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THE PRODUCT CONSISTENCY TEST (PCT): HOW AND WHY IT WAS DEVELOPED

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ABSTRACT

The Product Consistency Test (PCT), American Society for Testing Materials (ASTM) Standard C1285, is currently used world wide for testing glass and glass-ceramic waste forms for high level waste (HLW), low level waste (LLW), and hazardous wastes. Development of the PCT was initiated in 1986 because HLW glass waste forms required extensive characterization before actual production began and required continued characterization during production (≥ 25 years). Non-radioactive startup was in 1994 and radioactive startup was in 1996. The PCT underwent extensive development from 1986-1994 and became an ASTM consensus standard in 1994. During the extensive laboratory testing and inter- and intra-laboratory round robins using non-radioactive and radioactive glasses, the PCT was shown to be very reproducible, to yield reliable results rapidly, to distinguish between glasses of different durability and homogeneity, and to easily be performed in shielded cell facilities with radioactive samples. In 1997, the scope was broadened to include hazardous and mixed (radioactive and hazardous) waste glasses. In 2002, the scope was broadened to include glass-ceramic waste forms which are currently being recommended for second generation nuclear wastes yet to be generated in the nuclear renaissance. Since the PCT has proven useful for glass-ceramics with up to 75% ceramic component and has been used to evaluate Pu ceramic waste forms, the use of this test for other ceramic/mineral waste forms such as geopolymers, hydroceramics, and fluidized bed steam reformer mineralized product is under investigation.

INTRODUCTION

The first U.S. facility and the world's largest facility to immobilize high-level nuclear waste in durable borosilicate glass began radioactive operation in 1996 at the Savannah River Site (SRS). The product specifications on the glass waste form produced in the SRS vitrification facility, known as the Defense Waste Processing Facility (DWPF), required extensive characterization of non-radioactive waste glass simulants made in pilot scale facilities before actual production began in the engineering scale DWPF. In addition, glass quality needed to be continuously characterized during production to ensure that the glass made in 1996 was as durable as the glass made in 2008 or as durable as glasses to be made in 2020.

To aid in the HLW glass characterization, a glass durability (leach) test was needed that was easily reproducible, could be performed remotely on highly radioactive samples, and could yield results rapidly. A test known as the Product Consistency Test (PCT) was developed between 1986 and 1994. The initial basis for the PCT was a crushed glass durability test known as the Materials Characterization Center test #3 (MCC-3) that was performed with constant agitation. Some of the attributes of the MCC-3 test, other ASTM tests, and a Corning Glass Works (CGW) crushed glass test were all incorporated into the PCT. The features of each test that had the potential to optimize the following criteria were considered:

- sensitivity of the test method to glass composition and homogeneity
- minimum time necessary to demonstrate product quality
- ease of sample preparation for radioactive glass
- precision of the test results
- acceptance by waste form developers and repository projects

The PCT is a crushed glass test performed at 90°C performed under static conditions (during initial testing the constant agitation used in MCC-3 was determined not to be necessary). The PCT became an ASTM consensus standard in 1994¹ after thorough review by ASTM Committee C26 which consisted of waste form developers, repository representatives, and Nuclear Regulatory Commission (NRC) representatives.

In 1997, the scope was broadened to include hazardous as well as mixed (radioactive and hazardous) glasses. In 2002, the scope of the PCT was broadened to encompass glass-ceramic waste forms. Glass-ceramics are currently being recommended for second generation nuclear wastes yet to be generated in the nuclear renaissance, e.g. the Global Nuclear Energy Partnership (GNEP): glass-ceramics for GNEP are being referred to as Glass Composite Materials (GCM).² When dealing with glass ceramics, devitrified glasses, or GCM's of any type, one must deal with the release of certain radionuclides from the ceramic or crystalline phases versus the amorphous glass. The strategy for doing this is to choose a unique element from each phase (glass and crystal) in the final waste form. For example, in glass bonded sodalite a unique element and/or radionuclide is monitored from the glassy phase, the halide phases, and the sodalite phase. For example:

- Si, Al, Na, Li (from sodalite and glass phases)
- B (glass phase)
- Cl (sodalite and halite phases)

The only manner in which a crystalline phase does not have to be separately monitored is if the phase is shown, by extensive study, to be inert and not contain radionuclides, e.g. iron bearing spinels in DWPF glasses. The rationale for determining which unique element or radionuclides should be monitored from each phase, is detailed in ASTM C1285 for glass bonded ceramics and devitrified HLW glasses.

