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THE PRODUCT CONSISTENCY TEST FOR THE DWPF WASTEFORM

by

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**Abstract**

The preliminary specifications on the glass wasteform to be produced by the Defense Waste Processing Facility (DWPF) require extensive characterization of the glass product both before actual production begins and then during production. To aid in this characterization, a leach test was needed that was easily reproducible, could be performed remotely on highly radioactive samples, and could yield results rapidly. Several standard leach tests were examined with a variety of test configurations. Using existing tests as a starting point, the DWPF Product Consistency Test (PCT) was developed in which crushed glass samples are exposed to 90°C deionized water for seven days. Based on extensive testing, including a seven-laboratory round robin and confirmatory testing with radioactive samples, the PCT is very reproducible, yields reliable results rapidly, and can be performed in shielded cell facilities with radioactive samples.

**Introduction**

Construction of the nation's first, and the world's largest, facility to immobilize high-level nuclear waste is nearing completion at the Savannah River Site in Aiken, South Carolina. In the Defense Waste Processing Facility, waste resulting from over thirty years of reprocessing of nuclear fuels for national defense purposes will be immobilized in durable borosilicate glass.

Waste Acceptance Preliminary Specifications (WAPS) on the wasteform require extensive characterization of the glass product both before and after production begins. To satisfy the WAPS requirements, a leach test was needed which could reliably and easily provide rapid confirmation of the consistency of DWPF glass.

The initial basis for the DWPF Product Consistency Test (PCT) was derived from an intercomparison of crushed glass durability tests from the Materials Characterization Center (MCC), from the American Society for Testing Materials (ASTM), and from Corning Glass Works (CGW). The features of each test which optimized the following criteria were considered during PCT development:

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

In this paper, the vitrification process and product are described, as well as the product specifications, to provide a background for the use of the test. Test development, and the results of confirmatory testing are then presented. Based on extensive testing, including a seven-laboratory round robin, and confirmatory testing with radioactive samples, the PCT has demonstrated that it is reproducible, yields reliable results rapidly, and can be performed in shielded cell facilities with radioactive samples.

### **DWPF Process<sup>1, 2, 3</sup>**

A diagram of the waste immobilization process is shown in Figure 1. SRS waste is currently stored on site in carbon steel tanks, in three forms: sludge, salt cake, and salt solution. The sludge consists primarily of precipitates of the hydroxides of iron, aluminum, and manganese, and contains most of the radioactivity in the waste, except for radioactive cesium. The salt cake and salt solution is mainly composed of sodium salts of common anions, such as nitrate, nitrite, aluminate, and hydroxide. The salt fraction of the waste contains most of the radioactive cesium, and only trace amounts of other radioactive species.

