

## Standard Test Method for Measuring Waste Glass or Glass Ceramic Durability by Vapor Hydration Test<sup>1</sup>

This standard is issued under the fixed designation C1663; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 The vapor hydration test method can be used to study the corrosion of a waste forms such as glasses and glass ceramics<sup>2</sup> upon exposure to water vapor at elevated temperatures. In addition, the alteration phases that form can be used as indicators of those phases that may form under repository conditions. These tests; which allow altering of glass at high surface area to solution volume ratio; provide useful information regarding the alteration phases that are formed, the disposition of radioactive and hazardous components, and the alteration kinetics under the specific test conditions. This information may be used in performance assessment (McGrail et al, 2002 (1)<sup>3</sup> for example).

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

1.3 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:<sup>4</sup>

- C162 Terminology of Glass and Glass Products
- D1125 Test Methods for Electrical Conductivity and Resistivity of Water
- D1193 Specification for Reagent Water
- D1293 Test Methods for pH of Water
- E177 Practice for Use of the Terms Precision and Bias in

#### **ASTM Test Methods**

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

## 3. Terminology

3.1 *Definitions:* 

3.1.1 *alteration layer*—a layer of alteration products at the surface of specimen. Several distinct layers may form at the surface and within cracks in the glass. Layers may be comprised of discrete crystallites. The thickness of these layers may be used to estimate the amount of glass altered.

3.1.2 *alteration products*—crystalline or amorphous phases formed as a result of glass interaction with an aqueous environment by precipitation from solution or by in situ transformation of the chemically altered solid.

3.1.3 *glass*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing. C162

3.1.4 *glass ceramic*—solid material, partly crystalline and partly glassy, formed by the controlled crystallization of a glass. C162

3.1.5 glass transition temperature—on heating, the temperature at which a glass transforms from an elastic to a viscoelastic material, characterized by the onset of a rapid change in thermal expansivity. C162

3.1.6 *immobilized low-activity waste*—vitrified low-activity fraction of waste presently contained in Hanford Site tanks.

3.1.7 *performance assessment*—examines the long-term environmental and human health effects associated with the planned disposal of waste. Mann et al, 2001 (2)

3.1.8 *sample*—initial test material with known composition.

3.1.9 *specimen*—specimen is a part of the sample used for testing.

3.1.10 *traceable standard*—a material that supplies a link to known test response in standards international units by a national or international standards body, for example, NIST.

3.2 Abbreviations:

3.2.1 DIW-ASTM Type I deionized water

3.2.2 EDS-energy dispersive X-ray spectroscopy

3.2.3 OM-optical microscopy

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<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

Current edition approved June 1, 2009. Published July 2009. DOI: 10.1520/C1663-09.

<sup>&</sup>lt;sup>2</sup> The precision and bias statements are only valid for glass waste forms at this time. The test may be (and has been) performed on other waste forms; however, the precision of such tests are currently unknown.

 $<sup>^{3}</sup>$  The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>&</sup>lt;sup>4</sup> 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.

3.2.4 *OM/IA*—optical microscope connected to an image analysis system

3.2.5 *PTFE*—polytetrafluoroethylene (chemical compound commonly referred to as Teflon)

3.2.6 SEM-scanning electron microscope

3.2.7 SiC paper-silicon-carbide paper

3.2.8 *TBD*—to be determined

3.2.9 *TEM*—transmission electron microscope

3.2.10  $T_{\rho}$ —glass transition temperature

3.2.11 *VHT*—vapor hydration test

3.2.12 WDS—wave-length dispersive spectroscopy

3.2.13 XRD-X-ray diffraction

3.2.14 %RSD—percent relative standard deviation

#### 4. Summary of Test Method

4.1 For the vapor hydration tests, glass or glass ceramic specimens (referred to generally as glass samples in this test method) are suspended from a support rod inside the test vessel with platinum wire. A volume of water determined by the volume of the test vessel and the test temperature is added to the vessel. The vessel is then sealed and placed in an oven at the desired test temperature and left undisturbed. After the desired test duration, the vessel is removed from the oven and the bottom of the vessel is cooled to condense the vapor in the vessel. Specimens are removed and examined with optical microscopy, XRD, SEM, and other analytical methods. The remaining glass or glass ceramic thickness is measured and alteration phases are identified.

#### 5. Significance and Use

5.1 The vapor hydration test can be used to study the corrosion of glass and glass ceramic waste forms under conditions of high temperature and contact by water vapor or thin films of water. This method may serve as an accelerated test for some materials, since the high temperatures will accelerate thermally activated processes. A wide range of test temperatures have been reported in the literature  $-40^{\circ}C$  (Ebert et al, 2005 (3), for example) to 300°C (Vienna et al, 2001 (4), for example). It should be noted that with increased test temperature comes the possibility of changing the corrosion rate determining mechanism and the types of phases formed upon alteration from those that occur in the disposal environment (Vienna et al, 2001 (4)).

5.2 The vapor hydration test can be used as a screening test to determine the propensity of waste forms to alter and for relative comparisons in alteration rates between waste forms.

## 6. Apparatus

6.1 *Test Vessels*—Stainless steel vessels with closure fitting with unique identifiers (on both vessel and lid), (for example, 22 mL vessels, rated for service at temperatures up to 300°C and maximum pressure 11.7 MPa (1700 psi)).<sup>5</sup>

6.2 *Balance(s)*—Any calibrated two-point (0.00 grams) balance.

6.3 *Convection Oven*—Constant temperature convection oven with the ability to control the temperature within  $\pm 2^{\circ}$ C.

6.4 *Temperature Monitoring Device*—Resistance thermometers or thermocouples, or both, with a strip chart recorder or a data logger for periodic monitoring of the temperature of the convection oven during the test duration. It is recommended that the maximum period between recorded temperature measurements be 0.5 h.

6.5 *Pipettes*—Calibrated pipettes. Pipette tips that have been precleaned, sterilized, or individually packaged to avoid contamination from handling.

6.6 *Torque Wrench*—Torque wrench capable of torques up to 230 N·m (170 lbf·ft).

6.7 *Vessel Holder*—Appropriate device/stand for holding vessels during tightening/loosening processes.