The PCT was designed to relate waste form processing to waste form performance. It is a short-term test method yielding a response that is sensitive to both the effects of processing variables on the waste form and its chemical durability. Which attributes or constituents should be tracked will depend on the waste form and its degradation mechanism. The phase composition of multi-phase waste forms may be important to performance, or only the concentration of a key radionuclide or hazardous species.

In the case of homogeneous borosilicate HLW glasses, acceptable performance is defined as an acceptably low dissolution rate, which is controlled by maintaining the glass composition within an acceptable range. The approach can be represented in terms of the following relationships:

$$\begin{array}{c} \text{process control} \leftrightarrow \text{composition control} \leftrightarrow \text{dissolution rate control} \leftrightarrow \\ \text{performance control} \leftrightarrow \text{acceptable performance} \end{array} \quad (1)$$

SCOPE OF THE PCT

The current scope of the PCT procedure states:

“These product consistency test methods A and B evaluate the chemical durability of homogeneous glasses, phase separated glasses, devitrified glasses, glass ceramics, and/or multiphase glass ceramic waste forms hereafter collectively referred to as “glass waste forms” by measuring the concentrations of the chemical species released to a test solution.”

Based on the HLW waste acceptance process described below, the original scope of the PCT was to determine the chemical durability of homogeneous and devitrified HLW glasses by measuring the concentrations of the chemical species released from a crushed glass to a test solution. The test scope covered both radioactive and simulated waste glasses and their devitrified products formed during canister cooling. Between 1994 and 1996 the Mixed Waste Focus Area (MWFA) was actively pursuing vitrification of mixed wastes,^{3,4,5,6} e.g. those wastes that are both Resource Conservation Recovery Act (RCRA) hazardous and radioactive. In addition, vitrification of hazardous mining wastes^{7,8} and mill tailings⁹ was being investigated. Members of the C26.13 and C26.07 ASTM subcommittees requested that the scope of the PCT be broadened to include mixed waste glasses and hazardous waste glasses. The revised scope was incorporated in 1997.

Since the PCT procedure was already applicable to devitrified glasses, Argonne National Laboratory – West (ANL-W) suggested that the scope be broadened to accept their Ceramic Waste Form (CWF) which is a glass bonded sodalite containing about 25 wt% borosilicate glass and 75% ceramic phases (primarily sodalite and NaCl).^{10,11,12}

SIGNIFICANCE AND USE OF THE PCT

- The Product Consistency Test methods (A and B) provide data useful for evaluating the chemical durability of glass waste forms as measured by elemental release. Accordingly, it may be applicable throughout manufacturing, research, and development.
- Test Method A can specifically be used to obtain data to evaluate whether the chemical durability of glass waste forms have been consistently controlled during production (see Table I).
- Test Method B can specifically be used to measure the chemical durability of glass waste forms under various leaching conditions, for example, varying test durations, test temperatures, ratio of sample surface area (S) to leachant volume (V), and leachant types (see Table I). Data from this test may form part of the larger body of data that are necessary in the logical approach to long-term prediction of waste form behavior (see ASTM C 1174).¹³

THE PCT AND THE HIGH LEVEL WASTE (HLW) ACCEPTANCE PROCESS

Production of canistered borosilicate waste forms by waste form producers at SRS and West Valley began ~12 years before the submission of the first license application for the first federal geologic repository was being written.¹⁴ Thus, to ensure that the DWPF and West Valley waste forms would be acceptable at the federal repository and to allow production to begin, the Department of Energy, defined a Waste Acceptance Process for DWPF and West Valley canistered waste forms.