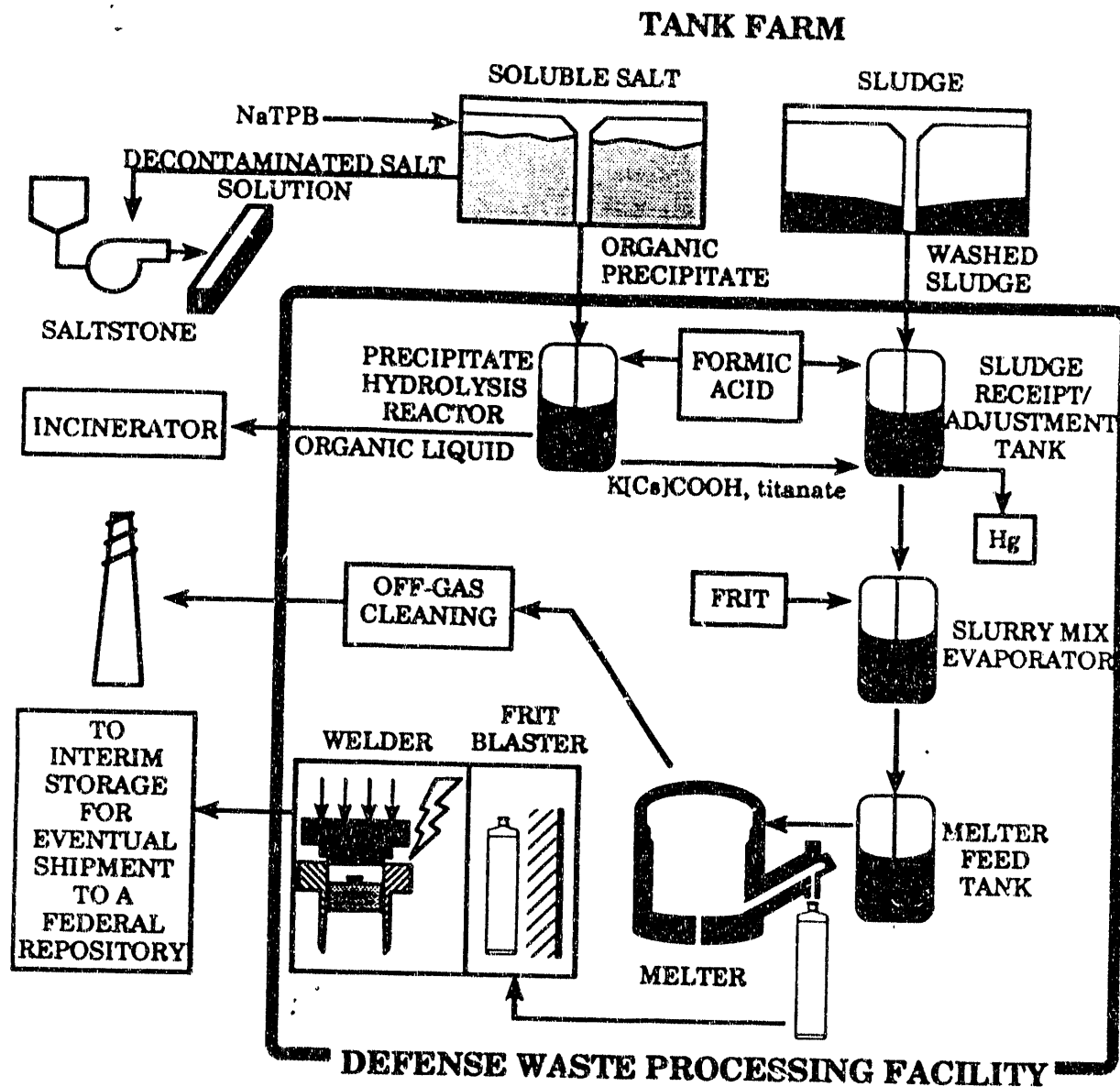


Figure 1. Immobilization of Savannah River Site High-Level Waste.

In the waste storage tanks, sludge waste is washed to remove as much of the soluble nonradioactive ingredients as possible. Cesium is removed from the soluble salt fraction of the waste by precipitation as cesium tetraphenylborate. Residual Sr and Pu in the salt waste are adsorbed on a small amount of sodium titanate. The resulting washed slurries are concentrated, and pumped to the DWPF.

Within the vitrification building, a formic acid hydrolysis process is used to remove most of the organic content of the tetraphenylborate salt before vitrification. The washed sludge slurry and the precipitate hydrolysis product are then mixed together. Glass-forming chemicals, in the form of glass frit, are then added to the resulting slurry which is now ready for vitrification. Frit (a previously melted mixture of chemicals) is used because it ensures that the mixture of glass-forming chemicals is uniform, and of the proper composition.

Vitrification of the frit-waste slurry is accomplished in a Joule-heated ceramic melter at 1150°C. The glass is poured from the melter into stainless steel canisters by a differential pressure technique. After completion of the fill, a temporary seal is inserted into the canister, to prevent the introduction of water during decontamination.

