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THE DWPF: RESULTS OF FULL SCALE QUALIFICATION RUNS LEADING TO RADIOACTIVE OPERATIONS

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ABSTRACT

The Defense Waste Processing Facility (DWPF) at the Savannah River Site in Aiken, SC will immobilize high-level radioactive liquid waste, currently stored in underground carbon steel tanks, in borosilicate glass. The radioactive waste is pretreated and then combined with a borosilicate glass frit. This homogeneous slurry is fed to a Joule-heated melter which operates at 1150°C. The glass is poured into stainless steel canisters for eventual disposal in a geologic repository. The DWPF canistered waste forms must comply with certain waste acceptance specifications in order to be acceptable for eventual disposal.

The DWPF has completed Waste Qualification Runs which demonstrate the facility's ability to comply with the waste acceptance specifications. During the Waste Qualification Runs seventy-one canisters of simulated waste glass were produced in preparation for Radioactive Operations. These canisters of simulated waste glass were produced during five production campaigns which also exercised the facility prior to beginning Radioactive Operations. The results of the Waste Qualification Runs are presented.

INTRODUCTION

Approximately 130 million liters of high-level radioactive waste, currently stored in underground carbon steel tanks at the Savannah River Site (SRS), will be immobilized in stable borosilicate glass in the Defense Waste Processing Facility (DWPF). The glass is poured into stainless steel canisters for eventual disposal in a geologic repository. In order to be acceptable for disposal the DWPF product (i.e. the canistered waste form) must comply with the Department of Energy Office of Environmental Management's Waste Acceptance Product Specifications (WAPS).[1]

The DWPF has recently completed the production of seventy-one canisters of simulated waste glass as part of its Startup Test Program in preparation for Radioactive Operations. The Waste Qualification Runs portion of the DWPF Startup Test Program was designed to demonstrate that the DWPF could comply with the WAPS prior to the start of Radioactive Operations. Fifty-five of the seventy-one canisters of simulated waste glass were produced during these runs. Varying feed compositions were used to demonstrate that the DWPF could control the glass product over a range of materials using the DWPF Glass Product Control Program.[2] The simulated waste was transferred into the DWPF and processed using the same methods as for the radioactive waste. The glass and canistered waste forms produced during Waste Qualification Runs were extensively characterized. The results of this characterization demonstrate the DWPF's ability to comply with the WAPS.

PROCESS/PRODUCT OVERVIEW

The radioactive waste in the SRS Tank Farms has been separated into a water soluble salt solution and saltcake, and an insoluble sludge of metal hydroxides and oxides. The salt solution and saltcake are decontaminated for disposal as low-level waste by removing the radionuclides by precipitation and sorption. Sodium tetraphenyl borate is added to precipitate soluble salts of primarily potassium and cesium. Sodium titanate is added to adsorb residual strontium and plutonium. The resulting slurry is filtered and the decontaminated filtrate is blended with cement, slag and flyash for disposal at SRS as a low-level cementatious waste. The slurry of the concentrated solids is transferred to DWPF for immobilization. The sludge portion of the waste is washed to remove soluble salts and insoluble aluminum is removed through high temperature caustic dissolution, if necessary. Thus, the radioactive waste from the SRS Tank Farms is transferred to the DWPF in two forms: precipitate slurry and sludge slurry. The waste is then processed and blended in the DWPF before it is vitrified, poured into canisters, sealed, and placed in interim storage.[3] See Figure 1 for a schematic of the DWPF process.

The sludge and precipitate are blended and processed in the Sludge Receipt Adjustment Tank (SRAT). The sludge is transferred directly into the SRAT while the precipitate must first be processed in the DWPF Salt Process Cell to remove most of the organic material. The tetraphenyl borate compounds in the precipitate react in the presence of formic acid and copper (II) catalyst. The products of this reaction are aromatic organic compounds (benzene, phenol, and minor amounts of higher boiling aromatics) and an aqueous phase known as Precipitate Hydrolysis Aqueous (PHA). The PHA contains the cesium, soluble formate salts, boric acid and excess formic acid. The PHA is added to the SRAT after the alkaline sludge is neutralized with nitric acid. In the SRAT mercury removal also occurs.