Table I. Applications and Test Conditions for PCT-A and PCT-B Tests

	PCT Test Method A	PCT Test Method B
Type of Waste Form	Radioactive, Mixed, Simulated, Hazardous	Radioactive, Mixed, Simulated, Hazardous
Usage	During production for rapid analysis; for waste compliance	Scoping tests; Crystallization studies; Comparative waste form evaluation
Test Vessel	Unsensitized Type 304L stainless steel; vessels rated to >0.5 MPa	Unsensitized Type 304L stainless steel or Teflon vessels rated to >0.5 MPa
Test Duration	7 days $\pm 2\%$	7 days $\pm 2\%$ or varying times
Leachant	ASTM Type I water	ASTM Type I water or other solutions
Condition	Static	Static
Minimum Mass	≥ 1 g	≥ 1 g
Particle Size	U.S. Standard ASTM - 100 to + 200 mesh	U.S. Standard ASTM - 100 to + 200 mesh or other sizes which are <40 mesh
Leachant Volume	10 ± 0.5 cm ³ /gram of sample mass	10 ± 0.5 cc/gram of sample mass or other volume/sample masses
Temperature	$90 \pm 2^\circ\text{C}$	$90 \pm 2^\circ\text{C}$ or other temperatures (note any changes in reaction mechanism)

As part of the Waste Acceptance Process, the DOE-repository program developed Waste Acceptance Product Specifications (WAPS) for the borosilicate waste forms. The WAPS identifies the characteristics that the waste forms must have in order to be compatible with the repository. DWPF must comply with the specifications to ensure that the canistered waste forms will be acceptable for disposal.

The WAPS specifications most relevant to public health and safety are those relating to release of radionuclides. WAPS Specification 1.3 relates to the ability of the vitrification process to consistently control the final waste form durability, i.e., the stability of the glass against attack by water:

1.3 Specification for Product Consistency

“The producer shall demonstrate control of waste form production by comparing, either directly or indirectly, production samples to the Environmental Assessment (EA) benchmark glass.”¹⁵

1.3.1 Acceptance Criterion

“The consistency of the waste form shall be demonstrated using the Product Consistency Test (PCT). For acceptance, the mean concentrations of lithium, sodium and boron in the leachate, after normalizing for the concentrations in the glass, shall each be less than those of the benchmark glass described in the Environmental Assessment for selection of the DWPF waste form¹⁶...One acceptable method of demonstrating that the acceptance criterion is met, would be to ensure that the mean PCT results for each waste type are at least two standard deviations below the mean PCT results of the EA glass.”

Lithium, sodium, and boron releases were compared to the rate at which various radionuclides were released. For example, in high level borosilicate waste glass, Tc⁹⁹, present at $\sim 4.1 \times 10^{-4}$ weight % in the waste form, was shown^{17,18,19,20,21,22,23,24} to be released at the same maximum normalized glass release as boron, lithium, and sodium. Since Tc⁹⁹ is released more rapidly than any other radionuclide from HLW glass, monitoring the boron, lithium, and/or sodium release provides a measure of the the maximum radionuclide releases expected for HLW glass. Therefore, for borosilicate glass waste forms, the leachates are routinely analyzed for boron, lithium, and sodium if these elements are present at > 1 mass % in the glass. Additional mechanistic information about high level borosilicate waste glass durability is gained by analyzing for other elements present at > 1 weight % in the glass.

In addition, data was needed on how crystallization of borosilicate waste glasses impacted the overall durability and radionuclide release. These specifications required extensive characterization of the glass product both before and after production. Before production, the DWPF had to extensively characterize the chemical durability of simulated and actual waste glasses to demonstrate that the DWPF could produce an acceptable product. After production began, the DWPF had to confirm^{25,26,27} that the glass produced did, in fact, satisfy the specifications for product consistency and radionuclide release.

It should be noted that the determination of the linkage between Tc⁹⁹ release and the boron, lithium, and/or sodium release required years of intense study.^{17,18,19,20,21,22,23,24} This linkage will not be the same for every waste form tested and new marker elements and/or the determination of the radionuclide release itself may be required for new waste forms. This strategy and required testing was detailed in the ASTM 2002 revision of the PCT.

DEVELOPMENT OF THE PCT

In 1986 the Savannah River Laboratory (SRL) now the Savannah River National Laboratory (SRNL), undertook the development of a leach test specifically designed to establish conformance with the WAPS. The primary objective of the test would be to confirm that the DWPF glass product was consistently acceptable from a durability perspective. Such a leach test would be easily reproducible, capable of being performed remotely on highly radioactive samples of glass and able to yield reliable results rapidly. Several standard leach tests were examined, with a wide variety of test configurations. Tests examined included those used widely in the nuclear industry for a variety of waste forms, such as the Materials Characterization Center (MCC-1 and MCC-3)²⁸ test protocols, as well as ASTM tests for commercial glass (ASTM C-225)²⁹ and container glass classified as municipal waste (ASTM D3987).³⁰ The MCC tests were of lengthy test duration (~ 28 days) while the ASTM and Corning Glass Works (CGW) tests were of short test duration (1 hour, 1 day, or 2 day). Some tests were room temperature, some 80°C, some 90°C, and one was run at 121°C in an autoclave. The tests were screened based on the following criteria:

- Sensitivity of the test to glass quality parameters, such as composition and homogeneity. The response of the test must be dominated by the waste form. Tests which are not glass dominated are not adequate measures of waste form product quality. For example, tests have been designed to measure glass performance under repository conditions or conditions of rapid flow. These tests are dominated by repository parameters such as groundwater chemistry or flow rate or both.

- Minimum test duration necessary to demonstrate product quality with a high degree of precision. Use of a short test during production provides rapid confirmation of waste form product quality. The extensive characterization of the product before the startup of the DWPF required thousands of samples. Long-term tests would have made it difficult to get the information needed in a timely manner and more susceptible to the effects of inadvertent errors, such as power losses.
- Feasibility of remote performance of the test. Once radioactive operations began the test was performed on highly radioactive samples in shielded cell facilities with manipulators. Thus the sample preparation and test procedure were kept mechanically simple.
- Precision of the test. The test must be precise enough so that during production the possibility of obtaining incorrect indications of glass quality is minimized.
- Acceptance of test results by the federal repository.

Based on the preliminary screening of the MCC, ASTM, and CGW tests no one test completely fulfilled these objectives. However, the MCC-3 test protocol came closest to satisfying most of the criteria. Thus, a limited text matrix variant of the MCC-3 test method using the shorter test durations and static conditions characteristic of the ASTM and CGW tests was used as a starting point for development of the PCT (Version 1.0).

Based on the results of the SRNL internal round robin (described below) and technical reviews of the test protocol by experts in other laboratories, the PCT protocol was modified (Version 2.0). The parameters necessary for determining glass quality with a high degree of precision were optimized.

Based on the results of the SRNL hosted external round robin (described below), the PCT protocol was again modified (Version 3.0). Versions 3.0 to 7.0 were submitted to ASTM subcommittee C26.13 (Repository Waste Package Interactions) for consensus review between 1990 and 1994. Version 5.0 was balloted at the C26.13 subcommittee level with no negative ballots. Several affirmatives with comments were addressed in Version 6.0. The revised Version 6.0 of the PCT was balloted at the C26 (Nuclear Fuel Cycle) full committee and subcommittee level with no negative ballots. Several affirmatives with comments were addressed in Version 7.0 which was balloted at the full Society level with no negative ballots. The PCT became an ASTM standard in late 1994.

HLW Glass: SRNL Internal Round Robin³¹

An internal SRNL round robin was held in 1987 using the initial (Version 1.0) test protocol. Three researchers participated in the round robin. Three glasses were used. One was partially crystallized, one was phase separated, and one was homogeneous). The round robin had three primary objectives:

- To determine the effects of various test parameters (duration, agitation, radiation, vessel material, and filtration) on test results.
- To select a set of test responses which were both reliable and sensitive to glass quality parameters (composition and homogeneity).
- To provide initial estimates of the achievable within-laboratory precision of the test.

The PCT was found to be sensitive to both glass composition and homogeneity. Consistent relative glass durability could be achieved in only one day. However, optimum precision was achieved at seven days. The 7 day precision (for triplicate tests) for any one investigator was 2-3%. Variations between investigators were 5-7%. The PCT could be performed remotely, with the same precision. The results showed no significant effect of radiation on glass durability and demonstrated that similar results were obtained whether Teflon[®] or stainless steel vessels were used for non-radioactive glasses. The most sensitive and precise indicators of glass quality were found to be B, Li, Na and Si. If K was present at greater than 2 wt% in the glass, it was also a good indicator of glass quality. Filtration of leachate samples improved the precision and was found to be necessary. Agitation was found to be unnecessary for the 7 day test duration.

