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Revision 5

DWPF GLASS PRODUCT CONTROL PROGRAM (U)

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REVISION PAGE

Revision 5, August 2004

General: Changed and clarified the organizations responsible for verifying the acceptability of the SME batch. Removed the Quality Assurance term "Hold Point" from the text. Updated organizational references.

Revision 4, June 2003

General: Updated definition of a macrobatch. Removed references to sampling of the MFT and removed references to performing direct analysis of radionuclides at DWPF. Updated organizational references.

Revision 3, February 2002 (incorporated G-DCF-S-00244)

General: Updated organization names/management titles and references.

Section 1.4: Added Note for precipitate processing status.

Section 2.1: Removed unnecessary details related to canister filling/venting.

Section 2.2: Deleted discussion of Frit 202.

Section 3.1: Clarified Engineering's role in certifying Production Records.

Sections 3.2 / 8.2: Deleted requirement for Operations' review/approval of Production Records.

Section 5.2: Included discussion of frit change for operational reasons.

Section 5.9: Replaced Frit 202 with Frit 200 and removed unnecessary details from frit discussion.

Sections 5.8 / 7.1: Deleted the term "periodically" from glass sampling discussion.

Section 8: Revised discussion of Production Record transmittal to DOE.

Section 8.1: Added additional details on IAEA WAPS requirements.

Revision 2, July 1997

General: Updates of organizational changes, terminology, definition of macrobatch, removal of reference to redox determination, and update of references.

Table 2: Revised data to match WQR Volume 1 - changes made to correct valence of Mn.

Section 5: Added reference to alternate standard.

Section 7.2: Clarified information to be received from SRNL.

Section 8: Added discussion on new WAPS 1.6 and 3.14.

Revision 1, January 1995

General: Updates of organizational changes and terminology.

Section 5: Deleted reference to Fe^{2+}/Fe (total) for redox determination.

Figure 8: Modified figure to reflect sampler modification.

Section 8: Modified concurrence process for Production Records.

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ABSTRACT

In the Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS), the radionuclides contained in the 130 million liters of liquid high-level waste currently stored at SRS will be converted into a durable borosilicate glass. This glass in its sealed stainless steel canister, the DWPF product, eventually must be sent to a federal repository for final disposal. In order to ensure that the DWPF product is suitable for acceptance by the U. S. Department of Energy's (DOE) Civilian Radioactive Waste Management System (CRWMS)*, the DOE has established the Waste Acceptance Product Specifications for Vitrified High Level Waste Forms (WAPS).[1]

The specifications are divided into five sections, dealing with the waste form (borosilicate glass), the canister, the canistered waste form, quality assurance, and documentation and other requirements. The DWPF is required to document its compliance with the specifications in the Waste Form Compliance Plan (WCP), the Waste Form Qualification Report (WQR), and in the Production Records, or the Storage and Shipping Records.

The WCP provides general information about the DWPF process and product, and a general description of the methods and programs by which the DWPF will demonstrate compliance with each specification in the WAPS.

The WQR is a compilation of the results of the testing and analysis programs identified in the WCP. The WQR details the method of compliance with each specification, and the testing which has been performed to demonstrate the effectiveness of each method. The WQR is published in separate volumes which each correspond to one or more of the specifications. The WQR also contains a description of the DWPF's Glass Product Control Program. Each volume contains Part and Item designators which indicate the corresponding item(s) in the WCP. The Production Records, and the Storage and Shipping Records, document that each DWPF product has been produced and stored in compliance with the WAPS.

This document, Volume 6 of the WQR, describes the DWPF Glass Product Control Program. The DWPF has developed the Glass Product Control Program to ensure that:

- The waste glass produced satisfies Specification 1.3 in the WAPS (Product Consistency Specification) and that
- There is documented evidence that this has been achieved.

The DWPF plans to assure an acceptable product by implementing a combination of administrative and process controls. The purpose of this document is to describe those controls in detail. Detailed discussions on the technical bases for this program are presented in Volume 5 of the WQR.

* The CRWMS is the DOE's Office of Civilian Radioactive Waste Management system for acceptance, transportation, storage, and disposal of spent nuclear fuel and high-level radioactive waste. The CRWMS does not include storage the DWPF.

The Glass Product Control Program is based on the operating philosophy that the best way to ensure an acceptable glass product is through control of the composition of the feed to the melter. The DWPF has chosen to control the product primarily through application of controls to the processing of each process batch of feed. The Slurry Mix Evaporator (SME), the final processing vessel where chemical adjustments are routinely made to the melter feed, is the point where DWPF will exercise primary control of the glass product. The Glass Product Control Program consists of the following elements:

- Qualification of waste. For each sludge batch or precipitate batch, the Savannah River National Laboratory (SRNL) will determine if that material can be processed in the DWPF. Samples of waste from the SRS Tank Farm will be analyzed and processed at SRNL prior to transfer of the waste to DWPF.

- SME sampling and analysis. Each SME batch must be sampled and analyzed prior to transfer of that material to the Melter Feed Tank (MFT). The uniformity of slurries in the SME and MFT must also be maintained as part of this element.

- Determination of acceptability of SME batches. An acceptance methodology currently implemented in the Product Composition Control System (PCCS) has been developed which employs a correlation between glass composition and Product Consistency Test results and statistical algorithms to determine whether SME batches will produce glass which will satisfy the product consistency specification.

- Remediation of SME batches. In the unlikely event it cannot be shown that a SME batch will produce glass which would satisfy the product consistency specification, remediation may be necessary. The same acceptance methodology discussed above will be used to determine whether possible remediating actions will produce an acceptable product.

- Reporting. The DWPF has elected to report on a macrobatch basis, and thus has identified the macrobatch as the waste type in the specification. In the Production Records, the DWPF will verify that the glass product satisfies the product consistency specification through two forms of evidence. The set of predicted PCT results (calculated as part of the determination of SME batch acceptability) from all SME batches in the macrobatch will be used to calculate an average and standard deviation which will be included in the Production Records. The average chemical composition of the macrobatch (see WQR Volume 2) will also be used to develop a separate prediction of the PCT results for inclusion in the Production Records. As requested by the Department of Energy's Office of Civilian Radioactive Waste Management, the DWPF will also include the predicted PCT results for each SME batch in the Production Records.

- Confirmation through glass sampling. A glass sampler has been developed and tested. Samples taken during production with the DWPF glass sampler will be subjected to the PCT, and the results used to confirm that the glass satisfies the product consistency specification. These test results will also be reported in the Production Records.

1. INTRODUCTION

1.1 Scope

The Defense Waste Processing Facility (DWPF) will control the consistency of the glass product by controlling the chemical composition of the melter feed. The chemical composition will be controlled using the DWPF Glass Product Control Program which has been designed to ensure that:

- The waste glass produced satisfies Specification 1.3 in the Waste Acceptance Product Specifications (WAPS)[1] (Product Consistency Specification) and that,

- There is documented evidence that this has been achieved.

This document focuses on the process and administrative controls that the DWPF will implement to ensure an acceptable product. The actions which will be taken to control the chemical composition and to verify that it has been controlled are described. The organizations within the DWPF and the Savannah River National Laboratory (SRNL) which will perform these actions are also outlined.

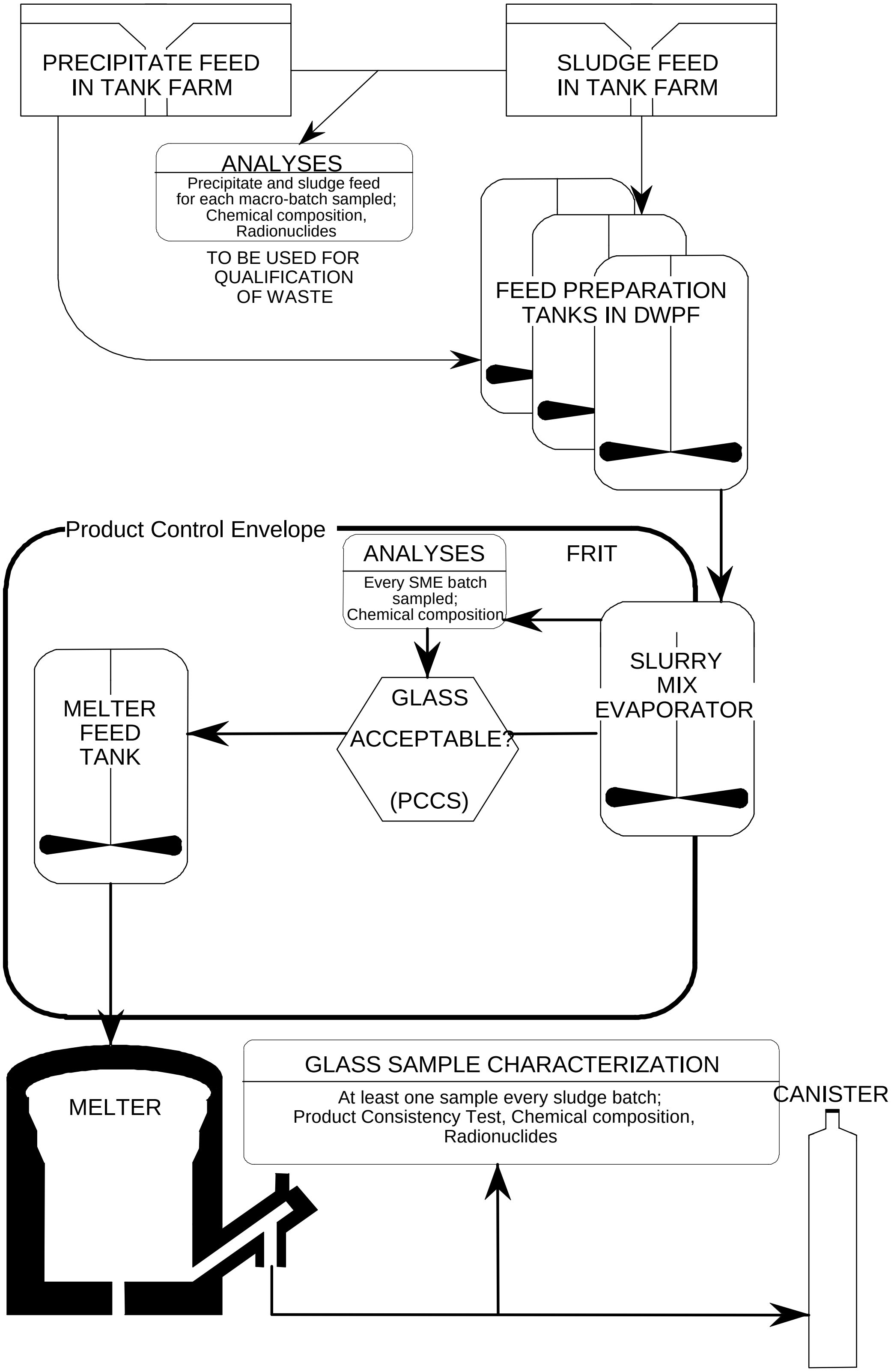
This document is limited to control of the glass product; controls for other WAPS-related items are described in other volumes of the Waste Form Qualification Report (WQR). Detailed technical information and results from laboratory, pilot plant studies, and the DWPF Startup Test Program for the Product Consistency Specification are provided in WQR Volume 5.[2] The other specifications relating to the glass product deal with reporting of its chemical composition and radionuclide inventory and phase stability. For completeness the reporting specifications are discussed in this document, but more detailed information is contained in Volumes 2[3] and 4[4] of the WQR. Specification 1.4, dealing with phase stability of the glass product, is discussed in Volume 7[5] of WQR.

1.2 Product Control Actions

The DWPF has chosen to control the chemical composition of the melter feed in the Slurry Mix Evaporator (SME). No feed will be allowed to leave the SME and be transferred to the Melter Feed Tank (MFT) until it has been determined that it will produce an acceptable glass product. The chemical compositions of samples taken from the SME will be used to determine if an acceptable glass product will be produced from that SME batch. The chemical compositions of the SME batches that make up each macrobatch (see Section 1.4) will be used to verify that the feed has been controlled. This portion of the DWPF process where control is applied is designated the product control envelope (see Figure 1). The elements of the Glass Product Control Program for controlling chemical composition are:

- Qualification of waste for DWPF processing (see Section 5.2)
- SME batch sampling and analyses (see Section 5.3)
- Determination of the acceptability of SME batch (see Section 5.4)
- Adjustment of process batch in SME, if necessary (see Section 5.5)
- Reporting of results, to show that an acceptable feed (thus an acceptable glass) has been produced, in the Production Records (see Section 5.7)

Figure 1. DWPF Product Control



- Confirmation that an acceptable glass has been produced, through glass pour stream sampling (see Section 5.8 and Section 7).

1.3 Product Consistency Specification for the DWPF Glass Product

The Department of Energy has established specifications for the DWPF product.[1] By meeting these specifications, the DWPF provides assurance to DOE that the DWPF product is consistent, durable glass with a bounded composition and geometry and that the DWPF will provide the repository program with information considered necessary for licensing.

The specifications require that the DWPF provide detailed information about, and control the production of: the waste form (borosilicate waste glass), the canister, and the sealed canistered waste form. The specifications further require that activities performed by the DWPF to comply with the specifications, be performed in accordance with approved quality assurance requirements.[6]

The Glass Product Control Program ensures that the DWPF will consistently produce an acceptable glass product which satisfies Specification 1.3 (see below). The DWPF has chosen to demonstrate acceptance using the approach that is described in section 1.3.1 of the specification (see WQR Volume 5[2]).

> **1.3 SPECIFICATION FOR PRODUCT CONSISTENCY**
>
> **The Producer shall demonstrate control of waste form production by comparing, either directly or indirectly of indirectly, production samples to the Environmental Assessment (EA) benchmark glass.[*] The Producer shall describe the method for demonstrating compliance in the WCP and shall provide verification in the Production Records. The Producer shall demonstrate the ability to comply with the specification in the WQR.**
>
> **1.3.1 <u>Acceptance Criterion</u>**
>
> **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 form.[***] The measured or projected mean PCT results for lithium, sodium and boron shall be provided in the Production Records. The Producer shall define the statistical significance of the reported data in the WQR. 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.**

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[*] C.M. Jantzen, "Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material," WSRC-TR-92-346, Westinghouse Savannah River Company, Aiken, SC (September 1992).
[**] ASTM Standards, American Society of Testing and Materials, Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT), C-1285-94.
[***] U. S. Department of Energy, **Environmental Assessment-Waste Form Selection for SRP High-Level Waste,** USDOE Report DOE/EA 0179, Washington, D. C. (1982).