The amount of sludge and PHA to be blended in the SRAT and the amount of SRAT material and borosilicate glass frit to be blended in the Slurry Mix Evaporator (SME) is determined by the Product Composition Control System (PCCS).[4] The PCCS is a computer program that uses glass property models and statistical algorithms to develop blending strategies and to determine the acceptability of the melter feed before it is transferred to the melter. The PCCS uses lab analyses of tank contents, tank volumes and existing tank heel volumes to determine appropriate tank blending. Any point in the acceptable blending region can be chosen by DWPF engineers as the blending target. PCCS will project a SRAT and Slurry Mix Evaporator (SME) target based on the chosen blend. After the PHA and sludge are blended in the SRAT, this SRAT product is transferred to the SME where borosilicate glass frit is added and the slurry is concentrated to produce melter feed. Again DWPF engineers use PCCS as a tool to determine how much frit to add based on the amount of SRAT material transferred to the SME. PCCS can also be used to predict SRAT and SME compositions based on existing tank compositions, tank transfers, and the uncertainties associated with sampling and processing. These predicted compositions for the SRAT and SME can be used to diagnose processing problems. This capability was used during Waste Qualification Runs as is described in a later section.

The SME is the hold point in the process where feed acceptability is determined as part of the Glass Product Control Program (see below). The SME is sampled and the samples analyzed. The analyses are used by the PCCS (or a similar numerical tool) to determine the acceptability of the melter feed. The acceptability of the melter feed is determined using glass property models to ensure the glass product will be acceptable and that processing constraints (such as viscosity and liquidus) are met.

Once the melter feed material in the SME is determined to be acceptable, it is transferred to the Melter Feed Tank (MFT) and then fed to the joule heated melter. The DWPF melter has two pairs of electrodes. The feed slurry is introduced from the top of the melter and forms a crust, or cold cap, on the surface of the melt pool as the water is evaporated and removed via the off-gas system. The cold cap melts from the bottom and forms the borosilicate glass matrix. The nominal glass melt pool temperature is 1150°C. The mixing behavior of the melter was evaluated during Waste Qualification Runs as described later. The glass is removed from the melter near the bottom through a riser and pour spout. A vacuum is drawn on the pour spout to pour the glass. A glass pour stream sample is taken occasionally from a canister to confirm that the glass durability (as determined by the Product Consistency Test[5]) is acceptable. After a canister is filled, it has a temporary seal installed to exclude free liquid from the canister during the decontamination process. Decontamination of the canister surface consists of blasting an air-injected frit slurry against the canister. The canister is then welded closed and transferred to an interim storage building. The DWPF canistered waste form contains approximately 1800 kg of glass. It is 300 cm in length and 61 cm in diameter.

WASTE ACCEPTANCE SPECIFICATIONS

To ensure that the DWPF product is acceptable for final disposal the Department of Energy Office of Environmental Management has developed the Waste Acceptance Product Specifications (WAPS)[1] which the DWPF product (i.e. canistered waste form) must meet. These specifications are divided into five sections: waste form (borosilicate glass), canister, canistered waste form, quality assurance, and documentation. The most important of the glass specifications is the product consistency specification which states that the DWPF must control its process so that the glass produced is better than the DWPF Environmental Assessment glass[6] as measured by the Product Consistency Test (PCT).[5] The PCT is a crushed glass durability test. DWPF has developed a Glass Product Control Program (see below) to ensure that this specification is met. During Waste Qualification Runs DWPF demonstrated that it can comply with this specification as well as the other glass, canister, and canistered waste form requirements.

GLASS PRODUCT CONTROL PROGRAM

The DWPF has developed the Glass Product Control Program[2] to ensure that the DWPF consistently produces a glass which satisfies the product consistency specification, and that there is demonstrable evidence that this has been achieved. In developing this strategy for producing an acceptable glass product the DWPF has considered the following:

- The only parameters which the DWPF can directly control which affect the results of the PCT are the chemical composition of the glass, and the uniformity of the feed to the melter. It has been shown in laboratory and large scale testing that chemical composition is the prime determinant of a glass's PCT results.[7]
- It is not possible to recycle or rework glass which does not satisfy the specification in the current DWPF processing equipment.
- DWPF waste glass is highly radioactive. Any testing to be performed for control or verification must be performed in shielded cells and, thus, must be simple and reliable.
- The waste stream coming into the DWPF will vary. Thus, the program must be able to handle varying waste compositions.