HLW Glass: Multi-Laboratory External Round Robin³²

From 1988 to 1989 a seven laboratory round robin was performed using Version 2.0 of the PCT procedure. The purpose of the round robin was to better determine the inter- and intra-laboratory precision and accuracy of the PCT protocol, for use in establishing product compliance. The participants were selected based on their experience in glass testing, and included the Materials Characterization Center (MCC), Argonne National Laboratory (ANL), Catholic University of America (CUA), Corning Engineering Laboratory Services (CELS), Pacific Northwest National Laboratory (PNNL), SRNL, and the University of Florida (UF).

Four glasses were used in the multi-laboratory round robin. These included a National Institute of Science and Technology (NIST) reference glass (SRM-623), an MCC standard reference glass (ARM-1), and two glasses which were based on possible compositions to be produced in the DWPF. The latter two glasses were fabricated and analyzed by CELS.

The MCC prepared test kits for each of the participants. The MCC crushed and sieved samples of glass, cleaned leach vessels, provided filters, sample handling equipment, and the test protocol (Version 2.0). In addition to the test materials, 100 mL of a multi-element standard solution for chemical analysis was also supplied. This allowed estimation of the effect of lab-to-lab variability in chemical analysis, an independent measurement of the variability in actually performing the PCT protocol.

Each of the laboratories tested all four glasses. Each laboratory tested at least one sample of each glass per week, for three consecutive weeks. Each laboratory tested one of the simulated waste glasses in triplicate during the first week. Each laboratory analyzed its own leachate as well as the multi-element solution standard. Leachate samples and the multi-element solution standard were analyzed for Na, Li, K, Al, Si, Fe, B, F, Cl, NO₃, and SO₄. The pH of all solutions was also measured.

The results of this external round robin confirmed the results of the SRNL round robin. The PCT was determined to be a precise indicator of glass quality. After statistical analysis of the results, the MCC concluded that a laboratory experienced in performing the PCT (i.e., one able to control the test precision) would be able to discriminate between glasses which differed by on 10% (based on B, Na, Si, and Li), to the 95% confidence level. This was approximately four times superior to the results from around robin of the MCC-1 test also conducted by the MCC. The single researcher precision varied between 1.8-2.3%. The pooled total within and between laboratory precision varied from 7.5-10%.

The glasses tested in the multi-laboratory round robin had not been “washed” of electrostatically adhering fines (Figure 1). During the analyses of the results of the multi-

laboratory round robin it was determined that the fines that electrostatically cling to the larger (100-200 mesh particles) gave a non-reproducible contribution to the PCT response and so a “washing protocol” to remove the electrostatic fines needed to be added to the PCT protocol. The washing protocol was incorporated into Version 2.1 of the PCT procedure.

HLW Glass: Multi-Laboratory Confirmatory Testing on Radioactive Glasses³³

Version 2.1 of the PCT protocol was used for confirmatory testing with radioactive glasses. Two laboratories (ANL and SRNL) participated in the radioactive round robin. All of the PCT operations which involved the glass were performed remotely with master slave manipulators in shielded cell facilities. This included grinding, sieving, and washing the glasses, assembling the test apparatus (see Figure 2), and leaching in a $90\pm 2^\circ\text{C}$ oven. Stainless steel vessels were used for the radioactive testing.

The test solutions only contained a small amount of radioactivity at the end of the testing. This allowed the leachate to be removed from the shielded cells, sampled in a radioactive hood and analyzed. The results measured by ANL and SRNL for the B, Na, Li, and Si agreed within 10%, providing confirmation of the results of the multi-laboratory non-radioactive round robin.

HLW Glass-Ceramic Waste Forms (CWF): Multi-Laboratory External Round Robin^{10,11,12}

ANL-W sponsored a four laboratory round robin that generated within-laboratory and between laboratory RSD data for glass-bonded ceramics which was incorporated into the PCT in 2002. The within-laboratory RSD for boron and lithium from the borosilicate glass was 8.1% and 5.3%, respectively. Sodium from the borosilicate glass and the sodalite and other minor sodium containing phases had a %RSD of 13.4%. The between-laboratory relative standard deviation for boron and lithium from the borosilicate glass was 19% and 12%, respectively. Sodium from the borosilicate glass and the other sodium containing phases had a %RSD of 18%.