Contamination is removed from the canister surface by air-injected slurry blasting. After decontamination, the canister is sealed, using an upset resistance welding technique. The cleaned and sealed canisters are then sent to an interim storage facility, where they will remain until shipment to a repository.

### **Waste Acceptance Process**

Production of canistered waste forms by the DWPF is scheduled to begin well before submission of the license application for the first repository. Further, it is likely that DWPF glass waste forms will not be shipped to a repository until after the year 2008. At this point, the DWPF will have produced approximately 7000 canisters, and processed all of the high-level waste currently at SRS. Thus, to ensure that the DWPF product will be acceptable at a federal repository and to allow production to begin, the Department of Energy, through its Office of Civilian Radioactive Waste Management, has defined a Waste Acceptance Process for DWPF canistered waste forms.

As part of the Waste Acceptance Process, the repository program developed Waste Acceptance Preliminary Specifications (WAPS) for the DWPF product. The WAPS identify the characteristics that the DWPF wasteforms must have in order to be compatible with the first repository. DWPF compliance with the specifications will ensure that canistered wasteforms produced in the DWPF will be acceptable for disposal.

### **Radionuclide Release Specifications**

The specifications most relevant to public health and safety are those relating to release of radionuclides. The three relevant specifications are as follows:<sup>4</sup>

- The DWPF must control its process so that the product is capable of limiting releases to no more than  $1 \text{ g/m}^2 \cdot \text{d}$  on the 28 day MCC-1 monolithic leach test<sup>5</sup>
- During production, the DWPF must verify that the product conforms to the specification, with a high degree of confidence
- The DWPF must provide radionuclide release data for glasses covering the expected range of compositions and a wider range of time and temperature treatments so that the response of crystallized glasses are included

These specifications require extensive characterization of the glass product both before and after production begins. Before production, the DWPF must extensively characterize the chemical durability of simulated and actual waste glasses to demonstrate that the DWPF can produce an acceptable product. After production begins, the DWPF must confirm that the glass produced does, in fact, satisfy the specifications for radionuclide release.

The monolithic MCC-1 leach test referred to in the specifications above is not well-suited to providing radionuclide release data on production glass nor on crystallized glasses.<sup>6</sup> The test method was originally developed to compare different waste forms, not the consistency of a

single waste form during production.<sup>7</sup> The error associated with the MCC-1 test method is large (~50% of the specified limit),<sup>8</sup> which translates into either extensive sampling of radioactive glass to compensate for the large uncertainty, or severe constraints on the glasses which can be processed. The MCC-1 test also requires fabrication of monolithic samples. Because DWPF glass is not annealed, cutting of samples taken during production in order to produce geometric monoliths for testing cannot be done reliably. The 28 days required for the test makes it difficult to provide the large amount of testing needed to conform with the specifications. Use of a 28 day test during production takes over 28 days to provide confirmation of glass product quality.

#### **Development of DWPF Product Consistency Test (PCT)<sup>9</sup>**

Because the MCC-1 test did not appear suitable for determining the consistency of the DWPF product, Savannah River Laboratory (SRL) 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. 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 (Table 1). Tests examined included those used widely in the nuclear industry, such as the MCC-1 and MCC-3 protocols,<sup>5</sup> as well as ASTM tests for commercial glass (ASTM C-225)<sup>10</sup> and container glass classified as municipal waste (ASTM D3987-81).<sup>11</sup> 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 glass. Tests which are not glass dominated are not adequate measures of glass product quality. For example, tests have been designed to measure glass performance under repository conditions. The responses of these tests are dominated by repository parameters such as groundwater chemistry or rock-groundwater interactions.<sup>8,12</sup>



Table 1. Comparison of Test Methods.