Based on the above the DWPF Glass Product Control Program ensures that the glass product satisfies the WAPS by controlling the composition of the melter feed. As described earlier, control of the melter feed is ensured by the hold point at the SME. No material is allowed to be transferred from the SME to the MFT until it has been determined to be acceptable. The PCCS, or other numerical tools, is used by DWPF engineers to ensure that the SME material is acceptable. A glass property model which uses a thermodynamic hydration approach to represent composition is used to predict PCT results based on the SME composition. The free energy of hydration reaction for each glass component is multiplied by that component's mole fraction in the glass. These partial quantities are summed to represent the free energy of hydration of the glass. The free energy of hydration is related to the glass PCT results. The ability of the Glass Product Control Program to produce an acceptable glass product was demonstrated during the Waste Qualification Runs portion of the Startup Test Program.

STARTUP TEST PROGRAM

The DWPF Startup Test Program was modeled on the testing required for startup of a commercial nuclear reactor. The Startup Test Program:

- Demonstrated the DWPF's ability to reliably produce an acceptable product that meets the requirements of the WAPS. This was accomplished during the Waste Qualification Runs portion of the Startup Test Program.
- Demonstrated the operability, reliability, and integrity of the major process systems.
- Provided operating experience to operations, maintenance, and engineering personnel.
- Baselined equipment and system operating parameters.

Melter Startup

Prior to Waste Qualification Runs the melter was heated up and one melter campaign (FA-13) was performed. Two thousand pounds of startup frit were loaded into the melter. The startup frit was formulated to avoid corrosion and other problems during startup. The melter dome heaters were turned on to melt the top surface of the startup frit. Once the glass became molten enough to allow joule heating the lower electrodes were energized. Once joule heating was established the first batch of melter feed material was fed into the melter until the normal melter level was reached. Once the melter glass level reached the upper electrodes they were energized. One canister was filled with the melter feed remaining from filling the melter. Two more melter feed batches were produced and eleven more canisters were filled. One more melter feed batch was produced and held in the Melter Feed Tank. The DWPF then entered an extended outage during which modifications were made to the process vessel vent system. Following the outage four more canisters were filled using the remaining feed from the first campaign. The melter feed for this first campaign was a composite type feed. The glass and canisters from this campaign were characterized to ensure that the facility was ready for Waste Qualification Runs and that the procedures and personnel were in place for glass/canister characterization.

During this first melter campaign problems with pour spout pluggage occurred. The blockage was cleared but this problem which continued to occur during Waste Qualification Runs initiated an effort to design and procure a manipulator arm which could remotely clear the glass blockages.

Waste Qualification Runs

During the Waste Qualification Runs, fifty-five canisters were filled, over four melter campaigns, with simulated waste glass which was produced in accordance with the Glass Product Control Program. During these four melter campaigns, the feed coming into the DWPF went through abrupt changes in composition. The purpose of making such abrupt changes was to demonstrate that the Glass Product Control Program is able to control the glass product even when the feed composition is rapidly changing. This should enhance the confidence in the use of the program during normal operations, when changes in feed composition will be more modest.

The specific compositions processed during the Waste Qualification Runs were selected based on the thesis that if the composition / PCT correlation has been used properly to judge the acceptability of the feed, the only possible cause of failure of the GPCP is segregation of the feed. This will occur only if the rheological properties of the material are not consistent with good mixing in the process vessels. Thus, the primary purpose of varying the feed composition during Waste Qualification Runs was to test a range of rheological properties, particularly for the melter, and not to qualify a set of compositions. The compositions made step changes from a Blend (design basis) composition with a dopant to track melter mixing behavior, to a low viscosity feed, to a high viscosity feed, and back to a Blend composition. The range of the major components in the sludge and then the range of the major components in the melter feed (SME composition) over Waste Qualification Runs is shown in Table I. Although, DWPF is not qualifying a set of compositions, these compositions represent the extremes of the waste currently present in the SRS Tank Farms.

The first campaign of Waste Qualification Runs, WP-14, was a composite feed doped with neodymium to serve as a tracer to monitor melter mixing. One batch of melter feed was produced which yielded seven canisters of simulated waste glass. Glass pour stream samples were taken from the first six of the seven canistered waste forms produced. These seven canistered waste forms were processed as shown in Table II. Neodymium was added to the SME prior to sampling of the SME for acceptability determinations.