PCT TEST DISCRIMINATION

An independent study³⁴ was performed at PNNL to evaluate the discrimination of the PCT test by varying the test duration (3 vs. 7 days), varying the SA/V ratio (5, 10 and 100 mL/g), washing of fines (with and without), and filtering (with $0.45\mu\text{m}$ and without). All four factors were found to influence the solution pH and the elemental releases from the glasses. The SA/V ratio and leachate filtration were found to have the largest effect on the precision of the elemental releases. Larger SA/V ratios (more glass to less solution) and leachate filtration improved the discrimination of the test. Washing of the fines suggested that this procedure might dampen the discrimination ability of the test but washing of the fines was found to improve the precision of the replicates.

The results of the PCT can be expressed in different units, e.g. NC_i (g/L), NL_i (g/m^2), or NR_i ($\text{g}/\text{m}^2\cdot\text{day}$) where “i” is the element or radionuclide of interest in the waste form. Each unit is representative of either the grams of glass dissolved or leached in the PCT based on the element “i”. The calculation of NC_i (g/L) does not require the surface area to be measured or calculated but assumes that similar waste forms are being compared, e.g. that the waste form has the same density and surface roughness as the waste form that it is being compared to. However, when reporting the PCT results in units of NL_i (g/m^2) and/or NR_i ($\text{g}/\text{m}^2\cdot\text{day}$) a surface area term is used in the calculation. For these units of reporting, the manner in which the surface area is determined (calculated or measured) is extremely important.

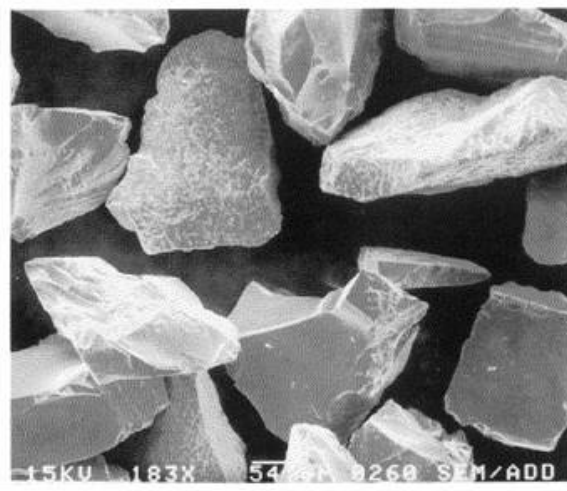
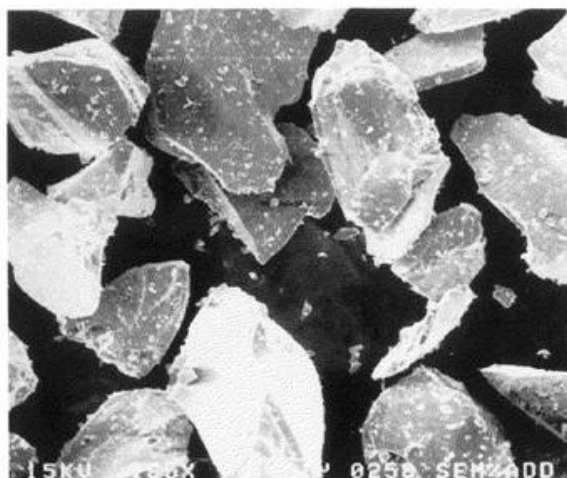


Figure 1. Adherent fines on – 100 to + 200 mesh glass particles before washing (Top). Glass particles after washing in ASTM Type I Water and Ethanol (Bottom). Ethanol only is specified when water soluble phases are present.

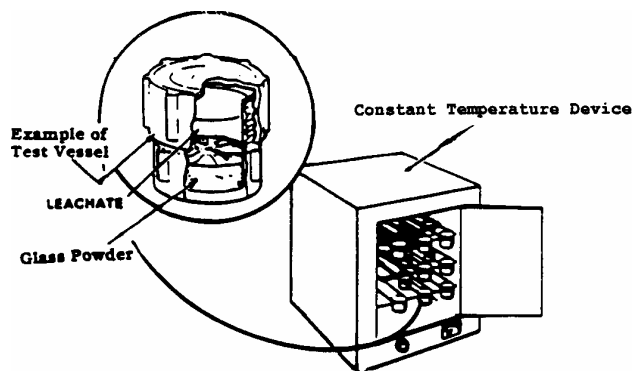


Figure 2. Test apparatus showing both stainless steel and Teflon® vessels.