1.3.2 Method of Compliance

The capability of the waste form to meet this specification shall be derived from production glass samples and/or process control information.

Production Records shall contain data derived from production samples, or process control information used for verification, separately or in combination. When using process control information to project PCT results, the producer shall demonstrate in the WQR that the method used will provide information equivalent to the testing of samples of actual production glass.

As required by the WAPS, the DWPF has prepared a Waste Form Compliance Plan (WCP)[7] which describes the strategy the DWPF will use to comply with each specification. The compliance strategy for each of the WAPS specifications has been extensively reviewed by independent technical groups, and has been found to be an appropriate means to meet the specifications. The activities which will be performed during production to satisfy specification 1.3 and the testing to demonstrate their effectiveness are described in Part 3, Item 500, of the WCP. The results of the testing are contained in Volume 5 of the WQR [2].

1.4 Definition of DWPF Production Batches

Glass production in the DWPF can be thought of as a series of batch processes with a continuous output. The DWPF will control the process at the process batch level but report at the macrobatch level (see Section 2.3). The following is a list of definitions of the production batches that will be referred to in this document (also see Figure 2).

Note: It has been determined that the precipitation process as described in the WCP and WQR cannot be performed in the In-Tank Precipitation Facility (ITP) as configured. Until details on the replacement technology for processing HLW salt are finalized, the new process is "to be determined" (TBD) and the discussion of the precipitation process will not be revised.

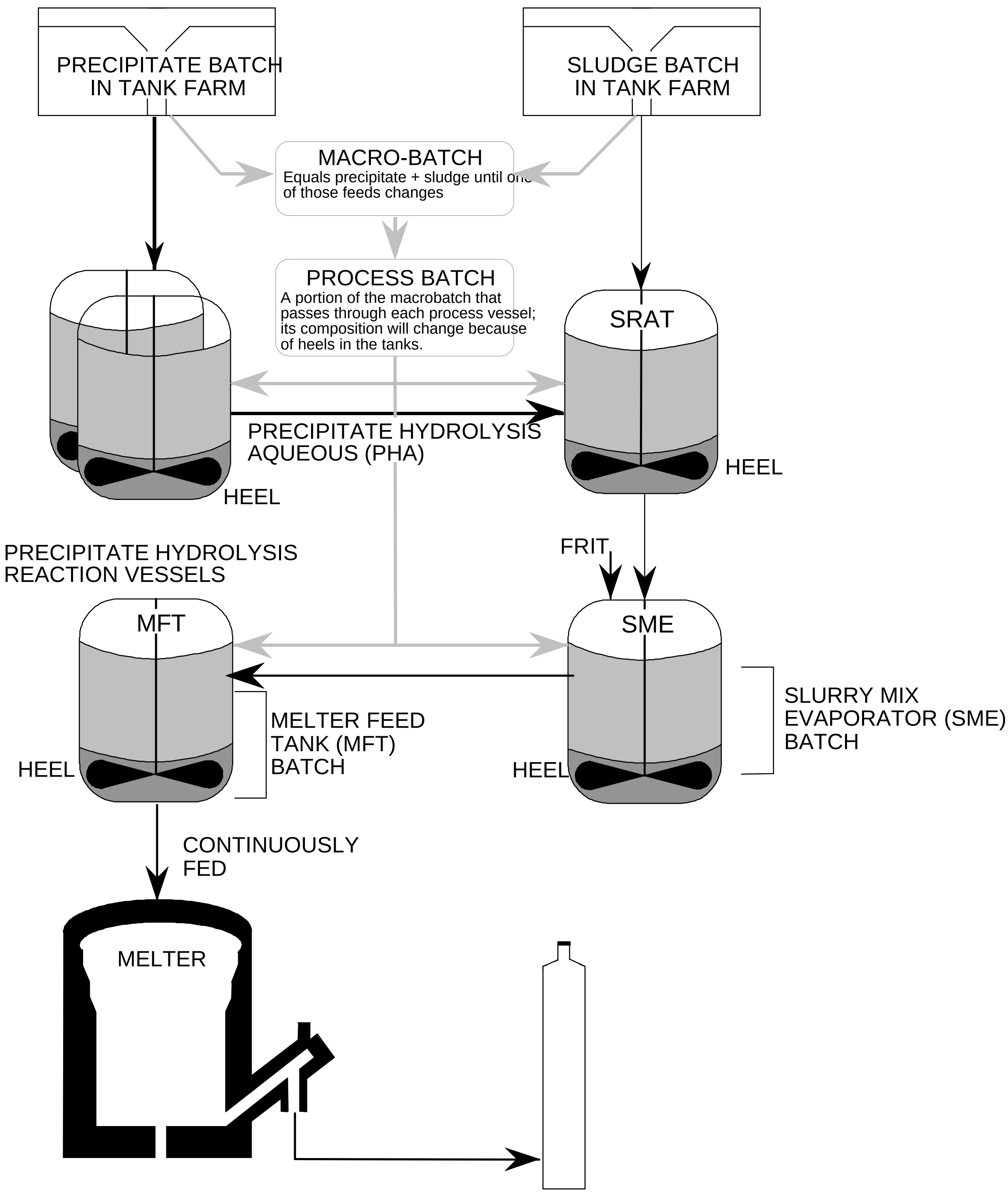
Macrobatch

A macrobatch is defined as follows. The sludge portion of the waste will remain constant for extended periods of time (e.g. years). The large quantity of sludge in the waste tanks will make up "sludge batches" for DWPF. However, the feed to DWPF may change more frequently based on additions to an already qualified sludge batch, the introduction of a new stream to DWPF, or a significant change to frit composition. The periods of relatively constant composition between process changes will constitute a macrobatch. A single sludge batch may provide enough feed for several different macrobatches.

MFT (Melter Feed Tank) Batch

The contents of the melter feed tank constitute an MFT batch. This includes the material transferred from the SME and the heel (see below) that was already in the MFT. A comparison of analyses between the SME and MFT has shown no significant composition differences between the two tanks.[39] One MFT batch will yield approximately 5-8 canisters.

Figure 2. DWPF Production Batches



Precipitate Batch

As currently planned, the precipitate is prepared in the Tank Farm in cycles. Each cycle constitutes a precipitate batch as defined in this document and together with the sludge batch, makes up the feed to DWPF. Each precipitate batch (cycle) will provide enough feed for the DWPF for several months at a time. However, even when transitioning from cycle to cycle, the precipitate composition will remain fairly constant.

Process Batch

A portion of the macrobatch as it passes through each process vessel; its composition changes because of heels (see below) in the tanks. The DWPF process is controlled at this level.

Sludge Batch

The sludge is prepared in the Tank Farm in 500,000 to 900,000 gallon batches, for delivery to the DWPF. This sludge batch will supply the DWPF with material for extended periods of time depending on production rates and high level waste processing schedules.

SME (Slurry Mix Evaporator) Batch

The contents of the Slurry Mix Evaporator (SME) constitute a SME batch. This includes the material transferred from the Sludge Receipt and Adjustment Tank (SRAT) as well as the heel (see below) remaining in the SME. The SME is the point where DWPF will exercise primary control of the glass product.

> **Heel:** Whenever a process batch is transferred from one DWPF vessel to another (e.g., from the SME to the MFT), a portion of the process batch remains behind. This remaining portion of the process batch is referred to as the heel. See WQR Volume 5[2] for a discussion of the role of the heels.

2. DWPF OVERVIEW

2.1 Process Description and Product Control

The waste generated from over thirty years of reprocessing of irradiated nuclear fuels for national defense purposes, is stored at the Savannah River Site (SRS). Approximately 130 million liters of alkaline waste are currently stored in underground carbon steel tanks in the SRS Tank Farm. This SRS high-level radioactive waste (HLW) will be immobilized in a durable borosilicate glass in the Defense Waste Processing Facility (DWPF).[8] Descriptions of the DWPF and its mission have appeared in the open technical literature.[9-13] A diagram of the entire waste immobilization process is shown in Figure 3.

2.1.1 Feed Preparation

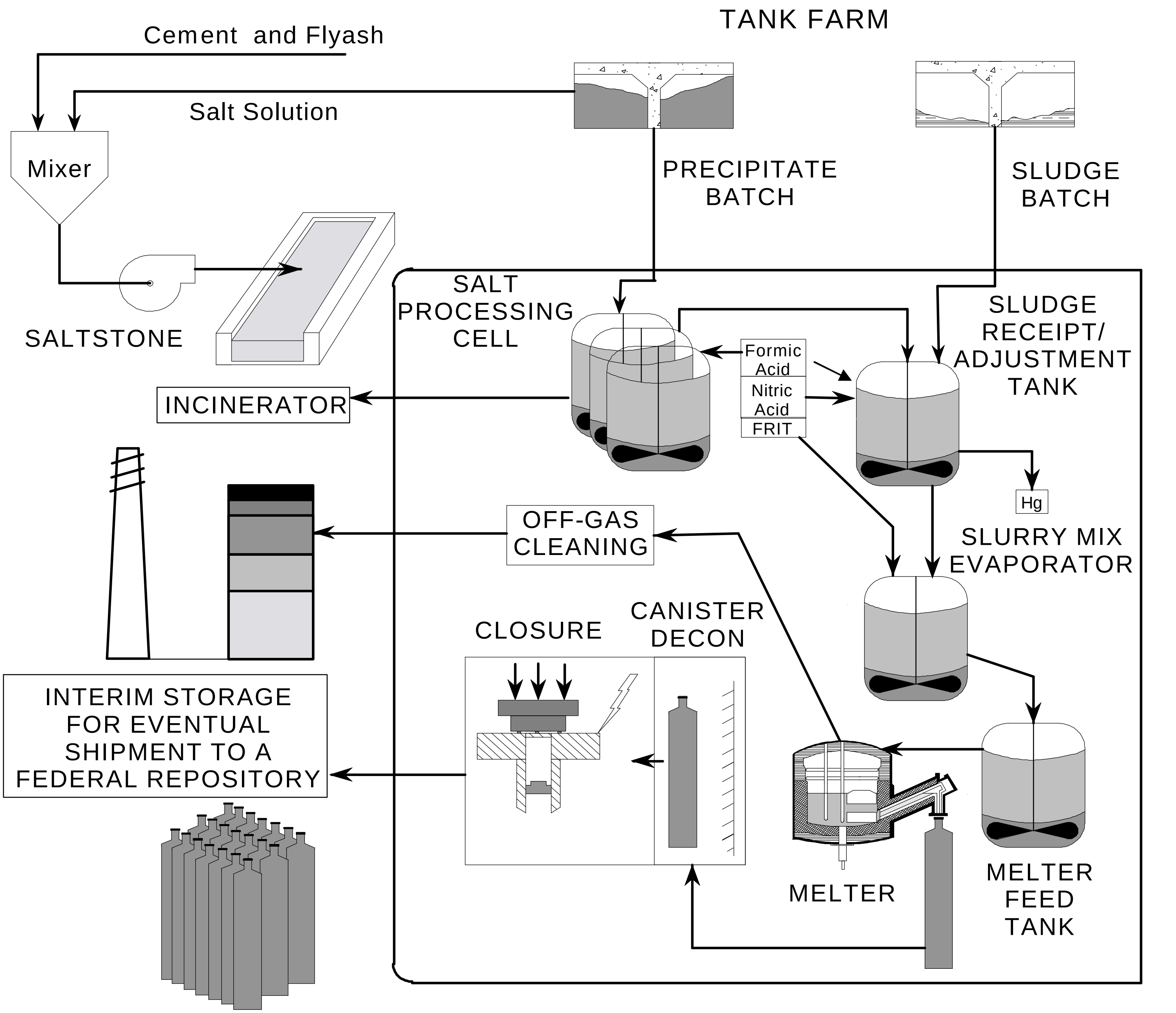
The feed to the glass production process in the DWPF will consist of three streams: sludge, the precipitate hydrolysis aqueous (PHA) product, and frit. The sludge is the insoluble solids fraction of the waste in the Tank Farm, and contains nearly all of the long-lived radionuclides in the waste. Before it is vitrified, it undergoes processing in the Tank Farm to remove soluble inert salts,[15] and further processing in the DWPF to remove mercury. In the DWPF, the sludge slurry is treated with nitric acid in the SRAT to neutralize the alkaline waste.

The PHA is derived from the soluble portion of the waste in the Tank Farm. The soluble wastes are treated with sodium tetraphenylborate in the Tank Farm to remove radioactive cesium from the soluble waste by precipitation, and with sodium titanate to absorb the trace quantities of plutonium and strontium in the soluble waste.[14] The solid materials (tetraphenylborate salts and titanate) which now contain most of the radioactivity from the soluble waste are separated from the soluble salts by filtration. This slurry is then reacted with formic acid in the DWPF to remove most of the organic material by hydrolysis. The aqueous product from this hydrolysis process (called PHA, and which is primarily sodium, boron, potassium, titanium, and cesium) is mixed with the sludge in the Sludge Receipt and Adjustment Tank (SRAT). The slurry is then passed forward to the Slurry Mix Evaporator (SME) where the frit is added.

For each new batch (see Section 2.3) of material, SRNL will analyze the chemical composition and the radionuclide inventory of the sludge and precipitate batches in the Tank Farm. These analyses are then used by SRNL to verify that the correct frit composition has been specified for procurement for the DWPF (see Section 5.2). SRNL will also use this information to evaluate the sampling regimen and the covariance matrix structure, as needed, so that the DWPF can be assured that the acceptance criterion specified in the WAPS (see Section 1.3) will be met for each SME batch before it is allowed to pass forward to the Melter Feed Tank (MFT). The radionuclide inventory of the waste is used to identify the radionuclides for reporting and calculate the radionuclide inventory of the glass waste form (see Section 8).