An example of the importance of the SME hold point occurred during the production of this first batch of material. During the evaluation of this batch for acceptability, the liquidus criterion (glass property related to processing) was not met. However, the glass durability (i.e. predicted PCT results) were acceptable. It was determined that not enough frit had been added to the batch. The PCCS was utilized to confirm that the addition of 3600 pounds of frit to the SME would bring the batch into the acceptable region for the liquidus criterion and keep the durability acceptable. Thus, following the Glass Product Control Program, 3600 lbs of frit were added to the SME batch, the batch was agitated, and then sampled again. The results of the samples analyses were evaluated and the batch was determined to be acceptable. The SME batch was then transferred to the MFT.

During the second campaign of the Waste Qualification Runs, WP-15, four batches of high iron feed were prepared, which yielded twenty canistered waste forms. Glass pour stream samples were taken from all but the first and the eighth canistered waste forms produced. These twenty canistered waste forms were processed as shown in Table II. Prior to the performance of this campaign, problems had been experienced in pilot plant tests with melter feeds which were high in iron, and contained alkali metal ions in relatively high concentrations. The glass produced from these feeds was much less durable (as measured by the PCT) than predicted by the PCCS. Many of the glasses produced in the pilot plant testing were also phase separated, indicating that this might be the cause of the discrepancy. As a result, a variability study was undertaken with this feed. This study showed that the PCT results for the PCCS target glass were reasonably consistent with the PCCS predictions, but that there were large discrepancies between the predicted and actual PCT results for most of the other glasses tested. As called for by the GPCP, a model was developed for use on this material, called the Purex model. The resulting model essentially fits a straight line between two clumps of data at either end of the free energy of hydration range, and overpredicts PCT results of glasses in the middle of the range. This provides additional conservatism in PCT predictions for glasses with intermediate free energy of hydration values. Thus, its use would prevent production of unacceptable glass.

The decision was made to proceed with the campaign, but to control the composition of the feed through the model discussed above; through the controls already in place in the Glass Product Control Program (including the use of the PCCS); and by taking samples of each SME batch, vitrifying them, and subjecting the vitrified samples to the PCT. If any of these indicated a problem with a SME batch, the SME batch would be remediated as called for in the GPCP, again using the same three control measures.

Also, during the production of one of the WP-15 batches of melter feed the predicted composition for the SRAT, which was discussed above, was used by the DWPF engineers to diagnose an operating concern. The measured composition of the SRAT was not consistent with the predicted composition from the PCCS. The analyzed sample results were lower in aluminum, iron, and silicon. After further investigation it was found that foaming occurred in the SRAT during processing and sludge was carried over to the Slurry Mix Evaporator Condensate Tank. As a result operating changes were made with the following batches to prevent reoccurrence.

During the third campaign of the Waste Qualification Runs, WP-16, four batches of high aluminum feed were prepared, which yielded nineteen canistered waste forms. Glass pour stream samples were taken from all but the sixteenth and seventeenth canistered waste forms produced.

These nineteen canistered waste forms were processed as shown in Table II. Mercury was added to the precipitate in the salt process cell during the last batch of WP-16 to demonstrate the mercury removal system. No significant processing problems were encountered during this campaign and the mercury was successfully removed from the melter feed.

During the fourth campaign of the Waste Qualification Runs, WP-17, two batches of feed were prepared. This feed transitioned back to a composite feed from a high aluminum feed. These two batches of feed yielded nine canistered waste forms. Glass pour stream samples were taken from all but the first canistered waste form produced. These nine canistered waste forms were processed as shown in Table II. No problems had been observed with this type of feed, which was similar to the feed used in WP-14, so no special control measures were used. Mercury was added to the precipitate in the salt process cell and noble metals were added to the sludge in the SRAT to further demonstrate the mercury removal system and simulate operations with noble metals in the sludge. The noble metals added were: ruthenium, rhodium, palladium and silver. No significant processing problems were encountered during this campaign - the mercury was successfully removed from the melter feed and no unusual problems were encountered with the addition of noble metals.