RELATION OF PCT RESULTS TO OTHER DURABILITY TEST RESPONSES

The surface area (S) to volume (V) of leachant ratio times the test duration in days, a parameter known as $(S/V) \cdot \text{time}$, is used as a test acceleration parameter and has widely been used to relate the response of a variety of laboratory glass durability tests to each other. For example:

- short-term crushed glass tests (PCT and MCC-3) have been related to long-term crushed glass tests (PCT and MCC-3)^{35,36,37,38} by increasing S, decreasing V, or increasing time

- short and long-term monolith tests (PCT and MCC-1) have been related to short and long-term crushed glass tests (PCT and MCC-3)^{37,38,39}
- short-term crushed glass tests (PCT) have been related to long-term burial tests⁴⁰
- long-term crushed glass tests (PCT) have been related to shorter term, higher temperature, Vapor Hydration Test (VHT) responses, e.g. the HLW Environmental Assessment (EA) glass reaches the same stage of durability within 56 days at 20,000 m⁻¹ or >313 days at 2000 m⁻¹ when tested by PCT at 90°C³⁹ or within 6 days when tested by VHT at 200°C³⁹
- the forward rate of the short term crushed glass test (PCT) has been shown to be an upper bound for accelerated durability behavior (the return to the forward rate or Stage III leaching behavior)¹⁴

The relation of the different test responses demonstrates that the test responses are related mechanistically by the acceleration factors being used in the leaching protocols. When performing comparative test results care must be taken⁴¹ during data interpretation since different pH values are achieved during static testing at different S/V ratios than in dynamic testing in buffer solutions. This affects the reaction rates and must be accounted for when comparing the results of the various durability tests.

COMPARISON OF PCT GLASS RESPONSE TO OTHER WASTE FORMS

When it is necessary to compare the response of ceramic/mineral or GCM waste forms to glass, the PCT is often the initial test used because of its simplicity, response, and reproducibility. If the ceramic/mineral or GCM waste form has significant surface roughness this presents variability in the PCT response as well as for other crushed waste form tests such as the Single Pass Flow Through Test (SPFT), and the Pressure Unsaturated Flow-through (PUF) test. McGrail⁴² has recommended that the geometric methodology of the determination of surface area given in the PCT test protocol (ASTM C1285) and SPFT (ASTM C1662) protocol is the correct way to assess the surface area of vitreous waste forms. He has also recommended that the BET surface area is the correct way to assess the surface area of mineral products due to their additional surface roughness in SPFT and PUF tests.⁴³ McGrail has also used the pore size measurement and calculation of surface area for the estimation of the surface area for SPFT testing of the foamy glass created by bulk vitrification,⁴⁴ i.e. there is no uniform surface area measurement methodology.

When the PCT was used for the ANL CWF (glass bonded sodalite) the geometric methodology of determination of surface area was used^{45,46,47} except when the particle size was too small to determine via sieving, then the Gaussian particle size distribution was measured⁴⁸ by particle size analysis. For other applications of the PCT to Pu ceramics,^{49,50,51} geopolymers,⁵² grout,⁵³ hydroceramics,⁵⁴ and fly ash,⁵⁵ the geometric surface area has been used.

Pareizs, et. al.⁵⁶ and Jantzen, et. al.⁵⁷ initiated a comparison of geometric versus measured (Microtrac and BET) surface area for various mineral waste forms with high surface roughness and these comparisons are in progress. The appropriate surface area determination, as a function of surface roughness, is an area that needs further investigation for all the crushed waste form durability tests such as the PCT, the SPFT, and the PUF test.

CONCLUSIONS

Over the last 21 years the PCT has been shown to be a robust and short duration test. The approach can be represented in terms of the following relationships:

process control ↔ composition control ↔ dissolution rate control ↔
performance control ↔ acceptable performance

While the current scope of the PCT does not currently include multiphase ceramic waste forms, progress has been made in establishing some of the relationships above, e.g. the process control ↔ composition control ↔ dissolution rate control, so that such applications can now begin to be considered. This will involve determining the best way to measure the S/V parameter for mineral waste forms versus glass for the PCT, the SPFT, and the PUF test. The 2008 revision of the PCT is currently used world wide for testing of glass and glass-ceramics and has been used successfully on ceramic waste forms although this application is not specifically covered in the current scope, significance and use statements in the standard.

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