6.8 *Diamond Impregnated Saw*—High or low density diamond-coated wafering blade and low speed saw.

6.9 *Polishing Equipment*—Polishing equipment capable of polishing to 600 grit.

6.10 *Calipers*—Calipers that have been calibrated with traceable standards.

6.11 Optical Microscope with Image Analysis System.

6.12 *Chemically Inert Wire*—Wire used to suspend the specimens (such as 0.25 mm Pt wire).

6.13 *Support Rods*—Typically 1.5 mm diameter 304L stainless steel (or comparable material) rods bent to the shape shown in Fig. 2. Used to suspend specimens within the pressure vessel during tests.

6.14 *Non-Combustible Tray*—For water to quench vessel bottom after test termination.

6.15 *Storage Vessels*—Polyethylene or glass vessels for specimen storage.

6.16 Ultrasonic Bath.

6.17 pH Paper.

6.18 SiC Paper.

6.19 Non-Talc Surgical Gloves.

6.20 Glass Slides.

6.21 *PTFE Tape*—The type commonly used for household plumbing.

6.22 Tweezers/Forceps.

6.23 Scissors.

6.24 *Glue or Thermoplastic Adhesive*, for attaching samples and specimen to glass slides (for example, crystal-bond, super-glue, or wax).

6.25 pH Probe, calibrated with traceable standards.

<sup>&</sup>lt;sup>5</sup> Series 4701-14 22 mL Vessels from Parr Instrument Co., 211 53rd St., Moline, IL 61265, have been found satisfactory.

## 7. Reagents and Standards

7.1 ASTM Type I Water—Type I water shall have a minimal electrical resistivity of 18.0 M $\Omega$ ·cm at 25°C (see Specification D1193).

7.2 Solvents—Absolute ethanol and reagent grade acetone.

7.3 Reagent Grade HNO<sub>3</sub>—6 M HNO<sub>3</sub> and 0.16M HNO<sub>3</sub>.

## 8. Hazards

8.1 All appropriate precautions for operation of pressurized equipment must be taken. To ensure safe operation, the test vessels should be rated to withstand the vapor pressure of water at the test temperature with an appropriate safety factor.

#### 9. Specimen Preparation

9.1 Glass or glass ceramic specimens are prepared from annealed bars (for example, anneal 2 hours at a temperature slightly above the glass transition temperature with subsequent slow cooling to room temperature inside the oven, care must be taken not to induce phase changes during annealing) using a diamond impregnated saw and SiC papers with different grits.<sup>6</sup> During the specimen preparation, it is important to use low cutting force and saw speed (dependent on sample). Rough surface and damaged edges of the samples indicate rough machining. This may cause cracks to form within the glass or glass ceramic specimen during the sample preparation and decrease the reproducibility of the test. Preparation of the specimen may vary according to the equipment used. Usually specimens are prepared slightly larger and subsequently polished to the desired dimensions. However, with certain types of diamond impregnated saws, it is possible to prepare specimens with the desired dimensions and polish the surface directly with 600 grit SiC paper. The details of one example of preparation technique are given below. These steps (9.1.1 -9.1.5) are only given as an example and can be adjusted to yield the desired specimen dimensions and surface finish.

9.1.1 Cut annealed glass or glass ceramic bars with a diamond-impregnated saw to roughly the dimensions 10.3 by 10.3 by 30–50 mm (with appropriate cooling fluid).

9.1.2 Slice from the square glass or glass ceramic bar using a diamond impregnated saw a roughly 1.6 mm-thick specimen (10.3 by 10.3 by 1.6 mm) (with appropriate cooling fluid).

9.1.3 Polish to roughly the dimensions 10.2 by 10.2 by 1.55 mm using 240 grit SiC (with appropriate cooling fluid).

9.1.4 Polish to roughly the dimensions 10.1 by 10.1 by 1.51 mm using 400 grit SiC (with appropriate cooling fluid).

9.1.5 Polish to the dimensions 10.0 by 10.0 by 1.50 mm using 600 grit SiC paper (with appropriate cooling fluid).

9.2 Ultrasonically clean specimen in ethanol for 2 min, decant, and discard ethanol.

9.3 Ultrasonically clean specimen in ethanol for 4 min, decant, and discard ethanol.

9.4 Dry specimen in an oven at 90°C for 15 min.

9.5 Examine each specimen with OM and record observations concerning specimen surface and heterogeneity (streaks, inclusions, and scratches).

## 10. Test Vessel Cleaning

10.1 *Cleaning of Stainless Steel Vessels and Support Rods:* 10.1.1 Degrease vessels and lids with acetone. (This step is performed only with new vessels.)

10.1.2 Use 400 grit SiC paper to remove debris and oxidation from inside parts of previously used vessels and rinse with DIW.

10.1.3 Ultrasonically clean vessels, lids, and stainless steel supports in ethanol for 5 min, decant and discard ethanol.

10.1.4 Rinse vessels, lids, and supports by immersing 3 times in fresh DIW.

10.1.5 Soak vessels, lids, and supports in reagent grade 0.16M HNO<sub>3</sub> at 90°C for 1 h.

10.1.6 Rinse vessels, lids, and supports by immersing 3 times in fresh DIW.

10.1.7 Soak vessels, lids, and supports in fresh DIW at  $90^{\circ}$ C for 1 h.

10.1.8 Rinse vessels, lids, and supports by immersing in fresh DIW.

10.1.9 Fill vessels (with supports placed inside) to 80-90 % of capacity with fresh DIW. Place lids on vessels. Do not tighten. Place them in an oven at  $90^{\circ}$ C for a minimum of 16 h.

10.1.10 After cooling, measure the pH of the DIW using the pH probe according to Test Methods D1293. If the pH value is not within the 5.0 to 7.0 range, repeat rinsing from step 10.1.6.

10.1.11 Dry vessels, lids, and supports in an oven at 90°C for at least 1 h.

10.1.12 Store vessels, lids, and supports in a clean, dry, environment until use.

10.2 Cleaning of PTFE Gaskets:

Note 1—Other gasket materials may be used, so long as they do not significantly impact the reactions between water and the sample. This may be an important consideration in high radiation environments.