Glass and Canister Characterization

After processing was completed the filled canisters were transported to a test facility for destructive examination. The canistered waste forms were either sectioned or a portion of the canister wall removed and glass samples taken (except for two of the canisters produced during WP-17 which were archived intact). Sectioning consisted of using a band saw to slice through the entire canister at three levels. One of the slices was made at the height corresponding to the level of glass in the canister when the pour stream sample was taken. Glass samples were taken at four radial locations for each of the three levels. All the glass samples, including the DWPF pour stream samples, were characterized for chemical composition, devitrification, and durability (measured by the PCT). The chemical composition results from the glass samples were compared to the results of the melter feed sample analyses. The PCT results of the glass samples were compared to the PCT results predicted by the PCT/chemical composition. Also, the glass pour stream sample results were compared to those of the glass samples taken directly from the canisters. These comparisons demonstrated that an acceptable product had been produced under the controls of the Glass Product Control Program.

The PCT results for all of the glass produced during the Waste Qualification Runs were far below the values for the benchmark EA glass (see Table III). The mean PCT values for each of the 55 canisters produced clearly lie far below the values for the EA glass. For each of the campaigns, there is greater than 99% confidence that at least 99.9% of the least durable glass produced has better PCT results than the EA glass. This demonstrates that use of the Glass Product Control Program by the DWPF will ensure production of a product which satisfies the product consistency specification.

For all of the campaigns, there were statistically significant variations in the PCT results as a function of sample location in the canister, and sometimes from canister-to-canister. This is not too surprising, because the feed was varying for each of the last three campaigns. However, none of these affected the ability of the product to satisfy the product consistency specification. Figure 2 compares the mean PCT results for each canister to the predictions for each batch of melter feed. The figure shows that the actual PCT results were adequately predicted and shows the lag in the glass as compared to the melter feed. This lag is due to heels in the process tanks and the melter volume.

Characterization of the canisters produced during the Waste Qualification Runs was also performed. The following activities were performed:

• six cansitered waste forms were tested for foreign materials by sampling the free air space above the glass level to demonstrate that DWPF can exclude foreign materials from the canister

• nine canister welds were leak tested, burst tested, and microstructurally examined to demonstrated DWPF's ability to make an acceptable weld

• five canistered waste form were dimensionally measured before and after filling to demonstrate that canister filling does not significantly affect canister dimensions

Melter Mixing

The Nd which was added as a tracer in WP-14 was tracked in the glass product and evaluated against models for a continuously stirred tank reactor and plug flow. As expected, excellent agreement was obtained between the predicted Nd concentration in the glass using the continuously stirred tank reactor model and the actual Nd concentration in the glass product. A four day hold was placed on glass pouring to allow the melter to mix after a portion of the third canister was filled. For a continuously stirred tank reactor the average concentration in the melter is the same as the outlet concentration. For a plug flow model the average concentration would be greater than the outlet. After the four day hold there was no significant effect on the difference between the predicted and actual concentrations further indicating that the DWPF melter was best represented by a continuously stirred tank reactor.

TRANSITION TO RADIOACTIVE OPERATIONS

Due to SRS Tank Farm processing and blending strategies the DWPF will begin Radioactive Operations with a sludge only (no PHA) process. A frit composition higher in alkali, to compensate for the missing PHA, will be mixed with this first radioactive sludge batch. A dilute formic acid solution will be added to the SRAT in place of the PHA. The major components of this first batch of radioactive sludge (on a dried solids wt% basis) are: Al - 6.39; Fe - 24.6; Na - 8.74; U - 3.42; Mn - 2.53; Ca - 2.38; Mg - 1.16.

This first batch of sludge has been washed five times to remove the soluble salts. Inhibited water has been added to the tank and mixing accomplished using four long-shaft slurry pumps. The wash water (containing the soluble salts) was transferred out of the tank and fresh water brought in five different times.

Currently, the DWPF is processing two sludge only batches of simulated feed using a sludge simulant that closely matches the first batch of radioactive sludge. After the completion of these batches and startup authorization has been given the DWPF will introduce radioactive sludge into the plant to begin the process of immobilization of high level waste in borosilicate glass.