<u>TEST PARAMETER</u>	<u>MMC-3</u>	<u>PCT</u>	<u>ASTM D-3987-81</u>	<u>ASTM C225-85</u>	<u>DIN-12111*</u>
Particle size	both <325 and 100-200 mesh	100-200 mesh (0.111mm)	Depends on waste form (<10mm)	40-50 (0.356mm)	40-50 (0.356mm)
wt <sup>s</sup> solid(g)	4	4	700	10	2
Use standard glass	NO	YES	NO	NO	NO
V <sub>soln</sub>	40	40	2800	50	50
V <sub>soln</sub> /wt <sup>s</sup> solid (mL/g)	10	10	4	5	25
SA/V (cm <sup>-1</sup> ) 100-200 mesh	19.55 for	19.55	7.8 x 10 <sup>-4</sup>	12.13	2.43
Time (days)	28, 56, 91, 182, 273, 364, and longer	7	2	0.04	0.042
(SA/V) • (t) (cm <sup>-1</sup> • days)	>547.4 for 100-200 mesh	136.85	1.6 x 10 <sup>-3</sup>	0.50	0.10
T (°C) 150 + 190	40, 90, 110,	90 depends on	18-27 but waste form	121	98
Leachant	ASTM Type I Water	ASTM Type I Water	ASTM Type IV Water	ASTM Type I Water	ASTM Type I Water
Filtration	<0.45µm	<0.45µm	<0.45µm	None	None
Leachate	pH and elemental	pH and elemental	pH and elemental	Titration (Si conc?)	Titration
Agitation per minute	10-14 cycles start. roll or shake	Shake at (minimum)	60-70cycles can vary	None	None
Sample Crushing	Grind under alcohol	Analytic Grinder (WC blade)	Crushing not specified	Steel Mortar & Steel Jar - use Mill - use magnet	Steel Mortar - use magnet
Sample Washing	Wash with water & alcohol to remove fines	Wash with water & alcohol to remove fines	None	Wash with acetone to remove fines	Wash with water to remove fines
Vessel Type Stainless	Teflon or Stainless Steel	Teflon or Waste Form Steel	Depends on Pyrex Flask	Predigested	Predigested Pyrex Flask

\* German glass durability standard DIN12-111 similar to ASTM C-225 added for comparison

- 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 glass product quality. The extensive characterization of the product before radioactive operations begin will require testing of thousands of samples. Long-term tests make it difficult to get the information needed in a timely manner, and are more susceptible to the effects of inadvertent errors, such as power losses.
- Feasibility of remote performance of the test. Once radioactive operations begin, the test must be performed on highly radioactive samples. These will necessitate performance of the test in shielded cells, with manipulators. Thus, the sample preparation and the test procedure must be mechanically simple to perform.
- 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. Ultimately, the DWPF product will go to a Federal repository. The repository must accept the test results as accurate indicators of the consistency of product quality. The test results must also be related to repository performance.

Based on the preliminary screening, none of the tests completely fulfilled these objectives. However, the MCC-3 test protocol came closest to satisfying most of the criteria. Thus, a limited test 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.

#### **SRL Internal Round Robin**

An internal SRL round robin was held using the initial protocol.<sup>9</sup>

The round robin had three primary objectives:

- To determine the effects of various test parameters (duration, agitation, radiation, vessel material, and filtration) on the 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.

A set of glasses was then selected for testing so that there would be a wide range of test responses. The set included a glass which would be unacceptable for disposal (Frit 131 - no waste); a simulated waste glass made according to the DWPF process, in large-scale equipment; and a standard glass (the MCC's ARM-1 glass). Also included was a radioactive glass which contained actual SRS waste, which had been prepared in SRL's Shielded Cells Facility according to the DWPF process. This glass was similar to the non-radioactive waste glass which allowed for comparison of the radioactive and non-radioactive results.

The PCT was found to be sensitive to both glass composition and homogeneity. Consistent relative glass durabilities 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 when Teflon<sup>®</sup> and 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 precision and was found to be necessary. Agitation was found to be unnecessary for the seven day test duration.<sup>9</sup>

Based on the results of the internal round robin and technical reviews of the test protocol by experts in other laboratories, the PCT protocol was modified. The parameters necessary for determining glass quality with a high degree of precision were optimized (Table 1).