10.2.1 Bake PTFE gaskets for 1 week at 200°C. (This step is performed only with new PTFE gaskets.)

10.2.2 Soak the gaskets in reagent grade 6 M HNO<sub>3</sub> at  $50 \pm 5^{\circ}$ C for 4 h.

10.2.3 Rinse the gaskets by immersing in fresh DIW 3 times.

10.2.4 Immerse the gaskets in fresh DIW and boil for 30 min.

10.2.5 Rinse by immersing the gaskets in fresh DIW.

10.2.6 Soak the gaskets for 8 h in fresh DIW at 80°C.

10.2.7 Rinse the gaskets by immersing in fresh DIW.

 $10.2.8\,$  Immerse the gaskets in fresh DIW and boil for 30 min.

10.2.9 Rinse the gaskets by immersing 3 times in fresh DIW (container with gaskets is filled 3 times with fresh DIW).

10.2.10 Submerge gaskets in fresh DIW. Measure pH using the pH probe according to Test Methods D1293. If the pH value is not within the 5.0 to 7.0 range, repeat step 10.2.9.

10.2.11 Dry gaskets in an oven at 90°C and store in a clean environment until needed.

<sup>&</sup>lt;sup>6</sup> For detailed discussion of the influence of surface finish on corrosion see Mendel et al, 1984 (5). Some example results of vapor hydration tests with varying surface finish are reported in Jiricka et al, 2001 (6).

## 11. Calibration

11.1 *Calibrations*—Initially calibrate all measurement instruments used in this test. Verify the calibrations during use of the instrument to indicate possible errors due to instrumental drift.

11.2 Calibration and Standardization Schedule:

11.2.1 *Temperature Measurement Devices*—Calibrate at least annually with traceable standards or an ice/boiling water bath.

11.2.2 *Balance*—Standardize with traceable standard masses on a regular basis. If a deviation in mass measurement is identified, all measurements since the last accurate standard measurement made with the balance must be marked appropriately. Have balance calibrated on an annual basis.

11.2.3 Water Purification System—Calibrate at least annually following the manufacturer's instructions. Standardize with the 10 M $\Omega$ ·cm at 25°C resistivity calibration cell (or equivalent) on the water purification system (see Test Methods D1125).

11.2.4 *Calipers*—Calibrate with traceable standards at least annually.

11.2.5 *Image Analysis System*—Calibrate with a micrometric calibration ruler designed for image analysis calibration.

## 12. Procedure for Conducting the Vapor Hydration Test

12.1 Amount of Water Needed—In order to conduct reliable VHTs, the amount of water added to the vessel must be sufficient to saturate the vessel's volume at the test temperature and provide excess water that can condense on the test specimen surface. The amount of water needed for saturating a 22 mL vessel was calculated assuming ideal conditions,  $H_2O$ ,  $N_2$ , and  $O_2$ . It consists of an amount of water needed to saturate the vessel at a given temperature (determined from steam tables) plus an additional 0.05 mL of excess water needed for each specimen with dimensions of 10.0 by 10.0 by 1.5 mm (surface area 260 mm<sup>2</sup>). The volume of the specimen assembly (specimen, support rod, and chemically inert wire) was not accounted for in steam saturation calculations. Similar calculations should be performed if using vessels with different volumes.

## 12.2 Test Set-Up:

12.2.1 Verify that the convection oven is at the desired temperature and temperature-monitoring device has been calibrated.

12.2.2 Verify that the stainless steel test vessels have been cleaned according to Section 10. The vessels must have a unique number permanently affixed to the vessel cap and bottom.

12.2.3 Wind the threads of the vessel closure fittings with PTFE tape to prevent binding of the closure threads (roughly 2 wraps).

12.2.4 Verify that a calibrated balance accurate to  $\pm 0.01$  g is available.

12.2.5 Verify that a pipette and fresh DIW are available.

12.2.6 Verify that a torque wrench is available and set to the desired setting between 203 N·m and 230 N·m (150 lbf·ft to 170 lbf·ft).

12.2.7 Verify that the stainless steel support rods have been cleaned and thoroughly rinsed with ethanol according to Section 10.

12.2.8 Verify that the required data sheet and test parameters for the VHT are available, see Appendix X1 for an example data sheet.

12.2.9 Verify that the test specimens are prepared according to Section 9.

12.3 Test Start-Up Procedure:

12.3.1 Record the following information on a VHT Data Sheet: (1) Test number, (2) Test temperature, (3) Planned test duration, (4) Vessel and cap identification number, and (5) Sample/specimen identification.

12.3.2 Wear non-talc gloves (or equivalent covering if in remote environment) and use tweezers when tying the specimen as shown in Fig. 1.

12.3.3 Place the support with specimen in proper position inside the assigned vessel as shown in Fig. 2.

12.3.4 Place a clean PTFE gasket (or equivalent) inside a vessel cap.

Note 2—The PTFE gasket should be replaced by a different material in those cases where high radiation fields and long test times are expected to introduce sufficient fluorine onto the test specimen to influence test result.

12.3.5 Place vessel bottom, cap and vessel closure (not assembled) on a balance accurate to  $\pm 0.01$  g and record the mass.

12.3.6 Using a pipette, add the targeted amount (in mL) of fresh DIW (see Table 1 as example for 22 mL vessel with one sample) to the vessel and record the mass. Verify that the amount of water added is the difference between dry assembly and assembly including water.

12.3.7 Place the assembly in an appropriate device/stand for holding vessels during tightening and tighten with torque wrench to between 203 N·m and 230 N·m (150 lbf·ft to 170 lbf·ft).