Frit is a premelted granular borosilicate glass, which allows the DWPF to introduce glass-forming chemicals into the melter feed in consistent and controlled manner. The frit composition is tailored to combine with the wastes to form an acceptable product. Frit is procured from outside vendors to tightly controlled specifications (see Section 5.9). The frit is blended into the waste slurry in the SME. Approximately 90% of the necessary frit is pumped directly to the SME. The remaining

Figure 3. Immobilization of Savannah River Site Waste.



10% of the frit is used for canister decontamination (frit blasting), and then is added to the SME. During processing of each macrobatch, the DWPF will control the chemical composition of the glass by controlling the composition of each SME batch (batch of feed processed in the SME). No feed will be allowed to leave the SME until it has been determined that the feed will make acceptable glass (see Section 4.2). Control will be achieved through the following steps:

- Sampling of the material in the Slurry Mix Evaporator (SME).[3]
- Analyses of the samples (chemical composition).[3]
- Evaluation of the acceptability of the feed (see Section 5.4).[2]
- If necessary, adjustment, resampling, and then re-evaluation of the acceptability of the feed (see Section 5.5).[2]

After the Slurry Mix Evaporator (SME) batch is determined to be acceptable it is transferred forward to the Melter Feed Tank (MFT). The results of the SME analyses will be used to report the chemical composition of the macrobatch. PCT results projected from this macrobatch composition, along with the predicted PCT results (using the PCT/chemical composition correlation) from each SME batch provide verification that the chemical composition of all of the glass produced for an entire macrobatch has been controlled (see Section 5.7). The number of samples taken from each process batch will be sufficient to verify that the glass produced from the entire macrobatch is acceptable (able to meet Specification 1.3). Glass pour stream samples are used to provide confirmation of glass acceptability. See Figure 1 and Table 1 for a listing of the sampling points and analyses important to the Glass Product Control Program.

2.1.2 Vitrification

Vitrification of SRS waste is accomplished in a joule-heated, slurry-fed melter. Only feed which will produce an acceptable glass product will be delivered to the melter. The feed slurry is introduced from the top of the melter trough feed tubes. The basic geometry and general characteristics of the melter are show in Figure 4. The glass melt is maintained at a nominal temperature of 1150ºC, by electric current passing through the melt. The current is supplied to melt by two pairs of opposing electrodes. The resistance of the molten glass converts the electrical energy into heat. Temperatures within the melter range between 1050 and 1175ºC. The nominal melter residence time is about 65 hours, which allows ample time for the melt to homogenize via thermal convection currents.[15]

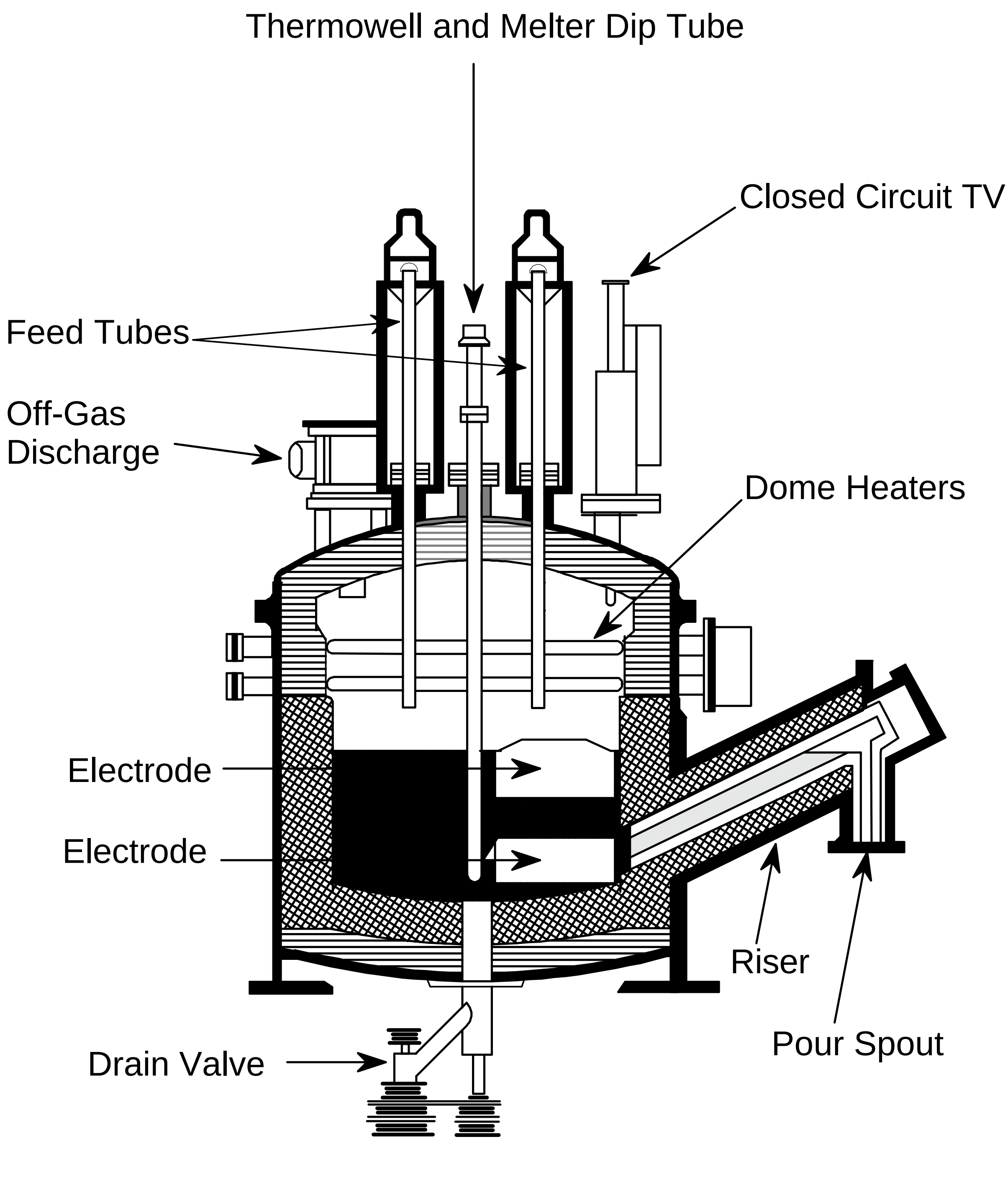
Four pairs of horizontal resistance heaters (dome heaters) are installed in the vapor space of the melter vessel. These heaters are used to supply the heat which initially melts the glass at melter startup (The electrodes are only effective when a molten pool is available.). During normal operation, the heaters are kept a 950ºC to maintain the melter plenum vapor space at 600-800ºC, to increase melter throughput.

Table 1. DWPF Analytical Sample Schedule for Glass Product Control and Reporting.

Sample	Analysis	Analysis Frequency	Analysis Performed by
Washed Precipitate (Tank Farm)	Radionuclides Elementals	Each precipitate batch	SRNL
Washed Sludge (Tank Farm)	Radionuclides Elementals	Each sludge batch	SRNL
Frit	Elementals Particle size	Each lot (~30,000 lbs)	Independent laboratory
Slurry Mix Evaporator (SME)	Elemental	Each SME batch	DWPF Analytical
Glass Pour Stream	Radionuclides Elementals PCT† Release (Na, Li, B)	One/sludge batch (min.) One/sludge batch (min.) One/sludge batch (min.)	SRNL SRNL SRNL

† Product Consistency Test

Figure 4. DWPF Melter.



2.1.3 Canister Filling

During the pour cycle the molten glass is drawn through the melter throat near the bottom of the melter, up into a heated riser, and down a heated pour spout into the stainless steel canister (see Figure 4). The nominal pouring temperature is 1050-1100ºC. A pour spout bellows assembly connects the pour spout and the canister, providing a flexible, leak-tight seal between them. Glass is poured from the melter by establishing a vacuum above the canister nozzle relative to the melter vapor space.[16] This pressure differential causes the glass to flow out of the melter, through the riser, and into the canister.

In order to fill a canister, the in-cell crane must lift an empty canister from the transfer cart or the storage rack and place it on the pour turntable assembly. A throat protector is inserted into the canister using the in-cell crane. The throat protector prevents glass or condensibles from depositing on the inside surface of the canister neck. Immediately prior to pouring, the canister is rotated into position beneath the melter pour spout, the canister positioning arm is secured, and the bellows assembly is sealed in place. If a glass sample is to be taken (see Section 7.2), a modified throat protector is used, which contains a glass sampler. At least one glass sample will be taken from each sludge batch (see Section 7.0). The glass sample(s) will be sent to the Savannah River National Laboratory (SRNL) for independent characterization which includes determining the chemical composition, radionuclide inventory, and performing the Product Consistency Test. These data will be used to provide independent confirmation that the properties of the glass were controlled.

Once the canister is filled with waste glass, the canister remains connected to the pour spout to vent radioactive gases to the off-gas system. After the canister has been vented, the pour spout bellows is disengaged and the canister is rotated out from under the melter pour spout. The throat protector is removed using the in-cell crane, and the inner canister closure (ICC) plug is inserted into the nozzle of the canister to make a temporary shrink-fit seal.[17] The primary purpose of this temporary seal is to prevent the entry of water into the canister from the canister decontamination process.

After decontamination, the canister is sealed by upset resistance welding of a plug into the canister nozzle.[18] The canister is then transferred to an air-cooled vault (in the Glass Waste Storage Building) for interim storage. The canistered waste forms will be held there to await shipment to a federal repository.

Detailed discussions of all facets of production of DWPF canistered waste forms are contained in other volumes of the WQR.

2.2 Product Description

SRNL began an evaluation of the ability of a wide range of candidate waste forms to immobilize SRS HLW in 1973.[19] Among the 17 waste forms studied were: borosilicate glasses, aluminosilicate glass, crystalline ceramics, coated particle forms and cement. In 1982, borosilicate glass was chosen as the DWPF waste form, based on its combination of good durability and ease of processing.[20]

The nominal DWPF waste form consists (on an oxide basis) of ~28 weight % sludge solids, 8 weight % PHA waste, and 64 weight % glass frit.

2.2.1 Composition

During the operation of the Savannah River Site, the composition of the waste generated during nuclear fuel production has varied.[12] As a result of these wastes having been stored in different tanks, the composition of DWPF waste glass will vary over the lifetime of the vitrification facility. To prevent large swings in waste composition, which might adversely affect the glass-making process, different streams of waste are blended in batchwise manner. Table 2 provides seven projected or potential DWPF waste glass compositions. These glass compositions represent:

- A nominal waste glass composition produced from an overall blend of existing waste inventory (the basis for the design of the process).
- The projected glass compositions to be produced in the DWPF during the first ten years of operation (Batches 1-4).
- A blend of high aluminum (HM) wastes that represents the upper design limit of glass viscosity.
- A blend of high iron (Purex) wastes which represents the lower design limit of glass viscosity. This glass composition is based on the following assumptions: maximum precipitate feed rate to the DWPF, minimum sludge feed rate, minimal removal of soluble salts during sludge processing in the Tank Farm, and use of a frit higher in alkali than the frit which will be used in the DWPF during initial coupled operations. This composition represents a possible, though unlikely, worst-case composition due to the high alkali and boron content which makes glass less durable.

The composition of the feed streams, i.e. sludge, precipitate hydrolysis aqueous (PHA), and frit, and the blending scheme determine the composition of the waste glass. The sludge consists of hydroxides and hydrous oxides, and contains most of the stable and radioactive fission products and actinide elements in the glass, as well as elements added in the SRS separations processes. These hydroxides are primarily iron, manganese and aluminum. The activity of the sludge feed is ~130 Ci/gal. Of this activity, about 75% is due to Sr-90, Y-90 and Pm-147.[12]

The PHA contributes a large fraction of the alkali present in the glass (Na_2O and K_2O), as well as B_2O_3 and TiO_2. The activity of the PHA feed is ~80 Ci/gal. Of this activity, about 99% is due to Cs-137 and its β-decay daughter Ba-137m.

The frit supplies the raw glass feed stock for the vitrification process. The chemical composition of the frit may be deliberately varied over the life of the DWPF to accommodate changes in composition of the blended waste batches (see Section 5.2).

2.3 Operational Considerations for Control and Reporting

Glass production in the DWPF can be thought of as a series of batch processes, culminating in a continuous output. In the Tank Farm, the sludge is prepared in large sludge batches for delivery to the DWPF, which supply feed to the DWPF melter for years at a time (dependent on production rates and high level waste processing schedules). Over this period, the sludge component of the waste glass will remain relatively constant. The tetraphenylborate/titanate precipitate will be prepared in cycles which provide feed to the DWPF for months at a time (dependent on production

Table 2. Projected DWPF Waste Glass Compositions.

GLASS COMPONENTS (weight %)	CONSTITUENT SLUDGE TYPE						
	Blend[d]	Batch 1	Batch 2	Batch 3	Batch 4	HM	Purex[w]
Al_2O_3	3.97	4.85	4.44	3.24	3.30	7.06	2.88
B_2O_3	7.98	7.66	7.68	7.68	8.07	6.92	10.17
$BaSO_4$	0.27	0.22	0.24	1.26	0.38	0.18	0.29
CaO	0.96	1.16	0.99	0.92	0.82	1.00	1.01
$CaSO_4$	0.08	0.12	0.11	0.10	0.0034	trace	0.12
Cr_2O_3	0.12	0.10	0.12	0.13	0.14	0.085	0.14
Cs_2O	0.12	0.079	0.081	0.079	0.14	0.073	0.080
CuO	0.44	0.40	0.41	0.40	0.46	0.25	0.42
Fe_2O_3	10.37	12.47	10.57	11.12	11.28	7.36	12.69
Group A[a]	0.14	0.10	0.14	0.10	0.20	0.20	0.077
Group B[b]	0.36	0.22	0.44	0.25	0.60	0.89	0.083
K_2O	3.85	3.47	3.49	3.46	3.98	2.13	3.57
Li_2O	4.38	4.40	4.40	4.40	4.31	4.61	3.10
MgO	1.35	1.36	1.35	1.35	1.38	1.45	1.33
MnO	2.02	2.05	1.62	1.81	3.06	2.07	1.98
Na_2O	8.70	8.58	8.58	8.48	8.85	8.15	12.09
Na_2SO_4	0.10	0.10	0.12	0.095	0.13	0.14	0.12
NaC1	0.19	0.31	0.23	0.22	0.089	0.092	0.26
NiO	0.88	0.74	0.89	1.06	1.08	0.40	1.21
SiO_2	50.01	49.61	50.00	49.81	49.09	54.26	44.39
ThO_2	0.19	0.36	0.62	0.76	0.24	0.55	0.011
TiO_2	0.89	0.65	0.66	0.65	1.01	0.55	0.64
U_3O_8	2.13	0.53	2.29	3.14	0.78	1.01	2.88
Total[t]	**99.50**	**99.54**	**99.47**	**99.52**	**99.39**	**99.43**	**99.54**

[a] Group A - see Table 5 of WQR Volume 1 for more information.