### **Multi-Laboratory External Round Robin**

An external round robin of the modified PCT procedure was then initiated, involving seven laboratories.<sup>13</sup> 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) which is operated by Battelle-Pacific Northwest Laboratory. The MCC has extensive experience in multi-laboratory testing.<sup>8</sup> The MCC participated in the round robin and conducted the multi-laboratory round robin for SRL.

Argonne National Laboratory (ANL). ANL has provided glass testing support to both the tuff repository program, and to the DWPF. ANL participated in the round robin and also tested duplicate radioactive samples with SRL.

Catholic University of America (CUA). CUA represented the West Valley Demonstration Project in the round robin. CUA is the developer of the pulsed flow leach test which has proven useful in investigating glass leaching mechanisms.

Corning Engineering Laboratory Services (CELS). CELS has extensive experience in performing a wide variety of glass tests, primarily on commercial glasses. CELS also produced the two simulated DWPF waste glasses used in the round robin

Battelle-Pacific Northwest Laboratory. This group represented the Hanford Waste Vitrification Project (HWVP) in the round robin, and used separate analytical and support facilities from the MCC.

Savannah River Laboratory (SRL), for the DWPF.

University of Florida (UF). For over a decade, UF has been a leader in testing the performance of waste glasses. UF was instrumental in gaining US involvement in the burial tests in the Stripa mine, and in involving foreign groups in the Materials Interface Interactions Tests currently being performed at the Waste Isolation Pilot Plant near Carlsbad, NM.

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

The MCC prepared test kits for each of the participants, which included crushed and sieved samples of glass, cleaned leach vessels, filters, sample handling equipment, and the test protocol, which had been prepared by SRL. 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 effects of variability in chemical analysis on the test results.

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. Blanks were included with each set of samples. Each laboratory analyzed its own leachate as well as the multi-element chemical standard. Leachate samples and the chemical analysis standard were analyzed for Na, Li, K, Al, Si, Fe, B, F, Cl,  $\text{NO}_3^-$ , and  $\text{SO}_4^{=}$ . The pH of all solutions were also measured.

The results of this external round robin confirmed the results of the internal round robin.<sup>13</sup> 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 only 10% (based on B, Na, Si, and Li), to the 95% confidence level. This was approximately

four times superior to the results from a round robin of the MCC-1 test also conducted by the MCC. This may be due to either inherently better precision of the PCT, or to a shorter learning curve for performance of the PCT. In either event, the precision which can be realized with production samples (considering multiple test performers, and personnel turnover) should be significantly greater with the PCT.

### **Confirmatory Testing on Radioactive Samples<sup>14</sup>**

The PCT will have to be performed remotely on radioactive samples when DWPF production begins. Hence, additional testing was performed at ANL and SRL to confirm that the results of the PCT were applicable to radioactive samples as well.<sup>14</sup>

Samples of glass containing actual SRS radioactive waste were prepared in SRL's Shielded Cells Facility, in a joule-heated melter. Molten glass was poured into stainless steel canisters, and allowed to cool. Samples of glass were then removed from the canister for testing according to the PCT. Two different radioactive waste formulations (designated 165/42 and 200R) were used, which covered a range of glass compositions representative of those which will be produced in the DWPF. The non-radioactive ARM-1 standard used in the multi-laboratory round robin was run simultaneously along with the radioactive glasses.

Because of the intense radiation from the glass, all of the PCT operations which involved the glass were performed remotely with master slave manipulators. This included grinding, sieving, and washing the glasses, assembling the test apparatus, and leaching in the 90°C oven. Because of the small amount of radioactivity released to the leachate during the test, the leachate could be removed from the shielded cells, and sampled in a radioactive hood.