12.3.8 Record the oven temperature and calibration status of the temperature monitoring device on the data sheet.

12.3.9 Place the test vessel inside the oven and record the time and date IN on the data sheet.

## 12.4 During Test:

12.4.1 It is allowed, but not required, to check the test vessels for fluid leakage during testing (for example, after one day) by quickly removing the vessel from the oven, weighing



FIG. 1 Glass Specimen Suspended with Thin Pt Wire (roughly 0.2 mm diameter)



FIG. 2 Apparatus for Conducting Vapor Hydration Tests

TABLE 1 Vapor Pressure and Amounts of Water Needed for the VHT with 22 mL Vessels for Temperatures from 5°C to 300°C

T [°C]	P [MPa] <sup>A</sup>	H <sub>2</sub> O [g]	T [°C]	P [MPa] <sup>A</sup>	H <sub>2</sub> O [g]
5	6.1E-04	0.05	110	1.4E-01	0.07
10	8.8E-04	0.05	115	1.7E-01	0.07
15	1.2E-03	0.05	120	2.0E-01	0.08
20	1.7E-03	0.05	125	2.3E-01	0.08
25	2.4E-03	0.05	130	2.7E-01	0.09
30	3.2E-03	0.05	140	3.6E-01	0.10
35	4.3E-03	0.05	150	4.8E-01	0.11
40	5.7E-03	0.05	160	6.2E-01	0.13
45	7.4E-03	0.05	170	8.0E-01	0.15
50	9.6E-03	0.05	180	1.0E+00	0.18
55	1.2E-02	0.05	190	1.3E+00	0.21
60	1.6E-02	0.05	200	1.6E+00	0.25
65	2.0E-02	0.05	210	1.9E+00	0.29
70	2.5E-02	0.05	220	2.3E+00	0.35
75	3.1E-02	0.06	230	2.8E+00	0.42
80	3.9E-02	0.06	240	3.4E+00	0.49
85	4.8E-02	0.06	250	4.0E+00	0.59
90	5.8E-02	0.06	260	4.7E+00	0.70
95	7.0E-02	0.06	270	5.5E+00	0.83
100	8.5E-02	0.06	280	6.4E+00	0.98
105	1.0E-01	0.07	300	7.5E+00	1.39

<sup>A</sup>"NBS/NRS Steam Tables," Lester Haar et al, Hemisphere Publishing Corp., 1984, pp. 9–14.

it on a calibrated balance, and returning it to the oven. This is particularly important in long-term tests when water loss has been found to be significant. Record the results on the data sheet.

12.4.2 Record the temperature during the test period in 0.5 h intervals with a calibrated measurement device.

## 12.5 Test Termination:

12.5.1 Record the temperature, date and time out of the oven on the data sheet when the test is complete.

12.5.2 Place a piece of refractory block on a calibrated balance accurate to  $\pm 0.01$  g and tare the balance.

12.5.3 Remove the test vessels, one at a time, and place on the refractory piece on the balance. Record the mass of each test vessel. The difference in mass between test initiation and termination indicates the amount of water evaporated during the test.

12.5.4 Place the vessel in cold water (roughly 20 mm deep) for approximately 20 min. For temperatures higher than 200°C ice water is recommended.

12.5.5 Place assembly in an appropriate device/stand for holding vessels during loosening. Loosen and remove the closure fitting.

12.5.6 Open the vessel, remove the test specimen, allow specimen to dry. It is recommended that the time between terminating the test and opening the vessel does not exceed 30 min due to the potential hygroscopic nature of the specimen's surface.

12.5.7 Record observations pertaining to specimen(s) surface, drying pattern, secondary phase development, and overall integrity of the specimen(s).

12.5.8 Place the specimen(s) into the prelabeled container(s). Labels should include test number (indicating the type of sample tested), test temperature, and test period.

12.5.9 Note the presence or absence of fluid remaining on the bottom of the test vessel and on specimens.

12.5.10 Measure and record the approximate pH of the fluid in the test vessel with pH paper capable of indicating pH in the range from 5 to 10.

#### 13. Interpretation of Results

13.1 Specimen Analyses for Alteration Products:

13.1.1 Specimens can be analyzed for the presence of secondary phases by visual observation and OM; type of secondary phases by XRD, OM, SEM/EDS, microprobe/WDS, and/or TEM/EDS; remaining glass layer thickness by OM/IA

or SEM/EDS (see Section 13.3); alteration layer thickness by SEM/IA or OM/IA (see section 13.4), and extent of corrosion (see section 13.5). Section 13.2 describes the advantages and disadvantages of the two methods for sample analyses and suggests how to select between the two methods.

#### 13.2 Selection of Method for Sample Analyses:

13.2.1 To determine the amount of glass or glass ceramic converted into alteration products, it is possible to measure the remaining glass thickness or the thickness of alteration layers. Generally, the method with the highest precision is preferred. Jiricka et al, 2001 (6) performed detailed analyses of the relative merits of each technique. In measuring the alteration layer, the layer density is typically lower than that of the glass or glass ceramic and varies widely with glass or glass ceramic composition and test condition; additionally, the alteration layers are non-uniform in thickness and density. In measuring the remaining glass or glass ceramic thickness, the measurement of a relatively small difference in thickness can have low measurement precision. The exact thickness at which the precision of remaining glass or glass ceramic measurement becomes preferable depends on the precision of the specific equipment used to measure samples. An example calculation to determine the appropriate method of sample analyses follows.

13.2.2 To illustrate the method for selecting sample analyses techniques we use a specimen reported by Vienna et al, 2001 (4)—HLP-51 glass tested at 300°C for 2 days. An optical micrograph of the cross section of this specimen is shown in Fig. 3. For measurement of this sample, we can choose between OM and SEM, and measurement of remaining glass or glass ceramic thickness or alteration products thickness. Ten measurements of the remaining glass or glass ceramic thickness return a thickness of  $0.39 \pm 0.06$  mm. The precision of the OM/IA was determined to be 0.003 mm while the precision of the SEM/IA method (not shown) was found to be 1 %, relative, or 0.004 mm for a measurement of 0.4 mm. In this case, the precision of remaining glass or glass ceramic thickness measurement by both methods are roughly equal and an order of magnitude less that the variation in the sample. So, either method could be performed without impacting measurement precision. The other key decision is the measurement of remaining glass or glass ceramic thickness or the thickness of alteration products. For this sample, the alteration products thickness is found to be  $0.91 \pm 0.35$  mm on the top and 1.14

 $\pm$  0.45 mm on the bottom. Clearly, in this particular sample there is significantly more variation in alteration products thickness making the measurement less precise than measuring remaining glass or glass ceramic thickness. In addition, the overall sample thickness grew from 1.52  $\pm$  0.008 mm before testing to 2.51  $\pm$  0.45 mm after testing. The mass grew only slightly due to the addition of water so the density of the alteration layer is significantly lower than the initial glass or glass ceramic. This difference in density can be corrected for as described in section 13.5.3, but, adds to imprecision.