[b] Group B - see Table 5 of WQR Volume 1 for more information.

[d] The "Blend" is the current DWPF Design-Basis glass (see the Glossary in the WCP[1]).

[w] The "Purex" glass is a possible "worst-case" composition.

[t] Minor components constitute difference between indicated total and 100%.

rates and high level waste processing schedules). For this time period, the precipitate feed will remain constant. (However, even within transitioning from cycle to cycle, the precipitate composition will remain fairly constant). Thus, the feed from the Tank Farm to the DWPF will be relatively constant for months at a time. The periods of constant composition at DWPF will constitute a macrobatch (see Section 1.4).

Each macrobatch will be processed in process batches which will pass through the process from vessel to vessel, one batch at a time (see Figure 2). Each vessel will have significant "heels" from previous batches. Therefore, as a process batch passes from the Sludge Receipt and Adjustment Tank (SRAT) to the Slurry Mix Evaporator (SME) to the Melter Feed Tank (MFT) to the melter, that batch no longer has exactly the same composition as it did in the preceding vessel.

2.3.1 Control

The DWPF has chosen to control the product primarily through application of controls to the processing of each process batch (equivalent to approximately 5-8 canisters of glass). SRNL's experience in producing over 1,000,000 pounds of simulated waste glass in a nonradioactive pilot plant leads to the conclusion that the most effective means of control is at that level. Actions taken to control the contents of a single canister necessarily involve the rest of the canisters produced from that process batch; thus the canister is not a useful level of control for this process. Control of the macrobatch can be useful for glass processing purposes; (and, in fact, is used in setting blending targets for qualification of waste - see Section 5.2) but any actions taken to control the macrobatch itself can easily be undone by upsets in processing individual process batches. Thus, the process batch is the appropriate unit of control. DWPF has elected to control the product through control of the process batch while it is in the Slurry Mix Evaporator (SME) because:

- If unacceptable feed is detected, the condition can be corrected in the SME. Either frit or trim chemicals can be added to the SME to adjust the feed composition, if needed. Neither the Melter Feed Tank (MFT) nor the melter have the capability to make such adjustments routinely.

- The SME is the last vessel where chemical additions are routinely made to the melter feed. No chemical additions can be made to either the MFT (without changing piping connections) or to the melter. Thus, actions performed at the SME will not be undone downstream.

- Process batches can be evaluated in the SME without disrupting feed to the melter since the MFT feeds the melter continuously.

2.3.2 Reporting

As discussed in Section 1.4, the feed to the DWPF will remain relatively constant for extended periods of time. This constant feed constitutes a macrobatch which the DWPF will use as the "waste type" referred to in the WAPS. By reporting at this level, the DWPF is likely to provide a more representative picture of the spectrum of glass produced. The results of processing the process batches in the macrobatch should provide estimates of both short- and long-term variability.

See Table 1 for a summary of the samples and analyses important to the Glass Product Control Program.

2.4 Administrative Controls

The DWPF has put in place a set of programs to ensure that the DWPF produces an acceptable product under a rigorous quality assurance program. Detailed discussions of these controls can be found in the DWPF Quality Implementation Procedure Manual[21] and the DWPF Waste Acceptance Reference Manual.[22] The Waste Acceptance Reference Manual contains a list of the items and activities important to product quality, including procedures, equipment, and administrative controls. Some of the controls important to this program include:

- Laboratory Quality Control[23] - All DWPF analytical procedures undergo review and approval and oversight of implementation. The training and qualification of laboratory specialists is controlled to ensure that DWPF analytical results are generated by qualified personnel. In addition, specific parameters of analytical methods that affect quality (e.g. calibration, standardization, and frequency) are controlled. Laboratory control charts are maintained and certified standards are used to monitor and adjust for biases, if necessary. In addition, round robins are held between the DWPF Analytical Laboratory and SRNL to maintain comparability of methods and results. SRNL provides independent confirmation of glass acceptability by performing analyses of production glass samples.

- Control of Measuring and Test Equipment[24] - A system has been established and implemented for controlling Category 1 and 2 measuring and test equipment (M&TE). M&TE includes the tools, gauges, fixtures, reference standards, and nondestructive test equipment that are used in measuring, inspecting, and monitoring items and tests that support product qualification.

- Personnel Qualification and Training - The DWPF uses a systematic approach to define job qualification requirements and performance based training concepts to ensure that the facility is staffed by qualified and trained personnel.

- Procedure Control - In order to ensure that procedures are current, complete, and available when needed, a procedure control program has been implemented.[25]

- Modification Control - Product quality control requires that the DWPF process equipment configuration and the DWPF processes be baselined and controlled. The DWPF has implemented a Configuration Management System.[26]

- Record Control - A Records Management Program has been developed to control the release, identification, control, protection, and distribution of DWPF records.[27] The Production Records specified in the WAPS[1] are DWPF's certification of compliance with the WAPS. The information contained in the Production Records is compiled from operating procedures/manuals (see Section 8 for more information on the Production Records).

- Quality Assurance Surveillance – The act of observing real-time activities and/or reviewing documentation to verify conformance with specified requirements and to evaluate their adequacy and effectiveness.[40]

3. ORGANIZATION

The DWPF is part of the Waste Solidification Projects in the Closure Business Unit and is currently organized in several departments: Engineering, Operations, Training, Plant Support and Quality Assurance. The composite organization responsible for ensuring the acceptability of the glass product is outlined in Figure 5 and is discussed below. Several departments are part of division wide organizations and are therefore independent on a managerial basis from the DWPF (represented in Figure 5 by dotted lines). Other organizations, such as Radiological Protection and Industrial Hygiene, are part of site wide divisions which support the DWPF.

The Savannah River National Laboratory (SRNL) also plays an important role in satisfying the WAPS and supporting the DWPF. SRNL will qualify waste for DWPF processing and characterize samples of production glass (see below).

3.1 DWPF Engineering

The DWPF Engineering department is part of the business unit organization that supports the DWPF. This department provides technical support of DWPF operations. This role includes, but is not limited to, process support and development, software development and control, analytical support services, plant engineering, engineering program development, and liaison activities with external agencies. Also within DWPF Engineering is a subject matter expert for waste acceptance affecting activities who supports cognizant engineers and Quality Assurance on waste acceptance/product quality issues.

DWPF Engineering determines the acceptability of each SME Batch using the Product Composition Control System as a tool (see Section 4.2). If it is determined that the feed is acceptable by Engineering, then the Shift Manager will verify the Engineering acceptability evaluation to ensure that the SME batch is acceptable to transfer forward to the MFT. If the SME batch is determined to be unacceptable, Engineering determines a remediation strategy. In addition, DWPF Engineering signs the Production Records (see Section 8), certifying compliance with the DWPF technical and compliance strategies contained in the Waste Form Compliance Plan (WCP) and the Waste Form Qualification Report (WQR) or exceptions to the strategies (if applicable).

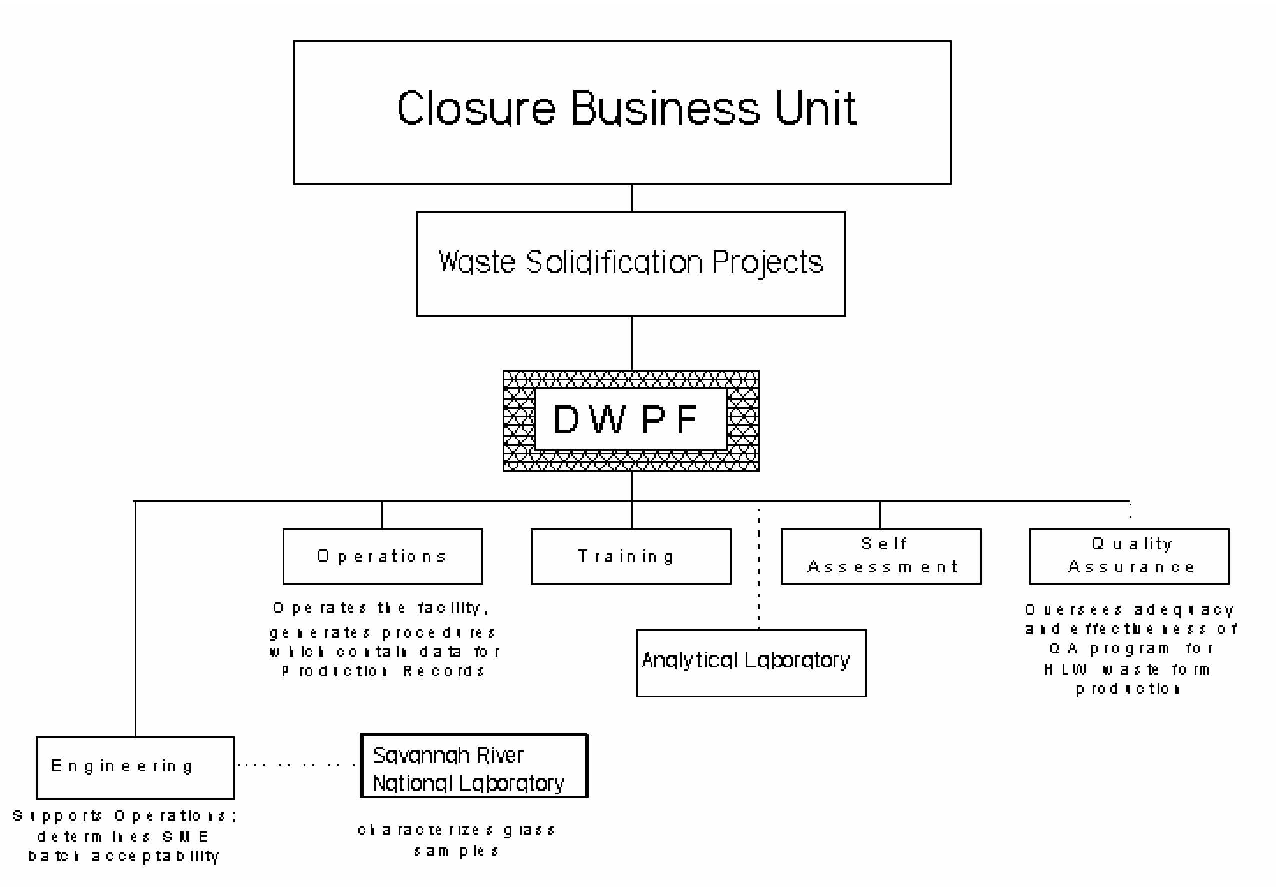
3.2 DWPF Operations

The DWPF Operations organization is responsible for operating the facility and for canistered waste form production.

3.3 DWPF Analytical Laboratory

The DWPF Analytical Laboratory takes samples from the processing vessel in the product control envelope as well as performs verifications. The laboratory group analyzes the samples and provides the results for determination of SME Batch acceptability and for reporting purposes in the Production Records (see Section 8). Analytical results are approved by a supervisor before being released by the laboratory.

Figure 5. DWPF Organizational Structure.



3.4 Quality Assurance

The Quality Assurance (QA) department is part of the business unit organization that supports the DWPF but is independent of the DWPF on a managerial basis. QA overviews the adequacy and effectiveness of the Quality Assurance Program for high-level waste form production. They also perform surveillances and audits at various points in the Glass Product Control Program process.

QA also collects the information from Operations and Engineering that is necessary for the Production Records and then develops the Production Records. QA will sign the Production Records certifying that the data has been verified and that the canistered waste form has been produced in accordance with all applicable quality assurance requirements.

3.5 Savannah River National Laboratory (SRNL)

For each sludge batch and precipitate batch, SRNL will receive samples from the Tank Farm, and characterize the chemical composition and radionuclide inventory. SRNL will demonstrate that the samples of waste from the Tank Farm will produce an acceptable glass, and that the PCT/chemical composition correlations can be used for the actual feed to be processed. SRNL will also characterize any glass pour stream samples that are taken (see Section 7). This includes confirmation of acceptability using the Product Consistency Test (PCT) and determination of the chemical composition, and radionuclide inventory. SRNL also archives these glass samples for the DWPF. Results from this characterization are provided to DWPF Engineering for transmittal to QA for inclusion in the Production Records (see Section 8.0).

4. PARAMETERS TO BE CONTROLLED

The Waste Acceptance Product Specifications require that the DWPF control the consistency of the waste glass product as measured by the Product Consistency Test (PCT) (see Section 7.2). Thus, the DWPF will achieve control of the glass product by controlling the process parameters which affect the results of the PCT. The reactions between the waste glass and aqueous solutions will depend on the environment (water composition including Eh and pH, water flow rate, amount of water in contact with the glass, temperature and duration of glass-water interactions), and the glass composition. The PCT fixes the environment that the glass is exposed to, thus the only parameter which the DWPF can control is the glass composition. Since the waste glass cannot be reworked or remediated, the melter feed material must be determined to be acceptable before being melted.