CONCLUSIONS

The DWPF Startup Test Program has allowed the DWPF to demonstrate the functionality of its systems, exercise and refine operating procedures, and provide operators and engineers an opportunity to run the facility prior to introducing radioactive feed. During the Waste Qualification Runs portion of the Startup Test Program, DWPF has demonstrated its ability to comply with the WAPS providing assurance that the facility can produce a product acceptable for disposal. The results of this testing provide valuable experience for future operations and provide assurance that DWPF can effectively immobilize the SRS liquid high level waste in borosilicate glass.

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Table 1. Range of Major Elements in Feed Compositions over Waste Qualification Runs.

Element	Simulated Sludge (amounts in wt%)
Al	4 - 9
Fe	14 - 25
Na	3 - 7

Element	Melter Feed (Frit, PHA, Sludge)	
	wt% elemental	wt% on oxide basis
Al	1.7 -3.2	3.3 - 7.1
B	1.9 - 2.3	6.0 - 7.3
Fe	5.0 - 8.9	7.2 - 12.8
Na	5.6 - 7.6	7.6 - 10.2
Si	21.8 - 24.6	46.6 - 52.2

Table II. Summary of Canister Production During Startup Test Program.

Campaign	Canisters Produced	Glass Pour Samples	Canisters Temporarily Sealed	Canisters Frit Blasted	Canisters Welded	Number of Glass Samples*
FA-13	16	13	16	10	11	40
WP-14	7	7	6	5	2	76
WP-15	20	18	19	17	17	86
WP-16	19	17	14	13	13	85
WP-17	9	8	5	6	2	40

* Glass samples taken after canister sectioning or wall removal (excludes glass pour stream samples).

Table III. Product Consistency Test Results for Waste Qualification Runs Campaigns Compared to the results of the Environmental Assessment Glass.

Element	Campaign Mean (g/L)	EA Glass (g/L)[8]
WP-14		
B	0.31	16.70
Li	0.35	9.57
Na	0.32	13.35
WP-15		
B	1.05	16.70
Li	1.01	9.57
Na	1.01	13.35
WP-16		
B	0.93	16.70
Li	0.91	9.57
Na	0.91	13.35
WP-17		
B	0.97	16.70
Li	0.95	9.57
Na	0.95	13.35

