		Test 2		Test 3	
	Interval~~FL~~	~~Incremental Time (t)~~CFL	IFL	~~Cumulative Time (Σt)~~FL	IFL	CFL
~~1~~	~~2 h~~	~~2 h~~E-02	6.06E-02	6.06E-02	6.09E-02	6.09E-02
0.083	6.12E-02	6.12E-02	6.06E-02	6.06E-02	6.09E-02	6.09E-02
~~2~~	~~5 h~~	~~7h~~1.19E-01	4.13E-02	~~1.02E-01~~	3.96E-02	~~1.01E-01~~
0.29	5.82E-02	1.19E-01	4.13E-02	1.02E-01	3.96E-02	1.01E-01
~~3~~	~~17 h~~	~~1 day~~	7.43E-02	~~1.76E-01~~	8.90E-02	~~1.90E-01~~
1.0	1.04E-01	2.23E-01	7.43E-02	1.76E-01	8.90E-02	1.90E-01
~~4~~	~~1 day~~	3.06E-01	~~6.61E-02 days~~	2.42E-01	~~7.45E-02~~	2.64E-01
2.0	8.27E-02	3.06E-01	6.61E-02	2.42E-01	7.45E-02	2.64E-01
~~5~~	~~1 day~~	~~3days.61E-01~~	4.68E-02	~~2.89E-01~~	6.10E-02	~~3.25E-01~~
3.0	5.51E-02	3.61E-01	4.68E-02	2.89E-01	6.10E-02	3.25E-01
~~6~~	~~1 day~~	~~4days.01E-01~~	3.85E-02	~~3.28E-01~~	3.95E-02	~~3.65E-01~~
4.0	3.98E-02	4.01E-01	3.85E-02	3.28E-01	3.95E-02	3.65E-01
~~7~~	~~1 day~~	~~5 days~~E-01	3.58E-02	~~3.63E-01~~	3.45E-02	~~3.99E-01~~
5.0	3.37E-02	4.35E-01	3.58E-02	3.63E-01	3.45E-02	3.99E-01
~~8~~	~~1 day~~	~~6days~~4.59E-01	2.48E-02	~~3.88E-01~~	2.45E-02	~~4.24E-01~~
6.0	2.45E-02	4.59E-01	2.48E-02	3.88E-01	2.45E-02	4.24E-01
~~9~~	~~1 day~~	~~7days~~4.84E-01	~~2.48E-02~~	~~4.13E-01~~	~~2.50E-02~~	~~4.49E-01~~
7.0	2.45E-02	4.84E-01	2.48E-02	4.13E-01	2.50E-02	4.49E-01
~~10~~	~~1 day~~	~~8days~~5.05E-01	2.20E-02	~~4.35E-01~~	2.65E-02	~~4.75E-01~~
8.0	2.14E-02	5.05E-01	2.20E-02	4.35E-01	2.65E-02	4.75E-01
~~11~~	~~1 day~~	~~5.23E-01~~	2.20E-02	~~4.57E-01~~	2.00E-02	~~4.9 days~~
9.0	1.84E-02	5.23E-01	2.20E-02	4.57E-01	2.00E-02	4.95E-01
~~12~~	~~1 day~~	~~1~~	~~0 days2~~	4.76E-01	2.25E-02	5.18E-01
10.0	1.53E-02	5.39E-01	1.93E-02	4.76E-01	2.25E-02	5.18E-01
~~13~~	~~1 day~~	~~1~~	~~1days.93E-02~~	~~4.95E-01~~	~~1.70E-02~~	~~5.35E-01~~
11.0	1.53E-02	5.54E-01	1.93E-02	4.95E-01	1.70E-02	5.35E-01

in a way appropriate for the analytical techniques. This may require that several aliquots be taken during each sampling. If particulates are present in the leachate, it is necessary to account for the quantity of the species of interest associated with them. 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 leachate. 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 to the vessel. If the amount sorbed is not negligible, the vessel shall be acid-stripped after every sampling, and the amount of the species of interest recovered in the acid strip shall be added to the amount in the leachate.