The properties affecting the short-term durability of the borosilicate glass have been extensively investigated over the last 15 years.[28] As part of this scrutiny, two in-depth studies of the mechanisms of reactions between the DWPF waste glass and aqueous solutions have been performed.[29-30] One study, which involved university, industrial and DOE laboratories, concluded that the reaction mechanisms depend on the composition of the glass, the environmental conditions (groundwater composition, pH and Eh, temperature, flow rate of water and amount of water), and the time of contact between glass and water.[29]

As part of its program to gain acceptance of DWPF glass, Savannah River National Laboratory (SRNL) has also carried out extensive investigations[30-32] of the reactions between waste glass and aqueous solutions. The first of these, which summarized the technical basis for the selection of borosilicate glass as the waste form for the DWPF, concluded that release from glass was governed by exactly the same factors identified in the previously mentioned study. Subsequent studies have reached similar conclusions.[31-32]

The PCT fixes all factors (including the initial pH), except glass composition, that govern the reactions between waste glasses and aqueous solutions. The studies discussed above lead to the conclusion that the property of the glass product which affects the PCT results and is subject to control by the DWPF is the composition of the glass product. DWPF has chosen to control glass composition through control of the composition of the feed in the last feed preparation vessel, the Slurry Mix Evaporator (SME). The method of control is described in Section 5. Direct control of the glass composition is not possible because of the continuous nature of the DWPF process, and the virtual impossibility of collecting and recycling glass at the DWPF. The SME has been selected as the control point for the melter feed composition because it is the last process vessel through which direct chemical additions are routinely made (see Section 2.3.1).

In order to achieve control of the glass composition, a means of consistently assessing its acceptability (ability to meet specification 1.3) is needed. A correlation has been developed between PCT results and chemical composition. This correlation is embedded in the Product Composition Control System (PCCS) which will be used as a tool to guide control of the chemical composition of the feed slurry. The details concerning this correlation are discussed in Volume 5 of the WQR.[2]

4.1 Process Parameters

The DWPF will monitor several hundred parameters as it carries out the process of converting waste slurries to a durable glass. Each of the process steps identified in Section 2.1 will be strictly controlled to ensure that the objectives of each process step are achieved. However, to be effective for the glass product, individual process controls must be applied in such a manner that they cannot inadvertently be overridden by subsequent actions. Conversely, those controls must also be sufficiently flexible so that any unexpected condition can be prevented from impacting the glass product. Specific limits, conditions and administrative controls are incorporated into operating procedures. Limits are set on predicted glass durability (as measured by the PCT), important processing properties, and melter temperature. Important conditions are items such as agitation time prior to sampling and transfer to ensure a uniform material in order to maintain the integrity of the composition control system (see Sections 5 and 6).

4.2 Methodology for Determination of Process Batch Acceptability

The Product Composition Control System (PCCS) is the tool which DWPF personnel are currently using to determine the acceptability of the chemical composition of the SME process batch. The PCCS contains composition/property correlations for important processing characteristics (e.g. viscosity), as well as the glass composition/PCT correlations. The PCCS takes into account uncertainties associated with the analytical system and the correlation between glass composition and the PCT results. A Student's t-test is used to determine if the SME batch composition lies within the region which will produce an acceptable glass product. The methodology, which is currently in PCCS, and its technical bases is described in detail in WQR Volume 5.[2]

The PCCS uses the chemical composition/PCT results correlation (see Volume 5 of the WQR for bases) to provide information for verification that the canistered waste forms from each macrobatch satisfy WAPS 1.3 (see Section 5.7). The PCCS converts the chemical composition of each SME batch to a projected PCT result. The average and standard deviation of these projected leach rates will be documented in the Production Records (see Section 8).

5. CONTROL OF COMPOSITION

For each sludge batch and precipitate batch (see Section 2.3) of material, SRNL will qualify the waste for processing in the DWPF. The DWPF will then control the glass composition through control of the feed composition. This control will be applied to the material in the last feed preparation vessel, the Slurry Mix Evaporator (SME). Each SME batch will be evaluated for acceptability (able to satisfy WAPS 1.3). If the SME batch is determined to be acceptable it is transferred forward to the Melter Feed Tank (MFT). If it is determined to be unacceptable it will be remediated and held in the SME until it is determined to be acceptable.

5.1 Point of Control

As noted in Section 4.0, the DWPF is not designed to collect and recycle glass which does not meet the specifications. Thus, the glass composition will be controlled through control of the feed composition. In implementing this operating philosophy, the DWPF has chosen to control the feed composition at the SME, for the following reasons:

- If unacceptable feed is detected, the condition can be corrected in the SME. Either frit or trim chemicals can be added to the SME to adjust the feed composition, if needed. Neither the Melter Feed Tank nor the melter has the capability to make such adjustments routinely.
- There is no routine downstream chemical processing which will affect the acceptability of the feed, i.e. no chemical additions can be made to either the MFT (without jumper changes), or the melter.
- Process batches can be evaluated in the SME without disrupting feed to the melter since the MFT feeds the melter continuously.

5.2 Qualification of Waste for DWPF Processing

Before processing of a sludge or precipitate batch begins in the DWPF, SRNL will characterize samples of the sludge and the precipitate, from the Tank Farm (see Figure 6). SRNL will measure the elemental content of the samples and identify the major elements for DWPF analyses and reporting (those present in the glass a >0.5 wt%). SRNL will identify these elements by identifying all elements present in the waste at greater than 1 wt% (equivalent to about 0.28 wt% in the glass for elements in the sludge and 0.08 wt% for elements in the precipitate). While the representativeness of individual Tank Farm samples cannot be demonstrated, SRS's experience with analyses of samples from the Tank Farm indicates that the range of analytical results for samples from a single tank is less than 30% for major elements. Thus, by choosing a conservative value based on Tank Farm sample analyses, the DWPF is assured of reporting all major species. SRNL will also measure the content of dilute radionuclides which must be reported to Civilian Radioactive Waste Management System (CRWMS)[*] but cannot be routinely measured in the DWPF laboratory. SRNL will then determine ratios between the concentrations of these dilute species, and the

[*] The CRWMS is the DOE's Office of Civilian Radioactive Waste Management system for acceptance, transportation, storage, and disposal of spent nuclear fuel and high-level radioactive waste. The CRWMS does not include storage at the DWPF.

concentrations of species which can be measured in the DWPF laboratory. These ratios will be used to report the radionuclide inventory of the glass product (see Section 8.1.2). The elements present and the radionuclide content will be reported to DWPF Engineering by SRNL.

The composition of the waste will also be used by SRNL to recommend the most suitable frit compositions for the DWPF. Using the compositions for the sludge and precipitate, SRNL will determine whether these wastes can be combined with the frits specified for DWPF use to produce a glass which satisfies WAPS 1.3. To establish the blend composition and determine if it will produce an acceptable glass product (able to satisfy WAPS 1.3), SRNL will use the methodology for determining process batch acceptability contained in the Product Composition Control System (PCCS). Processing constraints (viscosity, glass liquidus temperature) and compliance with WAPS 1.3 are taken into account. In general, the processing constraints are more stringent than the glass product specifications (see WQR Volume 5 for a more detailed discussion).

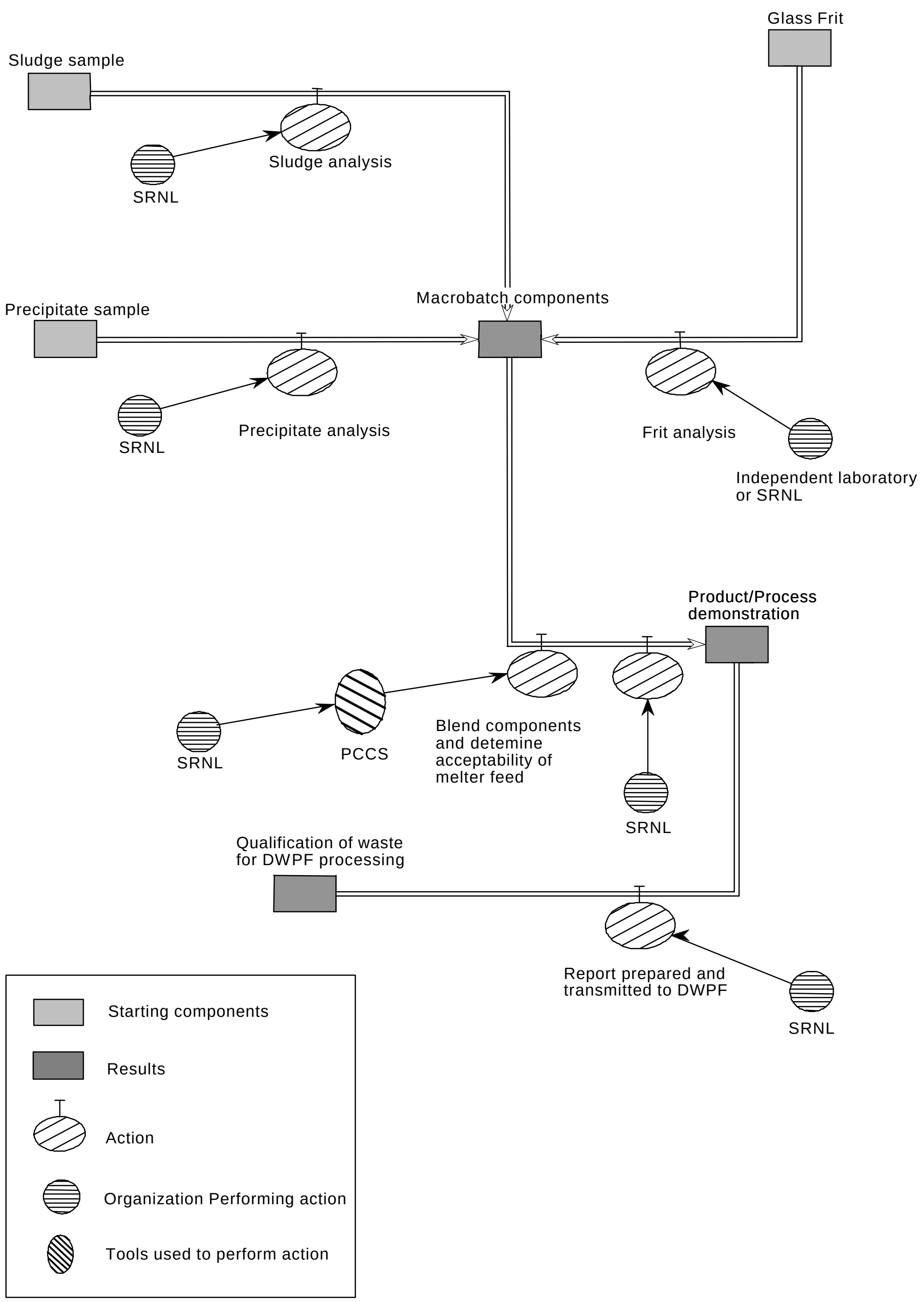
If there is a blend which satisfies the specification, SRNL will mix the samples of the sludge batch and the precipitate batch (i.e., actual waste) from the Tank Farm with frit and vitrify the slurry in the Shielded Cells Facility in the same manner as in the DWPF. The glass produced from the sludge and precipitate samples will then be characterized in the same manner as actual glass samples taken during production (see Section 7). The glass will be analyzed for chemical composition, radionuclide inventory, and subjected to the PCT. Acceptability will be determined in the same manner as for DWPF glass pour stream samples. This will demonstrate that an acceptable glass can be produced from the waste to be processed, and that the product control algorithms contained within the PCCS can be used for the actual feed (product/process demonstration, see Figure 6). This information is then reviewed by SRNL and transmitted to DWPF Engineering.

A variability study on each sludge batch will also be performed by SRNL. This study will establish the applicability of the correlation between PCT results and the chemical composition for that sludge batch. If the correlation does not apply over the entire range of compositions, then the DWPF will constrain the process to operate only within the region over which the correlation does apply or SRNL will develop a new correlation for that sludge batch. If a new correlation were needed, SRNL would use the data generated during the variability study to develop a new correlation.

Occasionally, the DWPF may need to change frit during the processing of a macrobatch for operational reasons (e.g., to improve melt rate). In situations such as this, adjustments to the frit composition will be made. A review will be performed to determine if the change in the frit composition impacts the reportable elements per WAPS 1.1.2 and if the resulting glass composition falls inside the window of the glass variability study. Based on this review a determination will be made as to whether a change in the macrobatch will be made. If there are no impacts on the reportable elements or the glass variability study, a macrobatch change will not be made.

If necessary, an evaluation of the current sampling regimen will also be performed to ensure that that the acceptance criterion of Specification 1.3 can be met for each SME batch. It is likely that the sampling regimen specified for the initial macrobatch will also be adequate for subsequent ones.

Figure 6. Qualification of Waste for DWPF Processing.



5.3 SME Sampling and Analyses

5.3.1 SME Batch Sampling

After all transfers have been made to the Slurry Mix Evaporator (SME) (i.e. frit, SRAT batch) and after evaporation, slurry sample(s) of the SME batch (the sampling regimen is described in Section 5.2) will be taken. The SME must be agitated for 2 hours on high speed before a sample can be taken. When these requirements are met and verified by the Shift Manager, a sample will be taken. Samples from the SME will be taken using a remote sampling system.[33] A recirculating feed loop Figure 7) circulates feed material from the SME to the sampling station in the DWPF sampling cell.

There, a small side stream passes through a recirculating sample loop to the Hydragard® sampler. The slurry must recirculate through the sample loop for at least 60 minutes before a sample is taken to ensure a homogeneous sample.[34] To obtain a sample, the manually activated ram on the sampler is opened. This allows material to flow into the sample vial (Figure 8).

5.3.2 Analyses of SME Samples

The DWPF Laboratory is responsible for the analyses of slurry samples taken from the feed preparation vessels. Slurry samples from the SME will be prepared, and then the chemical composition determined. Standards (see Section 5.3.2.1) will be analyzed at the same time. These standards will allow detection and correction, if necessary, of possible biases in the analytical system. The elements to be analyzed for the design-basis glass (Blend in Table 2) are listed in Table 3. They represent the set of elements which are present in the glass at greater than 0.5 wt% (see Part 3, Item 200 of the WCP). This list is expected to change very little for any of the projected compositions (see Table 2) which may be produced by the DWPF.

The results from analyses of SME samples are entered into the Laboratory Information Management System (LIMS). Before the information is transferred or released from LIMS, a supervisor from the DWPF Laboratory must approve the data. The information is then released by paper copy to DWPF Engineering for entry into PCCS for determination of SME batch acceptability.

5.3.2.1 Standards

Non-radioactive glass standards or other alternate standards are used by the DWPF laboratory to ensure that the laboratory methods are producing adequate results. These standards allow for bias correction, if necessary. The DWPF has on hand several hundred pounds of the Approved Reference Glass which is being used as the current standard. A round robin of several analytical laboratories established accepted values for each element (see WQR Volume 2). Once the initial radioactive feed to the DWPF is fully characterized, a different glass composition to be used as an analytical standard will be recommended by SRNL, if necessary.