13.2.3 For samples with lower variation in alteration layer thickness and lower corrosion extent, however, the measurement of alteration layer thickness by SEM with ~1 % relative error becomes a more precise method of determining the extent of specimen conversion to alteration products.

# 13.3 Measuring the Remaining Glass or Glass Ceramic Layer Thickness with OM/IA or SEM/IA:

13.3.1 Depending on the amount of corrosion and specimen condition, an epoxy resin can be used to mount the sample before cutting and polishing. If analysis of secondary phases is required, it is recommended to prepare the cross section of the specimen by dividing it into two parts with a dry diamond impregnated saw and store one part for further analysis. The cross section of the sample for OM/IA or SEM/IA evaluation is polished down to roughly 2 mm of thickness and 600 grit surface finish on both sides. However, the preparation technique may vary according to the equipment used. The following specimen preparation procedure is only recommended and can be adjusted to yield the required results.

13.3.1.1 Cross section preparation for OM/IA or SEM/IA evaluation: The side with the fresh cut is hand held polished to 600 grit surface finish.

13.3.1.2 The specimen is glued with the polished side to the microscopic glass or glass ceramic and allowed to dry in blowing hot air for about 10 min.

13.3.1.3 The other side of the specimen is cut with a diamond impregnated saw and polished with a 400 grit SiC paper to create a section roughly 2 mm thick.

13.3.1.4 Finally, the specimen is polished to a thin section roughly 1 mm thick with 600 grit surface finish, which enables the use of transmitted light during the OM/IA evaluation. Finer finishes may be desired for SEM/IA evaluation but are typically not required.



FIG. 3 OM/IA Measurement of HLP-51 Glass after 2 Days of VHT at 300°C (Vienna et al, 2001 (4))

13.3.2 The remaining glass or glass ceramic layer thickness is determined using OM/IA or SEM/IA. The thickness of the remaining glass or glass ceramic layer is determined by performing at least 10 measurements equally distributed across the areas of the specimen free of cracks (as illustrated in Fig. 3). The result of this step is the average thickness of the remaining glass or glass ceramic layer and the standard deviation of the 10 measurements.

13.3.3 An increase in length will occur if the cut is not exactly perpendicular. This increase is given by:

$$d_r = d_\phi \cos\phi \tag{1}$$

where:

- $d_r$  = the true remaining glass or glass ceramic layer thickness,
- $\phi$  = the angle that the cross section makes with the plane perpendicular to the specimen surface, and
- $d_{\phi}$  = the estimated remaining glass or glass ceramic layer thickness measured at an angle of  $\phi$ .

13.3.3.1 A cut of 20° off perpendicular would increase  $d_r$  estimates by roughly 6 %.

13.4 Measuring the Thickness of Alteration Layer with SEM/IA or OM/IA:

13.4.1 As described in section 13.2 it is typically beneficial to measure the thickness of alteration products layer only for specimens with relatively small alteration extents. It is therefore more typical to use SEM/IA analyses rather than OM/IA analyses since SEM measurement precision is much better for relatively small lengths (for example, if OM/IA has an uncertainty of  $\pm 0.003$  mm and SEM/IA has an uncertainty of  $\pm 1$  % then for absolute thicknesses below 0.3 mm SEM/IA is more precise). If analysis of secondary phases is required, it is recommended to prepare the cross section of the specimen by dividing it into two parts with a dry diamond impregnated saw and store one part for further analysis. The cross section of the sample for SEM/IA or OM/IA evaluation is polished down to roughly 2 mm of thickness and 600 grit surface finish on both sides (if OM will be used). However, the preparation technique may vary according to the equipment used. The following specimen preparation procedure is only recommended and can be adjusted to yield the required results.

13.4.1.1 Cross section preparation for SEM/IA or OM/IA evaluation: The side with the fresh cut is hand held polished to 600 grit surface finish. It is critical to ensure that portions of the alteration layer are not removed during sectioning and polishing steps.

13.4.1.2 The specimen is glued with the polished side to the microscopic glass or glass ceramic and allowed to dry in blowing hot air for about 10 min.

13.4.1.3 The other side of the specimen is cut with a diamond impregnated saw and polished with a 400 grit SiC paper to create a section roughly 2 mm thick.

13.4.1.4 Finally, the specimen is polished to a thin section roughly 1 mm thick with 600 grit surface finish, which enables the use of transmitted light during the OM/IA evaluation. Finer finishes may be desired for SEM/IA evaluation but are typically not required.

13.4.1.5 The sample is coated appropriately for the SEM instrument if SEM will be used.

13.4.2 The alteration layer thickness is determined using SEM/IA or OM/IA. Care must be taken to identify the alteration products from the glass as sometime the contrast is not great. The thickness of the remaining glass or glass ceramic layer is determined by performing at least 10 measurements equally distributed across the alteration layer on each side of the remaining glass or glass ceramic core. The result of this step is the average thickness of the alteration layer and the standard deviation of the 10 measurements from each side of the sample.

13.4.3 An increase in length will occur if the cut is not exactly perpendicular. This increase is given by:

$$l_r = d_\phi \cos\phi \tag{2}$$

where:

- $d_r$  = the true remaining glass or glass ceramic layer thickness,
- $\phi$  = the angle that the cross section makes with the plane perpendicular to the specimen surface, and
- $d_{\phi}$  = the estimated remaining glass or glass ceramic layer thickness measured at an angle of  $\phi$ .

13.4.3.1 A cut of 20° off perpendicular would increase  $d_r$  estimates by roughly 6 %.

13.5 Extent of Corrosion Data Evaluation:

13.5.1 The mass of specimen altered per unit surface area is given by:

$$m_a = \frac{1}{2} d_i \rho \left( 1 - \frac{d_r}{d_i} \right) = \frac{m_i}{2w_i l_i} \left( 1 - \frac{d_r}{d_i} \right) \tag{3}$$

where:

 $w_i, d_i, l_i$  = specimen width, thickness, and length, respectively,

 $d_i$  = initial specimen thickness,

- $d_r$  = average thickness of remaining glass or glass ceramic layer,
- $m_i$  = initial specimen mass,

$$m_a$$
 = mass of glass or glass ceramic converted to alteration products per unit surface area, and

 $\rho$  = glass or glass ceramic density.