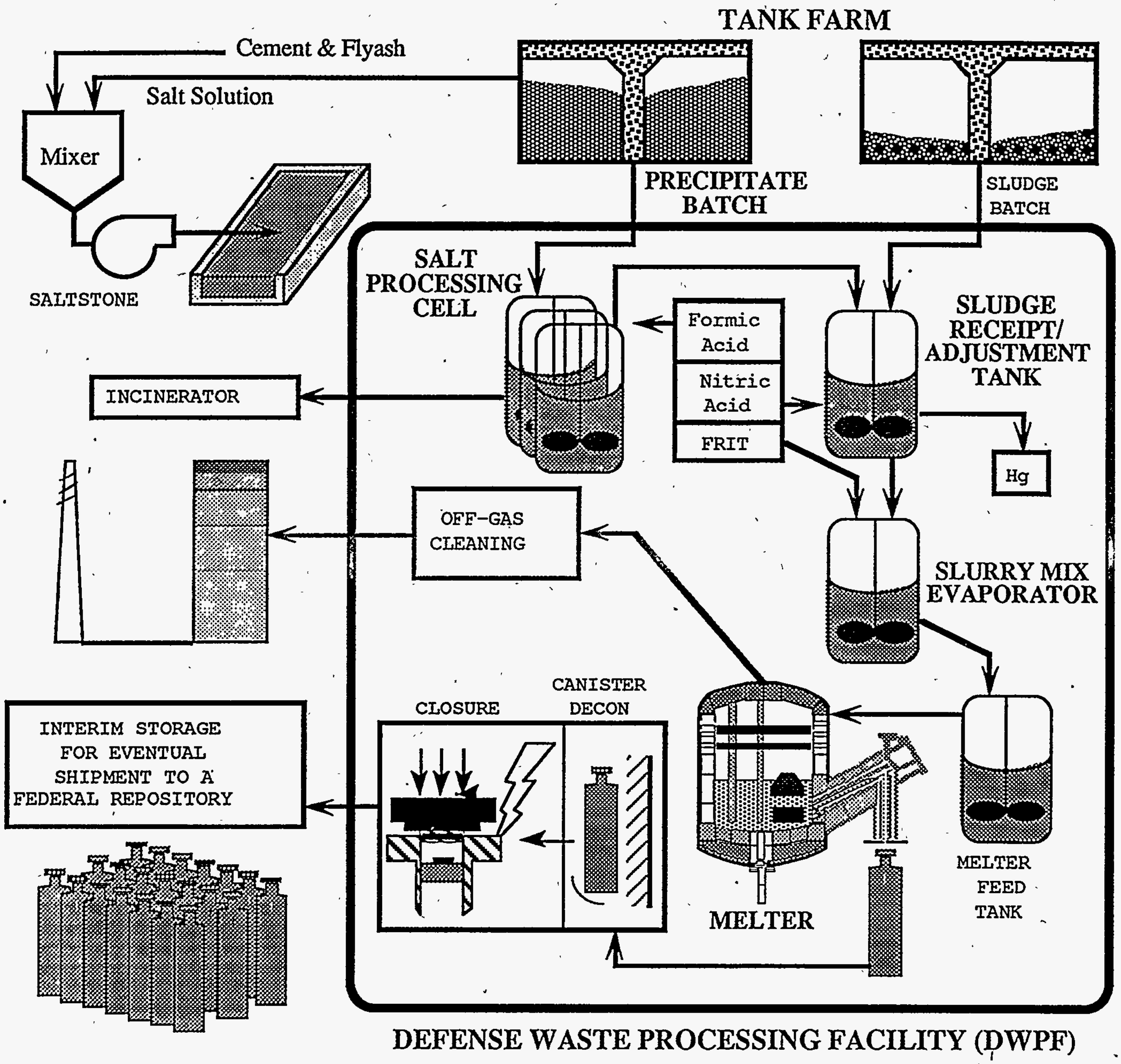

Figure 1. Immobilization of Savannah River Site Radioactive Waste.

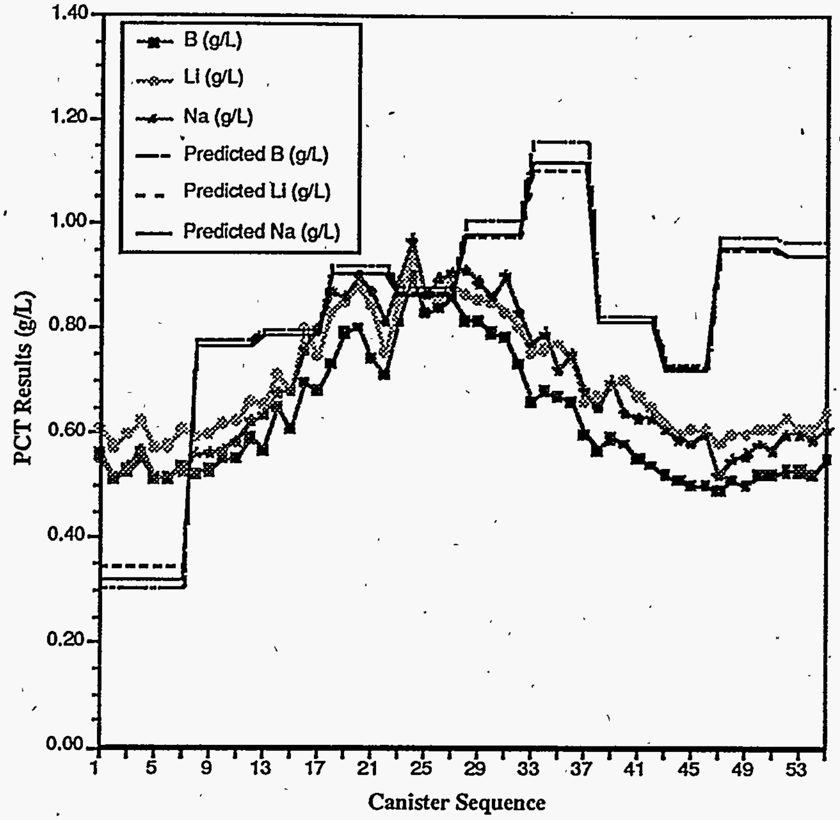

Figure 2. Product Consistency Test (PCT) Results from Waste Qualification Runs Canisters Compared to Predictions based on SME Batch Compositions.