Figure 7. DWPF Recirculating Feed Loop.

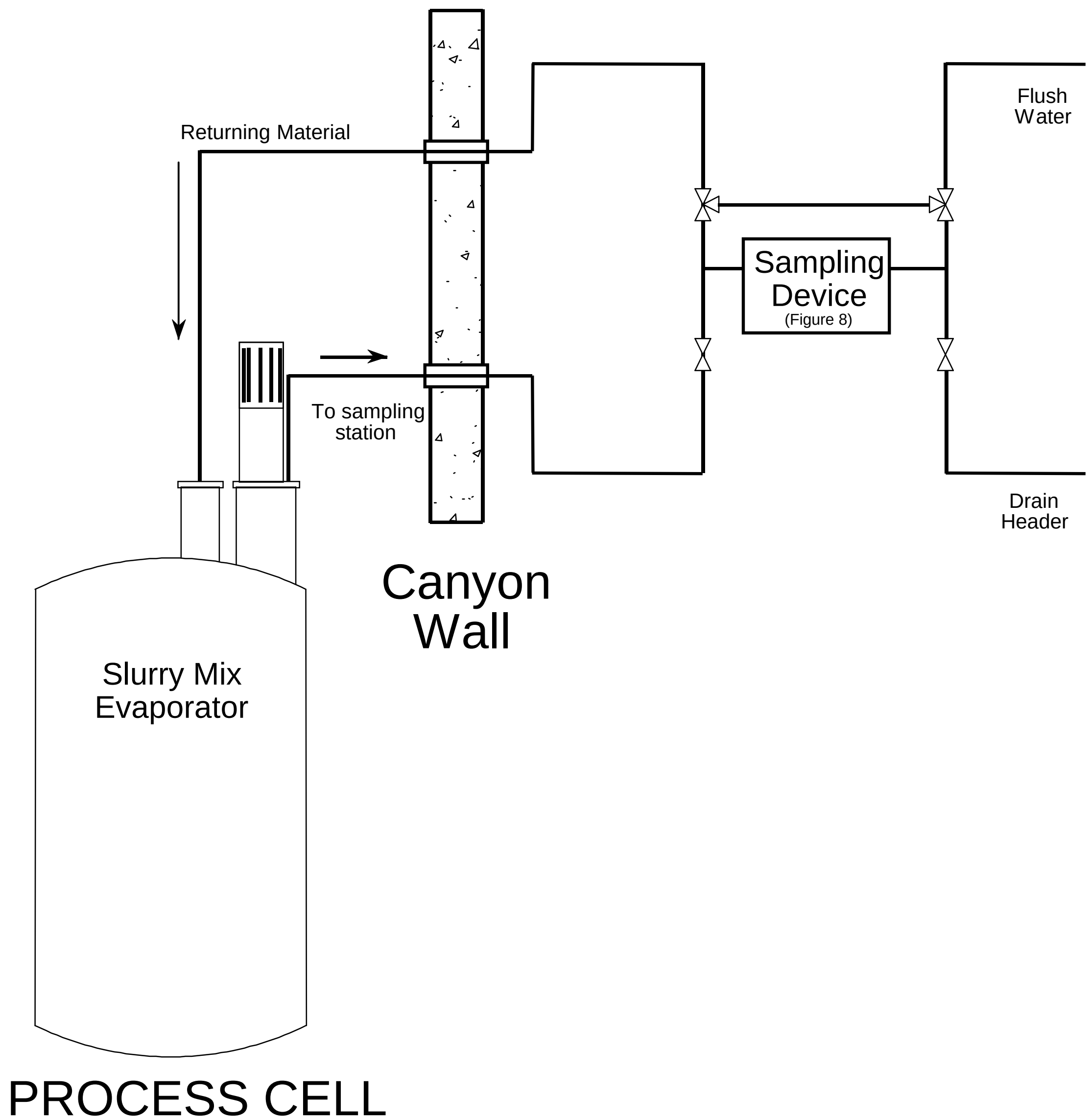


Figure 8. DWPF Remote Liquid/Slurry Sampler.

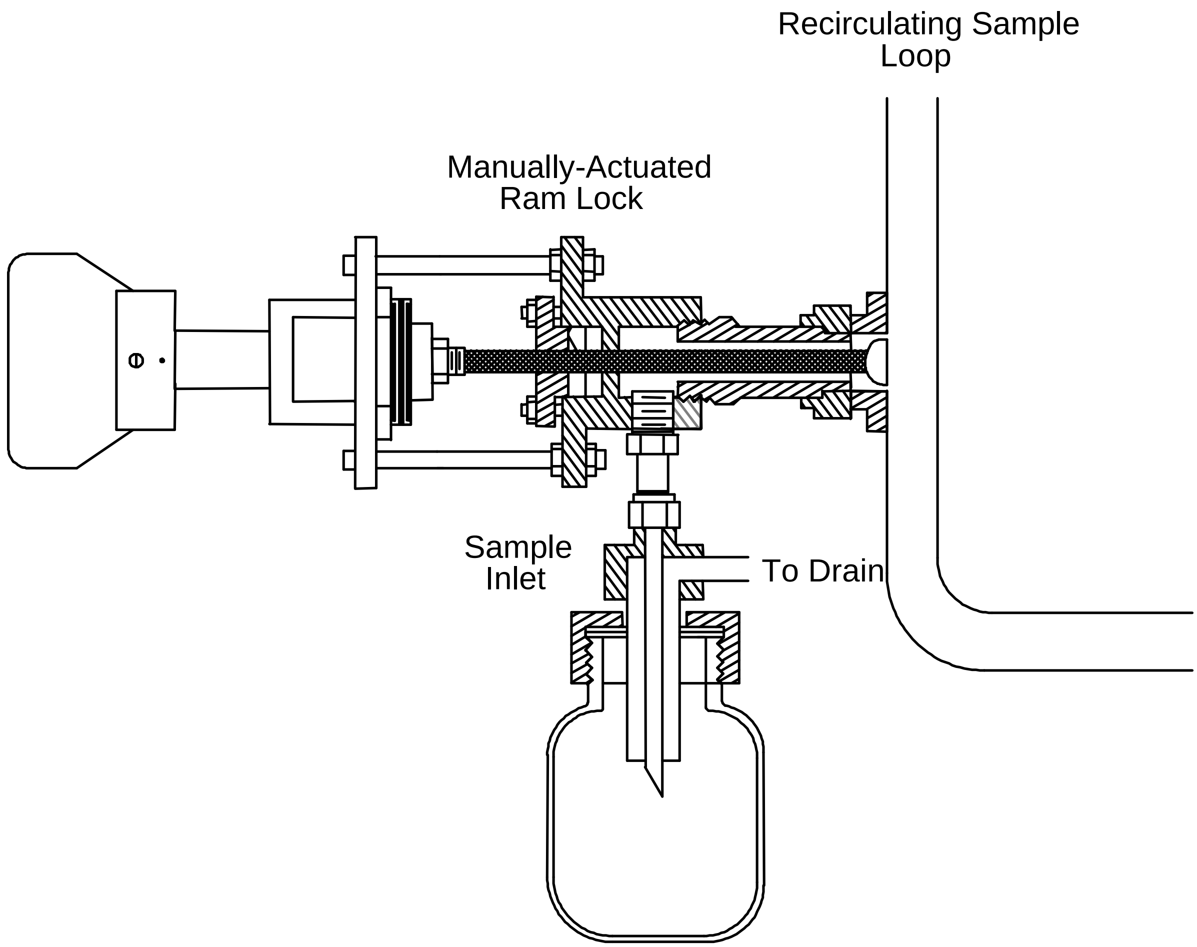


Table 3. Elements to be Determined for Design-Basis DWPF Glass.

Component*	Source
Al	Sludge
B	Frit, PHA
Ca	Sludge
Fe	Sludge
K	PHA
Li	Frit
Mg	Frit, sludge
Mn	Sludge
Na	Frit, PHA, sludge
Ni	Sludge
Si	Frit, sludge
Ti	PHA
U	Sludge

* analyzed by inductively coupled plasma spectroscopy (ICP)

5.4 Evaluation of Feed Acceptability

Each Slurry Mix Evaporator (SME) batch of feed will be held at the SME until it is determined that it will produce acceptable glass (able to satisfy WAPS 1.3). The DWPF Laboratory will determine the chemical composition of the SME samples. This chemical composition will be used to calculate projected PCT results using the PCCS, as described in Section 4.2. The Product Composition Control System (PCCS) will be operated by DWPF Engineering. If the PCCS determines the feed material (SME batch) to be acceptable it will be passed forward to the MFT and then to the melter. If the PCCS determines the feed material to be unacceptable, it will be held in the SME, its composition adjusted, and then re-evaluated using the PCCS. The SME batch will be passed forward to the MFT only if its composition is determined to be acceptable. A flowchart of this evaluation process is shown in Figure 9.

As discussed in Section 5.3.2, the DWPF Laboratory provides the sample analyses for determination of SME batch acceptability (currently using PCCS). DWPF Engineering will manually enter data into PCCS. After the data has been entered into the PCCS, the data will be checked by Engineering or Operations (not the Shift Manager) to ensure the correct information has been entered into PCCS. Once the data is checked, the engineer will direct the PCCS to determine if that SME batch is acceptable. If the SME batch is determined to be acceptable by Engineering, then the Shift Manager will verify the Engineering acceptability evaluation to ensure that the SME batch is acceptable to transfer forward to the MFT. If PCCS determines that the SME batch is not acceptable the material is held in the SME until it is determined to be acceptable (see Section 5.5).

5.5 Feed Adjustment

If a SME batch is determined to be unacceptable, Engineering will determine whether additional samples should be taken and analyzed or if the feed should be remediated. If the projected PCT results of a SME batch appear to be unacceptable but are close to being acceptable, additional sampling and analyses is likely to be the first course of action. The additional analyses will be used in conjunction with the original analyses to determine the acceptability of the feed. If the SME batch is determined to be acceptable by Engineering, then the Shift Manager will verify the Engineering acceptability evaluation to ensure that the SME batch is acceptable to transfer forward to the MFT.

If the results still indicate that the feed is unacceptable after resampling, or if the feed is far from acceptable to begin with, then, in general, addition of frit will be all that is necessary for adjustment. Engineering will determine to amount of frit to be added so that the feed material will be brought back within the target region. This is illustrated in Figure 10 for a case where not all of the frit has been added to the batch in the SME, and an extra portion of PHA material has inadvertently been added to the feed. As shown in the Figure, the DWPF would use the analyses of the SME material and discover that the feed would likely produce unacceptable glass. Addition of frit would bring the composition of the feed back into the acceptable region, and so a new target would be defined. Engineering would calculate the amount of frit to be added to reach the new target and confirm this calculation using PCCS. After the frit was added, the SME would be sampled again, and the material re-evaluated using the PCCS to determine its acceptability (see Sections 5.3 and 5.4). If the SME batch is determined to be acceptable by Engineering, then the Shift Manager will verify the

Figure 9. Evaluation of SME Batch Acceptability.