13.5.1.1 In the case where the alteration layer thickness  $(d_a)$  is measured instead of  $d_r$ , the calculation is the same once  $d_a$  is used to estimate  $d_r$ . The simplest method is to assume the alteration layer is of the same density as the initial glass or glass ceramic and no mass change occurs (for example, mass of water in the alteration layer is negligible). These assumptions lead directly to the relationship:  $d_r = d_i - 2d_a$ . The difference in density between the glass or glass ceramic and its alteration products can be taken into account using the final sample thickness  $(d_f)$  leading to:  $d_r = d_f - 2d_a$ . These methods of estimating  $d_r$  lead to the following relationships:

$$m_a = \rho \, d_a \tag{4}$$

$$m_a = \frac{\rho}{2} \left( 2d_a - d_f + d_i \right) \tag{5}$$

13.5.2 *Example Calculation Using Thickness of Remaining Glass or Glass Ceramic:* 

13.5.2.1 A specimen of given size was altered during VHT so that the thickness of the samples decreased from 1.52 mm to 0.39 mm (see example in section 13.2). Eq 3 is used to calculate the mass of specimen converted to alteration products per unit surface area.

$$w_i = 9.98 \text{ mm}$$
  
 $l_i = 10.0 \text{ mm}$   
 $d_r = 0.39 \text{ mm}$   
 $m_i = 0.37 \text{ g}$   
 $d_i = 1.52 \text{ mm}$ 

$$m_{a} = \frac{m_{i}}{2w_{i}l_{i}} \left(1 - \frac{d_{r}}{d_{i}}\right) = \frac{0.37}{2 \cdot 9.98 \cdot 10.0 \cdot 1 \times 10^{-6}} \left(1 - \frac{0.39}{1.52}\right) = 1378 \frac{\text{g}}{\text{m}^{2}}$$
(6)

13.5.3 Example Calculation Using Thickness of Alteration Layer:

13.5.3.1 The  $m_a$  value obtained using alteration layer thickness, corrected for differences in density between the unaltered glass and the alteration products layer is given by:

$$\rho = 2.7218 \text{ g/cm}$$
  
 $d_a = 1.03 \text{ mm}$   
 $d_i = 1.52 \text{ mm}$   
 $d_f = 2.51 \text{ mm}$ 

$$m_a = \frac{\rho}{2} \left( 2d_a - d_f + d_i \right) = \frac{2\,721\,800}{2} \left( 2 \cdot 1.03 \times 10^{-3} - 2.51 \times 10^{-3} + 1.52 \times 10^{-3} \right) = 1456 \,\frac{g}{m^2}$$
(7)

#### 13.5.4 Uncertainties in Reported m<sub>a</sub> Values:

13.5.4.1 To calculate uncertainty in  $m_a$  determined via Eq 3 through Eq 7, a propagation of error analysis is performed. For uncorrelated random errors, the standard deviation of a function  $f(x_1, x_2, ..., x_n)$  is given by:

$$\sigma_f = \sqrt{\sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 \sigma_i^2} \tag{8}$$

where,  $\sigma_f$  is the standard deviation of function f,  $x_i$  is the parameter i, and  $\sigma_i$  is the standard deviation of parameter i. Assuming that the error in density measurement is negligible and substituting Eq 3 into Eq 8 yields the standard deviation of  $m_a$ :

$$\sigma_{ma} = \frac{1}{2} \sqrt{\left(\sigma_{di}^2 + \sigma_{dr}^2\right) \rho^2}$$
(9)

where,  $\sigma_{di}$  is the standard deviation of  $d_i$  measurement and  $\sigma_i$  is the standard deviation of  $d_r$  measurement. Using the same example, first discussed in section 13.2, the  $\sigma_{ma}$  is 81.74 g/m<sup>2</sup> (with a  $\rho$  of 2.7218 g/cm<sup>3</sup>). With a two standard deviation estimated measurement uncertainty, the  $m_a$  of this glass is 1378 ± 163 g/m<sup>2</sup> when measured by OM/IA of the remaining glass layer.

13.5.4.2 To estimate the  $\sigma_{ma}$  when analyzing the alteration layer thickness, we will consider the method that accounts for density differences between the alteration layer and assume that the uncertainty in glass density is negligible. Combining Eq 7 and Eq 8 yields:

$$\sigma_{ma} = \frac{1}{2} \sqrt{\left(4\sigma_{da}^2 + \sigma_{df}^2 + \sigma_{dl}^2\right)\rho^2}$$
(10)

where,  $\sigma_{da}$  and  $\sigma_{df}$  are the standard deviations for  $d_a$  and  $d_f$  measurements, respectively. Using the same example, first

discussed in section 13.2, the  $\sigma_{ma}$  is 694 g/m<sup>2</sup> (with a  $\rho$  of 2.7218 g/cm<sup>3</sup>). With a two standard deviation estimated measurement uncertainty, the  $m_a$  of this glass is 1456 ± 1388 g/m<sup>2</sup> when measured by OM/IA of the altered layer.

#### 13.6 Test Evaluation:

13.6.1 Depending upon the purpose for performing the test, the following conditions indicate that the test should be repeated:

13.6.1.1 Loss of greater than 50 % of the added water.<sup>7</sup> It is determined from the difference in whole assembly mass recorded at the test initiation and termination.

13.6.1.2 Specimen slips from the platinum holder and falls to the vessel bottom.

13.6.1.3 Brief fluctuations from the desired temperature are unavoidable when specimens are placed into or removed from the oven. However, cumulative time of these fluctuations greater than  $\pm 2^{\circ}$ C of the target temperature must not exceed 5 % of the test period.

13.6.1.4 A post test solution pH of greater than 10 may signify the water from the sample surface dripped onto the vessel bottom. It is an indicator of possible reflux condition during the test.

13.6.2 If any of the indicators listed above (or any other observations of abnormal response) should be recorded on the Test Termination data sheet.