9.5.1 Discard any remaining leachate solution from the vessel and rinse with demineralized water.

9.5.2 Fill vessel with an amount of demineralized water equal to or greater than the amount of leachate that was removed.

9.5.3 Add the appropriate amount of concentrated ultrapure nitric acid to produce a 2 volume% acid solution.

9.5.4 Cap the container and agitate, then let settle for several minutes.

9.5.5 Collect a sample of the acid strip solution for analysis.

9.6 *Leachate Sampling*—Immediately after opening the vessel, the old leachate should be stirred thoroughly and sampled quickly to minimize any artifacts that could occur during cooling (for example, precipitation). Several aliquots may be required at each sampling for desired analyses.

9.6.1 If the specimen is suspended from the vessel lid, place the lid on the vessel with fresh water and initiate the next test interval before removing aliquots of the leachate for analysis.

9.6.2 If the specimen is placed on a stand at the bottom of the vessel, stir solution and remove aliquots of the leachate for analysis before initiating the next test interval.

9.6.3 The solution aliquots should be collected and preserved in ways appropriate for the analytical technique(s) to be employed.

9.6.4 If particulates are visible in the leachate, it is necessary to account for the quantity of the species of interest associated with them.

9.6.4.1 If the particulates form by spalling from the specimen, they should be removed prior to analyzing the solution and the species of interest associated with the spalled material should be excluded from the amount released.

9.6.4.2 If the particulates formed after the species of interest were leached, 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 (for example, analyze filtered and unfiltered solutions), depending on the information desired.

9.7 *Analysis and Standards*—Analysis of species in the leachate will be conducted by standard methods. Appropriate standards will be used for analysis. If necessary, standards will be prepared to match the matrix elements in the samples. For radioactive specimens, the preferred method of analysis includes use of a standard prepared from an aliquot of the original solution (or waste) used to make the specimens. From this aliquot, dilutions are made from which the standard is taken. With this method, leachate samples can be counted relative to standards without the need for absolute standards, detector efficiencies, or decay corrections. —Analysis of the leachate for the species of interest shall be conducted by standard methods and using appropriate calibration standards. If necessary, standards should be prepared to match the matrix elements in the samples. For radioactive specimens, a series of waste reference solutions can be prepared by diluting an aliquot of the original solution (or waste) that was used to make the specimens 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, detector efficiencies, or decay corrections.

9.8 *Standard Test*—One or more ALTs with an equivalent specimen shall be conducted at 20°C for use as a standard for comparison with ALTs conducted at other temperatures and ALTs conducted 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.

9.9 *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 help detect contamination that may occur during the procedure or provide background levels for leachants that contain the species of interest.

10. Calculations

10.1 *Incremental Fraction Leached*—Several parameters shall be calculated from the data obtained. The incremental fraction leached (IFL) is calculated by the following equation: —The incremental fraction of species i leached (*IFL*) during test interval n is calculated by using Eq 2:

$$IFL = \frac{_i a_n}{_i A_0} \qquad (2)$$

0

where:

$_i a_n$ = the quantity of species i observed in the leachate at any given time interval. This value is corrected for radioactive decay to the time of the beginning of the test. the quantity of species i measured in the leachate from the nth test interval, and

$_iA_0$ = the source term, the total original quantity contained in the leaching specimen at the beginning of the test. ~~the quantity~~ of species i in the specimen at the beginning of the test.

~~10.1.1 The rate of release for any interval can be calculated by dividing IFL by the elapsed time of the interval. The rate can then be divided by the surface area of the specimen to obtain the fraction released per square cm per s.~~

In the case of radionuclide i, both terms must be corrected for radioactive decay to the beginning of the test.

10.1.1 It may be necessary to calculate the value of $_ia_n$ from the measured solution concentration using the leachant volume. In that case, the uncertainty in the measured concentration and the uncertainty in the leachant and leachate volumes must be taken into account. (See Annex A2.)