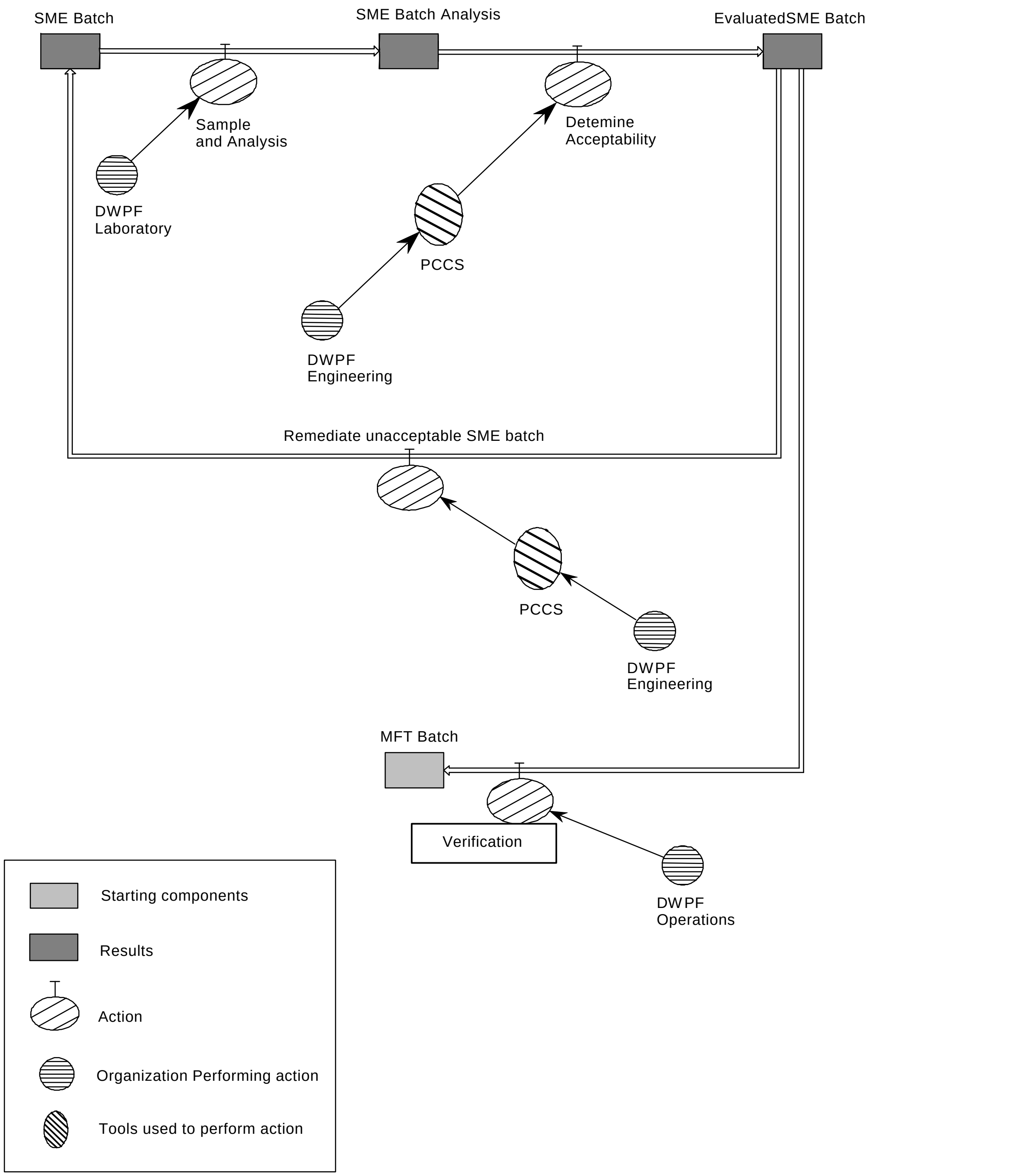
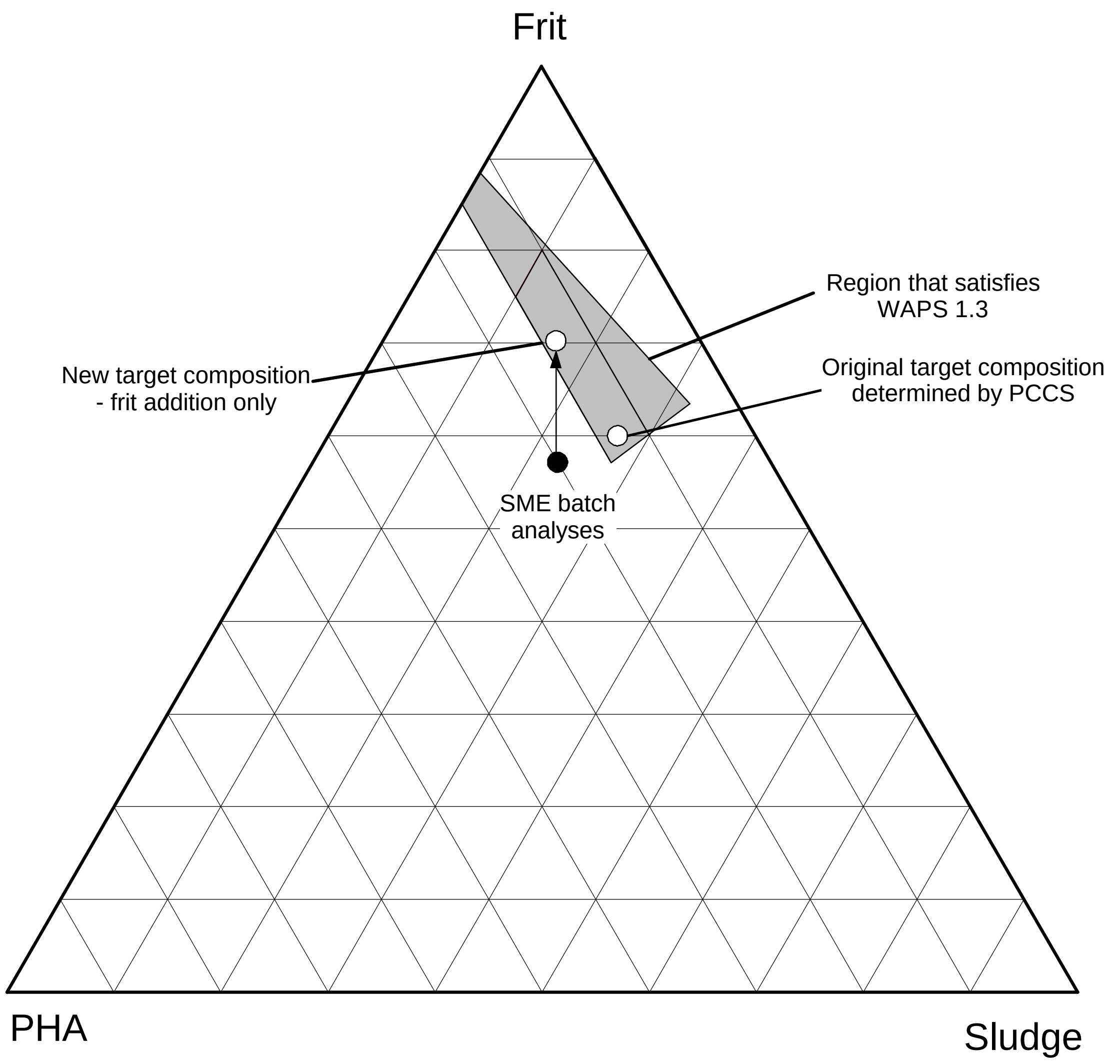


Figure 10. Example of SME batch adjustment through addition of frit.



Engineering acceptability evaluation to ensure that the SME batch is acceptable to transfer forward to the MFT. If the SME batch is still unacceptable, it will have to be remediated again.

If the addition of frit does not bring the SME batch into the acceptable region, trim chemicals can be added. The SME is equipped with feed lines which will allow additional flexibility to add trim chemicals. Engineering will determine the amount of trim chemicals to be added. After the trim chemicals are added, the SME would be sampled again, and the material re-evaluated using the PCCS to determine its acceptability (see Sections 5.3 and 5.4). If the SME batch is determined to be acceptable by Engineering, then the Shift Manager will verify the Engineering acceptability evaluation to ensure that the SME batch is acceptable to transfer forward to the MFT. If the SME batch is still unacceptable, it will have to be remediated again.

5.6 MFT Sampling

The MFT was originally sampled as part of the reporting program at DWPF. A study of analytical results from the SME and MFT for macrobatches 1 and 2 has shown that the data taken from the MFT is essentially redundant to the data from the SME, and as a result adds no further information.[39] DWPF no longer requires sampling of the MFT and the SME sample will provide all necessary input for the Production Records.

5.7 Reporting

The DWPF will provide verification that the canistered waste forms from each macrobatch satisfy WAPS 1.3 using two forms of information. Each will be reported in the production Records (see Section 8):

- Average of predicted PCT results over the macrobatch - During production, the predicted PCT results for each process batch (SME batch) in the macrobatch (from the PCCS) will be recorded. At the completion of macrobatch processing, these predicted PCT results will be averaged and the standard deviation calculated. The standard deviation will address each of the sources of uncertainty in the PCCS prediction as well as process batch-to-process batch variability.

- Predicted PCT result based on the average chemical composition of the macrobatch - PCT results will be projected using the average chemical composition of the macrobatch, determined from SME and MFT batches (see Section 8.1.1 and WQR Volume 2[3]), using the correlation between chemical composition and PCT results.

The two sets of results discussed above will be analyzed and any differences reconciled. These results, including standard deviations, will be compared to the mean PCT results of the EA glass in the Production Records to verify compliance with WAPS 1.3 (see Section 8.2 for a discussion on review and approval of Production Records). As additional evidence that the DWPF glass product complies with the WAPS and as requested by the DOE Office of Civilian Radioactive Waste Management, the PCT protections for each SME batch in the macrobatch will also be provided in the Production Records.

5.8 Confirmation

Confirmatory glass samples will be taken from the glass pour stream during actual DWPF production (see Section 7). As least one glass sample will be taken from each sludge batch to confirm that the glass produced satisfies the WAPS. Chemical and radiochemical analyses will be performed on each sample by SRNL. Each glass sample will also be subjected to the PCT (in conjunction with samples of the EA glass). The PCT results will be recorded at the conclusion of the processing of that macrobatch and reported in the Production Records.

5.9 Frit and Trim Chemicals

5.9.1 Frit

SRS high-level radioactive waste will be mixed with premelted granular glass frit prior to vitrification. The composition of the frit has been specified to provide a durable borosilicate waste glass product able to satisfy WAPS 1.3 which can be processed in the DWPF melter. Since the glass frit comprises the major portion of the glass product by weight, control of the frit composition is important to control of the composition of the waste glass product. As discussed in Section 5.5, addition of frit will probably be the first course of action for remediating SME batches which would otherwise produce unacceptable product.

Tightly controlled procurement specifications are implemented to assure control of the frit composition. These specifications establish upper and lower limits of composition variability for the major components, and maximum levels for the minor components (i.e., frit impurities). These limits are based on studies which are discussed in WQR Volume 5[2]. The minor components are introduced by impurities in the frit raw materials and from dissolution of the furnace refractories during manufacture of the frit. An example of impurity limits is contained in the specification for Frit 200[35] (Tables 4 and 5).

The DWPF frit specifications describe the product requirements, and the controls which must be applied during manufacture, evaluation and storage of the frit prior to shipment to DWPF. Rework of the frit by the vendor that is not specifically allowed by the specification must be approved by DWPF. Weather protection shall be provided during storage and shipment.

A representative composite sample will be obtained by continuous sampling during loading of the frit and is shipped to an independent certified laboratory for product conformance testing. The results of this testing are included in a “Certificate of Analysis.” The frit is then shipped to the DWPF after it has been determined that the material meets the specifications as measured by an independent laboratory.

5.9.2 Trim Chemicals

Feed samples will be drawn from the SME and analyzed to determine if acceptable glass will result from processing. If feed adjustment becomes necessary, addition of frit will most likely be the first course of action. If it becomes necessary to add other trim chemicals to the SME, the amount of chemical additions required will be minimized to retain maximum waste loading (see Section 5.5).

Sodium hydroxide, potassium nitrate, and boric acid are the materials that may be used for feed adjustment. Trim chemicals will be added only as necessary. The trim chemicals are commercial and/or technical grade chemicals and are received on the basis of a Certification of Analysis which is traceable.

Table 4. Chemical Composition of Frit 200.

Major Oxide Components	Amount (weight %)
SiO_2	70.0
Na_2O	11.0
B_2O_3	12.0
Li_2O	5.0
MgO	2.0

Table 5. Maximum Allowable Levels of Frit Impurities.

Elemental Components	Maximum (weight %)
Al	1.0
Fe	0.2
Mn	0.2
Ni	0.2
Cr	0.1
Pb	0.1
Ti	0.15
F	0.05
Cl	0.05
Moisture	0.25
Total Impurities	2.25

6. FEED AND GLASS HOMOGENEITY

Once an acceptable feed material (based on its composition) has been prepared in the SME, the DWPF will ensure that it remains acceptable by preventing segregation of the feed as it passes from the SME to the MFT, and from the MFT to the melter. The uniformity of the melter feed material must be maintained so as not to invalidate the composition control.

6.1 Feed Homogeneity

Successful application of the methodology for determining SME acceptability requires that the relationship between samples taken from a process batch and the process batch itself is known, and that steps are taken to ensure that this relationship is maintained. Both the SME and the MFT have been designed to ensure that no segregation of material will occur which will affect glass product quality. The agitator in each of these tanks is designed to thoroughly homogenize the contents of the vessels. Based on experiments performed by SRNL with full-scale prototypic vessels, the degree of nonhomogeneity in these vessels will be negligible. (These results are reported in Volume 2 of the WQR[3]). The design of the vessels and the agitators are controlled as part of the DWPF modification control program (see Section 2.4). Minimum agitation times are also specified in DWPF operating procedures/manuals as discussed in Section 5.

Occasionally, a process batch which has been determined to be acceptable in the SME will be held in the SME or MFT due to upsets downstream (e.g., cleaning the melter off-gas line). Both the SME and MFT have lines which allow addition of acid to prevent irreversible segregation (settling such that the material cannot be rehomogenized) of feed material. However, if formic or nitric acid is the only material added to the process batch after acceptability has been determined, resampling and analyses for glass product acceptability is not required.

6.2 Glass Homogeneity

The role of the DWPF melter is to ensure that feed material is sufficiently vitrified to produce an acceptable product before the glass is poured into the canister. It has been shown that the melter feed needs only to be heated to 850°C to produce an acceptable glass.[15,36] The DWPF will control pouring from the pour spout and the drain valve so that the melt temperature will be > 950°C while pouring. This assures that the melter feed has been converted to glass. The DWPF operating limit contained in the operating manual states that the melt pool temperature must not drop below 1050°C for more than one hour.[16]

7. PRODUCTION GLASS SAMPLES

7.1 Glass Sampling

The DWPF ensures that an acceptable glass has been produced by a series of administrative and process controls relating to the melter feed material in the SME. However, glass samples are also taken to provide additional direct confirmation that acceptable glass has been made. As discussed in Part 3, Item 500, of the WCP, the glass will be sampled at least once during processing of each sludge batch.

A glass sampler suitable for DWPF use has been developed and tested. Figure 11 shows the glass sampler that will be used during DWPF operations. The DWPF sampler is a modified canister throat protector, equipped with a small retractable flat cup which is inserted directly into the stream of molten glass, as it pours into the canister. Approximately 40 g of glass will be taken from the flowing stream of molten glass as it is poured from the melter into the canister. Glass sampling does not interrupt normal operations.

The glass sampler is placed on the neck of the canister before it is rotated under the melter pour spout. During the pour, the sample cup is pushed into the glass pour stream for a couple of seconds and then retracted into the throat protector. The canister number is recorded for each glass sample taken. The sample cannot be retrieved until the pouring operation is complete and the canister has been rotated from beneath the melter. The modified throat protector is lifted from the canister, set aside, and allowed to cool for one hour. The sample is then removed from the modified throat protector, and placed into a glass sample container. The container is then loaded into a secondary container and a shipping drum and transferred to SRNL, where the glass samples are characterized, and then those portions not consumed in characterization are archived by SRNL for the DWPF.