#### 14. Precision and Bias

14.1 Precision:

14.1.1 The data used to generate the measures of precision for VHT are the result of intra- and inter-laboratory testing described by Vienna et al, 2001 (4). These measures are typical of the methods as applied to the glasses or glass ceramics used in the tests, and are not all-inclusive with respect to other types of glasses. The measures of precision were determined in accordance with procedures in Practices E177 and E691. As described above, each measurement is reported with a standard deviation ( $\sigma_{ma}$ ). As a rough indicator of measurement precision a grouped standard deviation ( $\sigma_g$ ) is estimated for each measurement of like sample, time, and laboratory. In addition, the mean value ( $<m_a>$ ) and percent relative standard deviation (%RSD, standard deviation divided by the mean and multiplied by 100 %) are reported for each group of measurements.

14.1.2 Tests with HLP-48 at 200°C—For HLP-48, a single glass bar was fabricated and a number of test specimens cut from the bar. The VHT response was measured by OM/IA of the remaining glass layer after 10 days and 20 days with samples prepared and measured at two laboratories (A and B). The results yield the  $<m_a >$ ,  $\sigma_g$ , and %RSD listed in Table 2.

14.1.3 *Tests with HLP Baseline Glasses at 250°C*—A series of four glasses HLP-01, -25, -26, and -43 were fabricated from the same target composition and tested as individual glasses. The VHT response was measured by OM/IA of the remaining glass layer after roughly 2, 3, 4, 5, and 6 days at 250°C at the same laboratory. The results are given in Table 3.

14.1.4 *Tests with HLP Baseline Glasses at 300°C*—A series of four glasses HLP-01, -25, -26, and -43 were fabricated from

 $<sup>^{7}</sup>$  Jirica et al, 2001 (6) demonstrate the effects of different amounts of water and conclude that 50 % loss should be the limit for acceptable testing.

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TABLE 2 Measurement and Uncertainty Summary for HLP-48 Glass Subjected to 200°C VHT at Two Laboratories

		Indiv	idual		Grouped					
#	Lab	time (d)	m <sub>a</sub> (g∕m²)	σ (g/m²)	<m<sub>a&gt; (g/m<sup>2</sup>)</m<sub>	σ <sub>g</sub> (g ∕m² )	%RSD			
1	А	10	789.2	6.24	790.6	2.0	0.3			
2	A	10	792.0	9.10						
3	В	10	738.8	6.57	778.1	55.6	7.1			
4	В	10	817.4	9.05						
5	A	20	1208.3	40.8	1266	81.8	6.5			
6	A	20	1324.0	174						
7	В	20	1141.1	18.4	1272	186	14.6			
8	В	20	1403.8	64.2						

TABLE 3 Measurement and Uncertainty Summary for HLP-01, -25, -26, and -43 Glass Subjected to 250°C VHT at One Laboratory

	Indiv	vidual			Grouped	
Glass ID	time (d)	m <sub>a</sub> (g∕m²)	σ <sub>ma</sub> (g∕m²)	<m<sub>a&gt; (g/m<sup>2</sup>)</m<sub>	σ <sub>g</sub> (g ∕ m² )	%RSD
HLP-01	2.0	547.4	3.52	613.5	45.9	7.5
HLP-25	2.0	653.7	4.36			
HLP-26	2.0	627.4	2.48			
HLP-43	2.1	625.3	4.50			
HLP-01	3.0	830.5	6.79	851.6	44.1	5.2
HLP-25	3.0	799.7	10.4			
HLP-26	3.0	892.6	11.08			
HLP-43	3.0	883.5	7.66			
HLP-01	4.0	987.5	9.23	1119	106	9.5
HLP-25	4.0	1245.3	16.0			
HLP-26	4.0	1138.0	19.5			
HLP-43	4.0	1103.5	11.0			
HLP-01	5.0	1384.2	27.7	1386	76.4	5.5
HLP-25	5.0	1489.2	48.7			
HLP-26	5.0	1367.0	20.5			
HLP-43	5.0	1305.3	40.0			
HLP-01	5.9	1561.4	73.3	1581	109	6.9
HLP-25	5.9	1738.2	85.3			
HLP-26	5.9	1486.0	18.4			
HLP-43	5.9	1538.6	50.4			

the same target composition and tested as individual glasses. The VHT response was measured by OM/IA of the remaining glass layer after roughly 0.5, 1.0, and 1.5 days at 300°C at the same laboratory. The results are given in Table 4 with the resulting  $\langle m_a \rangle$ ,  $\sigma_g$ , and %RSD.

14.1.5 Tests with LRM Glasses at 200°C—A series of three glasses HLP-47, -76, and -77 were fabricated from the same target composition and tested as individual glasses. The VHT response was measured by OM/IA of the remaining glass layer after roughly 5, 10, and 15 days at 200°C at two different laboratories. However, difficulties in measurement (described

by Schulz et al, 2000 (7) ), precluded the precise measurement of alteration from this glass. Precision was not determined for this glass.

14.1.6 *Precision Summary*—The  $\sigma_g$ , gives a rough estimate of method precision. In the above listed examples,  $\sigma_g$  varies from 2 g/m<sup>2</sup> to 186 g/m<sup>2</sup> and increases with increasing <m<sub>a</sub>>. To account for the change in  $\sigma_g$  with <m<sub>a</sub>> we consider the %RSD which ranges from 0.3 to 14.6 % with an average of 6.9 %.

14.2 Bias:

TABLE 4 Measurement and Uncertainty Summary	for HLP-01, -25, -26, and -43 Glass	Subjected to 300°C VHT at One Laboratory
---	-------------------------------------	--

	Indi	vidual	Grouped					
Glass ID	time (d)	m <sub>a</sub> (g∕m²)	σ (g/m²)	<m<sub>a&gt; (g/m<sup>2</sup>)</m<sub>	σ <sub>g</sub> (g∕m² )	%RSD		
HLP-01	0.5	865.5	3.01	798.2	70.4	8.8		
HLP-25	0.5	845.4	4.03					
HLP-26	0.5	769.0	5.81					
HLP-43	0.5	712.9	3.24					
HLP-01	1.0	1330.0	16.0	1313	40.0	3.0		
HLP-25	1.0	1357.4	56.8					
HLP-26	1.0	1263.8	36.4					
HLP-43	1.0	1301.6	25.1					
HLP-01	1.5	1794.1	95.3	1599	130	8.2		
HLP-25	1.5	1541.1	19.8					
HLP-26	1.5	1524.9	94.6					
HLP-43	1.5	1535.0	48.8					

14.2.1 No definitive statement about bias can be made for this test method since there is no reference material traceable for a national or international reference source (for example, traceable standard).