10.1.2 The average rate of release for any interval can be calculated by dividing *IFL* by the duration of that interval. The rate can then be divided by the surface area of the specimen to obtain the average fraction released per area per time. This allows comparisons of tests conducted with samples having different surface areas.

10.2 *Cumulative Fraction Leached*—~~Another parameter is cumulative fraction leached, which is calculated as:~~ —The cumulative fraction of species i leached through the jth interval (CFL_j) is calculated by using Eq 3:

$$CFL = \frac{\Sigma_i a_n}{_iA_0} \tag{3}$$

$$CFL_j = \frac{\sum_{n=1}^{j} {}_ia_n}{_iA_0} \tag{3}$$

~~which is the sum of the fractions of a species of interest leached during the test. This value for each interval, plotted against cumulative time, provides a useful means of graphically comparing data to other test results and to modeling results. An example of this type of plot is shown in Fig. 2~~ $0 = n=1j\ IFLj$

NOTE 2—The indices for the species and interval are excluded for convenience hereafter.

10.2.1 Plotting the *CFL* value for each interval against the cumulative time provides a graphical comparison of data from various tests with each other and with modeling results. An example of this type of plot is shown in Fig. 1.

~~10.3 Diffusion Coefficient—This test method has an associated computer program that calculates a best fit effective diffusion coefficient based on the solution to diffusion from the finite cylinder~~

10.3 *Effective Diffusion Coefficient*— The results of this test method can be used to determine the effective diffusion coefficient (D_e) for the release of the species of interest based on a model. A computer program has been developed at Brookhaven National Laboratory to calculate a best fit effective diffusion coefficient (D_e) based on the equations for diffusion from a semi-infinite medium or from a finite cylinder (4,~~–~~). The ALT computer program also evaluates the possible influence of partitioning and solubility limits on the diffusive release. That program is available from ASTM for use with this test method (2) ; see also **(5, 6)** ~~. The computer program calculates~~ . *The computer program determines the value of the effective diffusion coefficient by regressing analytical expressions for diffusion from a semi-infinite solid and from a finite cylinder to the CFL* determined from the test results. The analytical expressions are provided in Annex A1. The uncertainty in the diffusion coefficient can be calculated using the formula for diffusion from a semi-infinite solid.
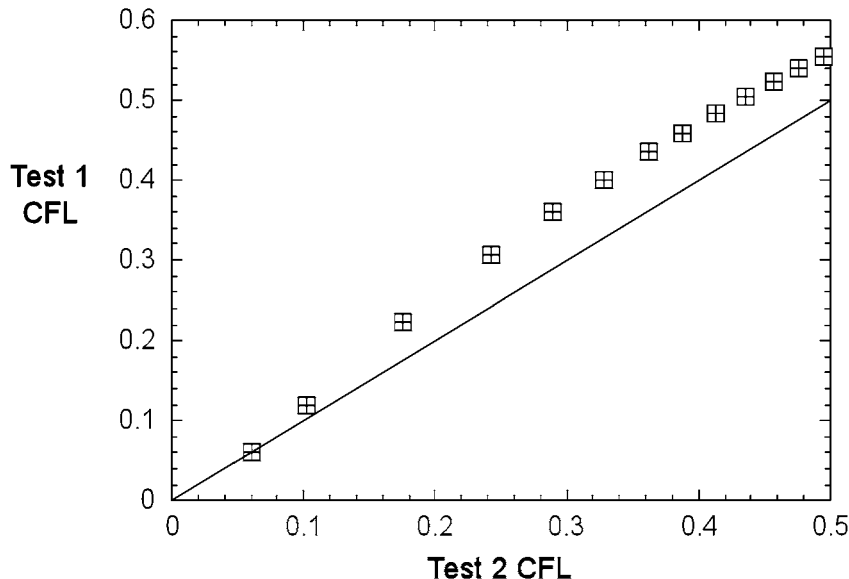

FIG. 2 Plot of the Results of Test 1 versus the Results of Test 2