The results measured by ANL and SRL for the non-radioactive elements in the glasses agreed within 10%, providing confirmation that the results of the round robin using non-radioactive samples applied to radioactive samples. The normalized concentrations of Na, B, and Li are similar indicating that they are being released nearly congruently from the glass. Thus, their release represents a "worst case" maximum release of any species from the glass.

## Sensitivity of the PCT to Glass Composition

The PCT has been shown to give reproducible results for replicate analyses of each glass tested and to give good discrimination between glasses of varying quality, e.g. varying composition and homogeneity.<sup>9,15</sup> In order to determine the sensitivity of the test response to composition, glasses of varying composition have been tested. The PCT response, based on B release from the glasses, is plotted as a function of the thermodynamic free energy of hydration (calculated from the glass composition) in Figure 2. The relation of the thermodynamic free energy of hydration to the response of the MCC-1 durability test have previously been demonstrated.<sup>16</sup> The radioactive and non-radioactive glass PCT results which are included in Figure 2 clearly indicate that the PCT is sensitive to glass composition. The results from testing of radioactive glasses are synchronous with the results from testing of non-radioactive glasses of similar compositions. The PCT response is, therefore, directly correlated to glass quality and not adversely affected by radioactivity.

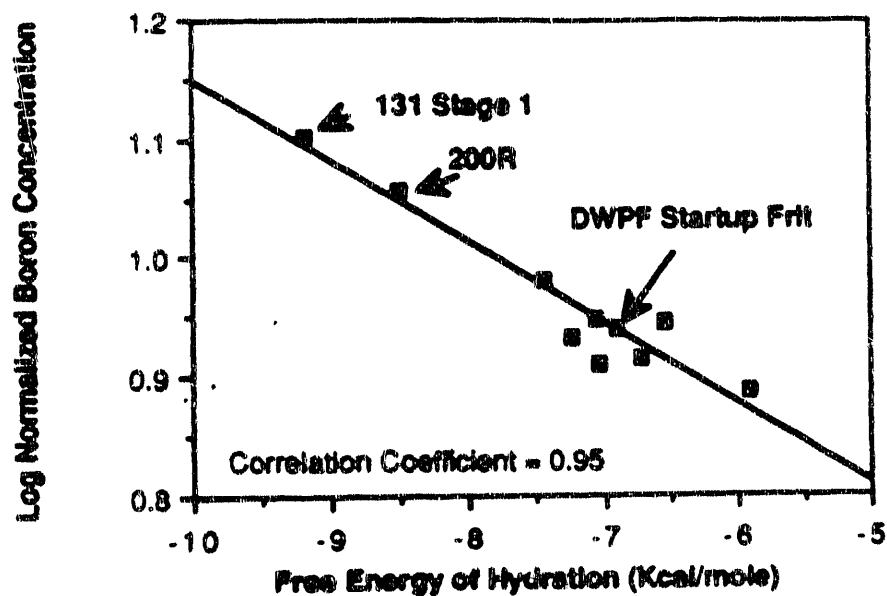


Figure 2. Response of the PCT to Glass Composition.

## Conclusions and Future Directions

A rapid and reproducible glass durability test, the PCT, has been developed to confirm the consistent acceptability of the DWPF glass product. It has been shown that the PCT can be performed remotely with radioactive glass samples and is sensitive to glass quality parameters such as composition and homogeneity, but is not adversely affected by the presence of radiation. These conclusions are based on extensive testing, including inter- and intra-laboratory round robins, and confirmatory testing with radioactive samples.

During DWPF production, small samples of glass will periodically be taken during filling of DWPF canisters. These samples will then be brought from the production facility to SRL's Shielded Cells Facility, where they will be unloaded and prepared for PCT testing and chemical analysis. SRL is currently working with both the repository program and the ASTM to gain acceptance of the PCT. Because of the much better precision of the test results, the PCT may be substituted for the MCC-1 test in the repository program's specifications. Elevating the PCT to ASTM standard status will expose the test to greater scrutiny and usage.

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