#### 15. Keywords

15.1 alteration; durability; glass; glass ceramics; hydration; hydrothermal; performance; rate; reaction; repository; test; vapor; waste; water

## APPENDIXES

#### (Nonmandatory Information)

## **X1. IDENTIFICATION OF ALTERATION PRODUCTS**

#### **INTRODUCTION**

Section 13 of this method gives a detailed description of the procedure for measuring the extent of corrosion by the VHT. However, as stated in Section 1, this test can be used to identify the alteration products that form as glass or glass ceramic reacts with water under the test conditions. There are many procedures that can be used to identify alteration products formed during VHT. A standard procedure for their identification and evaluation cannot be prescribed, since, different procedure(s) are required for different samples and types of information desired. This Appendix gives only a couple of examples of methods for alteration product identification and evaluation that may be used.

In all analyses methods care must be taken to ensure that the alteration products aren't significantly altered by specimen preparation technique. For example, the alteration products were formed primarily by precipitation from the over saturated solution. Therefore, introduction of water to the sample may redissolve some of the products. Conversely, after test termination there is typically liquid water on the specimen surface, drying of the specimen further could cause soluble components to precipitate.

## X1.1 X-Ray Diffraction of Ground Sample

X1.1.1 After sectioning the VHT specimen (as described in Section 13) a portion of the sample is ground in a mortar and pestle or other suitable device. The ground specimen is loaded into an X-ray powder diffractometer (XRD) for analyses. Typically, scans from 5° to 75° 2 $\Theta$  are used to evaluate the sample with relatively long hold time (for example, 6 to 40 s per step). This method will generate an XRD pattern that can be used to identify crystalline alteration products that exist in sufficient concentration and particle size for the analysis. Fig. X1.1 shows and example XRD pattern of three VHT specimens.

## X1.2 Scanning Electron Microscopy with Energy Dispersive Spectroscopy

X1.2.1 The use of SEM-EDS for characterization of alteration products is common. By this technique the specimen cross-section or surface are examined. The specimen cross section is prepared by the procedure described in Section 13. This specimen is then mounted on a conductive SEM sample holder with either the cross-section or surface facing up. Typically, the specimen is coated with a thin layer of C or Au to conduct electrons from the specimen surface to the stage. The specimen is then loaded into the SEM and analyzed using the method described in the SEM-EDS users' manual. The information that can be gained is a qualitative assessment of the composition of layers of alteration, the quantitative assessment of layer thicknesses, amount of porosity of the layers, and composition of individual crystalline alteration products. Fig. X1.2 gives an example SEM micrograph of a VHT specimen cross-section. Fig. X1.3 shows four SEM micrograph of a VHT specimen surface.





FIG. X1.1 Example XRD Pattern of Typical VHT Specimen



FIG. X1.2 Back-Scatter Electron Micrograph of Typical VHT Sample Specimen (showing 5 distinct layers with the locations of 6 EDS spectra as stars) (from Vienna et al, 2001 (4))



FIG. X1.3 Scanning Electron Micrograph of VHT Specimen Surface (showing a number of distinct phase morphologies) (from Vienna et al, 2001 (4))

**X2. MODEL VHT DATA SHEETS** 

## TABLE X2.1 Model Test Initiation and Termination Sheet for Single Sample

DATE OF INITIATION:														
	c] er		L] [ays]	days]	5			Specimen dimensions			าร	rt [g]	t H <sub>2</sub> O [g]	<sup>2</sup> 0 [g]
Test number including glass or glass ceramic type	Specimen numb	Planned Temp [°	Targeted H <sub>2</sub> O [m	Planned Duration [	Planned Date Ol	Cap #	Vessel #	Width [mm]	Length [mm]	Thickness [mm]	Mass [g]	Specimen + suppo	Assembly mass withou	Assembly mass + H
L														

## DATE OF TERMINATION: \_\_\_\_\_

#### TIME OF TERMINATION: \_\_\_\_\_

Test number including glass or glass ceramic type	Specimen number	Termination temperature [°C]	Date of termination	Time of termination	Assembly mass [g]	Specimen + support [g]	Hď	Remaining glass or glass ceramic [mm]	St. Dev. (remaining glass or glass ceramic)	Observation

Balance Cal. #/Expiration:

TC readout Cal. #/Expiration:

Notes/Comments:

Procedure is performed in:

Building: \_\_\_\_

\_\_\_\_\_ Room: \_\_\_\_\_ Operator: \_\_\_\_\_

Date: \_\_\_\_\_

## TABLE X2.2 Model VHT Initiation Data Sheet for a Set of Tests

DATE OF INITIATION:	TIME OF INITIATION:					OVEN NUMBER:							
	ē	U U U	ГJ	days]			s	pecimen	dimensio	าร	t H <sub>2</sub> O [g]	2 <sup>2</sup> 0 [g]	ų
Test number including glass or glass ceramic type	Specimen numbe	Planned Temp [°	Targeted H <sub>2</sub> O [m]	Planned Duration [d	Cab #	Vessel #	Width [mm]	Length [mm]	Thickness [mm]	Mass [g]	Assembly mass without	Assembly mass + H <sub>2</sub>	Position in Over
Balance Cal. #/Expiration:													
Procedure is performed in: Building:			Room:			Operator	:				Date:		

#### TABLE X2.3 Model VHT Termination Data Sheet for a Set of Tests

Test number including glass or glass ceramic type	Specimen number	Termination temperature [°C]	Date of termination	Time of termination	Assembly mass [g]	Specimen + support [g]	Hd	Remaining glass or glass ceramic [mm]	St. Dev. (remaining glass or glass ceramic)	Observation
Notes/Comments:	1	1	1		1			1	<u> </u>	
Procedure is performed in:										

Building:

Room:

Operator:

Date:

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