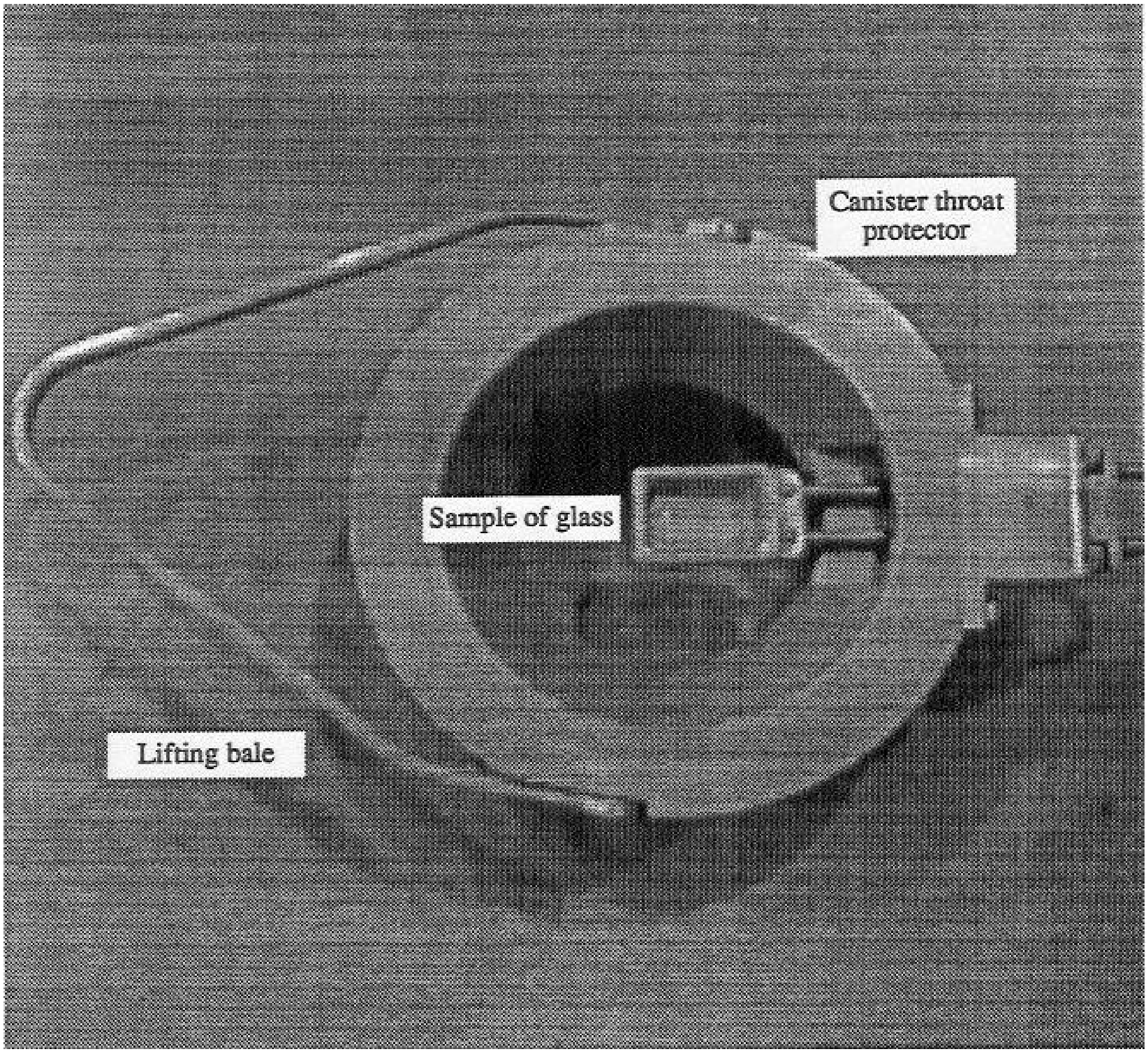
7.2 Glass Characterization

All glass samples received at SRNL are characterized by performing chemical analyses, performing analyses for radionuclides, and by leach testing using the PCT (see Section 7.3.2). The analytical methods and instrumental techniques used by SRNL to analyze the glass will be similar to those used by DWPF to analyze feed slurries.

Personnel performing the PCT are trained and their proficiency is evaluated and documented, before they are considered qualified to perform any operations involving glass samples. All SRNL results will be collected and reported to DWPF. In the unlikely event that the PCT yields unacceptable results, for a particular macrobatch of material, that group of canisters will be flagged as potential non-conforming items and the procedure of Part 6, Item 300, of the WCP followed.

Based on the results of Waste Qualification Runs (see WQR Volume 5[2]), the PCT results and chemical composition results which are to be reported are sufficiently accurate for expected DWPF glass compositions to judge compliance with the product consistency specification without glass sampling. However, the DWPF is committed to take at least one glass sample from each sludge batch. In addition, during initial radioactive operations DWPF will take more glass samples to confirm the conclusions from Waste Qualification Runs and in order to judge the accuracy of the method for reporting radionuclide inventory.

Figure 11. DWPF glass sampler.



If discrepancies between glass sample analyses and the PCT results predicted from the melter feed composition are found, which are larger than those which can be attributed to expected errors (see WQR Volume 5), the results will be evaluated and the glass may be sampled more frequently. However, as long as the PCT results are acceptable (comply with WAPS 1.3) that group of canisters (macrobatch) is acceptable.

7.2.1 Administrative Controls for Glass Sample Analyses

The same elements analyzed for in the SME will be determined for the glass samples. All samples will be analyzed with the highest level of quality assurance, using an analytical study plan approved by all affected organizations. All glass samples will be identified in such a way that the identity can traced back to the canister from which the glass sample was taken. The data from glass characterization will be collected, evaluated, and transmitted to DWPF for inclusion in the Production Records (see Section 8).

7.2.2 The Product Consistency Test (PCT)[37]

To perform the PCT, samples of the glass to be tested are crushed, washed, and sieved. For the PCT, -100,+200 mesh glass that has been carefully sieved is used. The sieved sample is washed with water and alcohol to remove fines and then dried at 90°C. The washed glass is leached in ASTM Type I water[*] (1.5 grams glass/15 mL of water) at 90°C for 7 days. Stainless steel containers are used for radioactive production glass samples. The PCT is performed in triplicate for each test glass; as well as for a previously characterized glass standard (EA glass). The glass standard enables test results to be quantitatively compared to results from earlier tests, and eliminates certain types of bias. Duplicate blank tests are always performed, as well.

At the conclusion of the test, the leachate is filtered through a 45 micron filter to ensure that it has been separated from the glass particles. The solution pH is then measured, and the filtrate is acidified and analyzed. The concentrations of the elements released to solution are then measured. The results are then normalized to take into account the amount of the element in the glass sample. The WAPS requires that the normalized concentrations of lithium, sodium, and boron be compared to that of the EA glass. This information will be reported in the Production Records (see Section 8). This test is described in more detail in WQR Volume 5[2].

[*] ASTM Type I water; Purified water with a maximum total matter content of 0.1 mg/L, electrical conductivity of 0.06 μmhos/cm at 25°C, a minimum electrical resistivity of 16.67 M ·cm at 25°C and no detectable soluble Si.

8. PRODUCTION RECORDS

The Waste Acceptance Product Specifications[1] (WAPS) require that the DWPF provide evidence of compliance with the WAPS during production. This evidence is to be documented, and sent to the Civilian Radioactive Waste Management System (CRWMS), in the form of Production Records for DWPF canistered waste forms. The Quality Assurance (QA) organization will develop the Production Records from the procedures, data sheets, etc. that are collected in canister and batch "wallets". DWPF Engineering and Quality Assurance will review and concur with the Production Records. The DWPF Facility Manager will then approve the Production Records. The content of the Production Records relevant to the glass product specifications (see Section 1.2) is presented below (the content of the Production Records relating to other portions of the WAPS can be found in the WCP).

Quality Assurance will transmit the original Production Records to Records Management for maintenance as lifetime records until the canistered waste forms are shipped to the repository. At such time, the original Production Records will be transmitted to CRWMS.

8.1 Content

The Production Records summarize the detailed records of DWPF canistered waste form production. The Production Records provide references to the detailed records (primarily through canister and batch identifications) so that more information can be retrieved, if necessary. The information contained within the Production Records is compiled from operating manuals/procedures, canister supplier information, analytical laboratory results, and PCCS printouts, which are collected in canister and batch wallets during processing of the macrobatch. The "macrobatch" is a key concept for compliance with WAPS 1.1, Chemical Composition; WAPS 1.2, Radionuclide Inventory; WAPS 1.3, Product Consistency; WAPS 1.6, IAEA Safeguards Reporting; and WAPS 3.14, Concentration of Plutonium. The definition of a macrobatch is discussed in Section 1.4. These periods of constant feed constitute macrobatches, which the DWPF will treat as the "waste types" referred to in the specifications. The exact number of batches and canisters is dependent on waste processing schedules and DWPF production rates. For each canister produced from a given macrobatch, the chemical composition, the radionuclide inventory, and the projected PCT results are reported as the averages of each over the entire macrobatch (standard deviations are also reported). Thus, the reported values will be the same for all canisters produced from a given macrobatch. If additional information is needed, the canister number can be used to retrieve more detailed information about a particular macrobatch, or process batch, of feed.

The Production Records will identify particular canisters by the code on the label affixed to them. This code, unique to each canister, will be the key to tracing the records for each canistered waste form. All of the records which support the information reported in the Production Records will be keyed to that code.

8.1.1 Chemical Composition During Production

The WAPS require that the DWPF report the content of all elements, excluding oxygen, which are present in concentrations greater than 0.5 wt% of the glass, expressed as oxides. For each process SME batch, the content (in wt%) of the oxide of each major element in that batch is calculated from the elemental analyses of the feed. These results are then accumulated for the entire macrobatch and averaged, to provide the values reported in the Production Records (see Figure 12).

The Production Records for the macrobatch will contain:

- The canister numbers and the dates the canisters were filled.
- The macrobatch number.
- The process batch numbers.
- Each major (present at greater than 0.5 wt %) element, expressed as its oxide, the content of the oxide, and the calculated standard deviation for the analysis (see WQR Volume 2 for a more detailed description).
- The results of determining the chemical composition of actual production samples of glass (see Section 8.1.4).

8.1.2 Radionuclide Inventory During Production and International Atomic Energy Agency (IAEA) Safeguards Reporting

The WAPS require that the DWPF report estimates of the inventory of all radionuclides with half-lives greater than 10 years, and present in the glass a greater than 0.05 % of the total radionuclide inventory at any point up to 1100 years after productions. Additionally, the WAPS require that the DWPF report the uranium and plutonium content of each canister and verify that the plutonium concentration is less than 2,500 grams/cubic meter. This information will be calculated from radiochemical/chemical analyses of samples from each process batch.

The DWPF will report, as the radionuclide inventory of the glass, the contents of all reportable radionuclides (see Table 6 for the list for macrobatch 1).[38] The particular radionuclides to be reported for each macrobatch will be determined during the qualification of waste activity (see Section 5.2). Radionuclides are reported based on analyses of SME samples and on analyses of Tank Farm samples (for each macrobatch) (see Figure 13). Volume 4 of the WQR contains detailed information on the bases for reporting.

Figure 12. Chemical Composition Reporting.

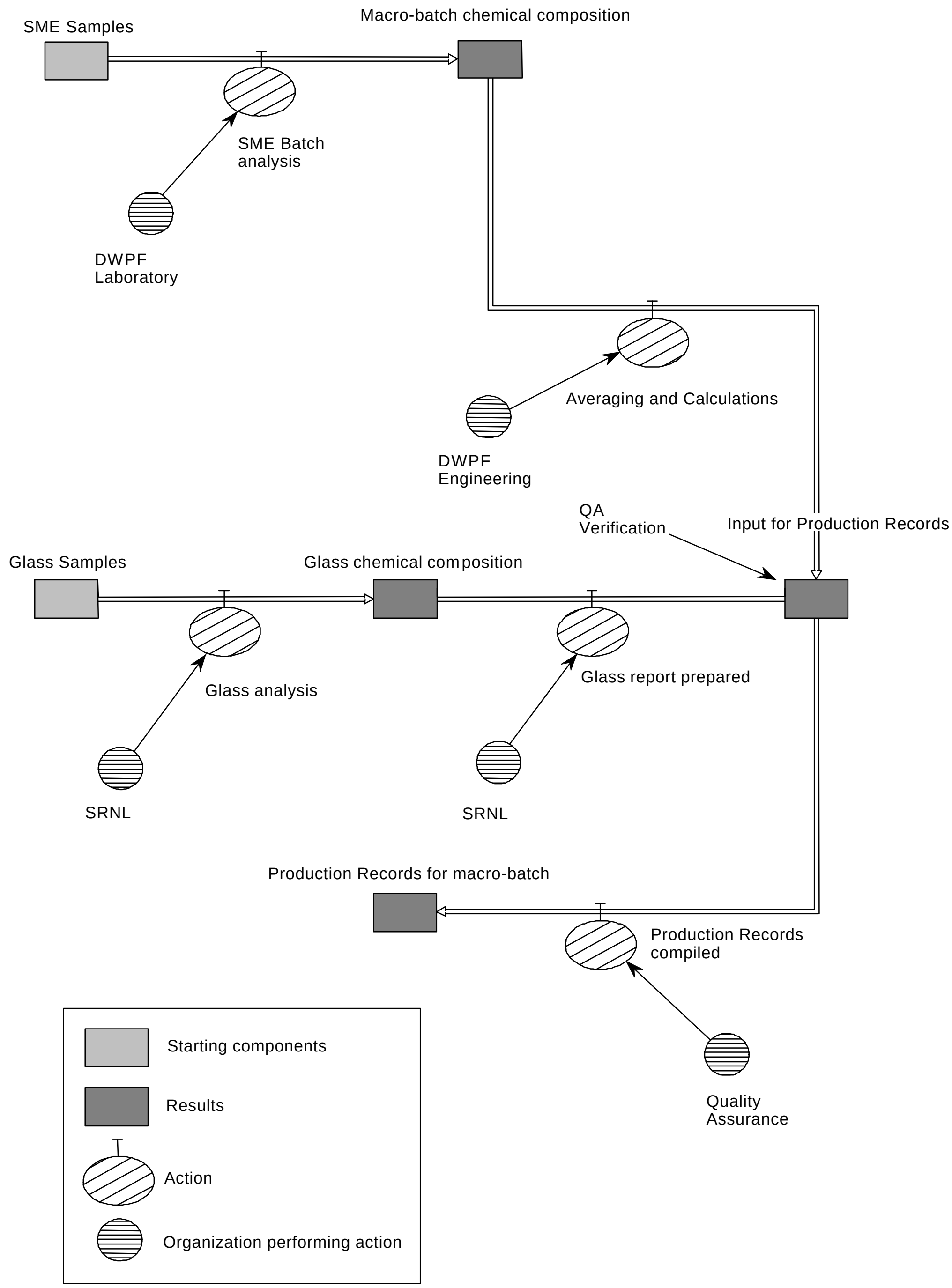
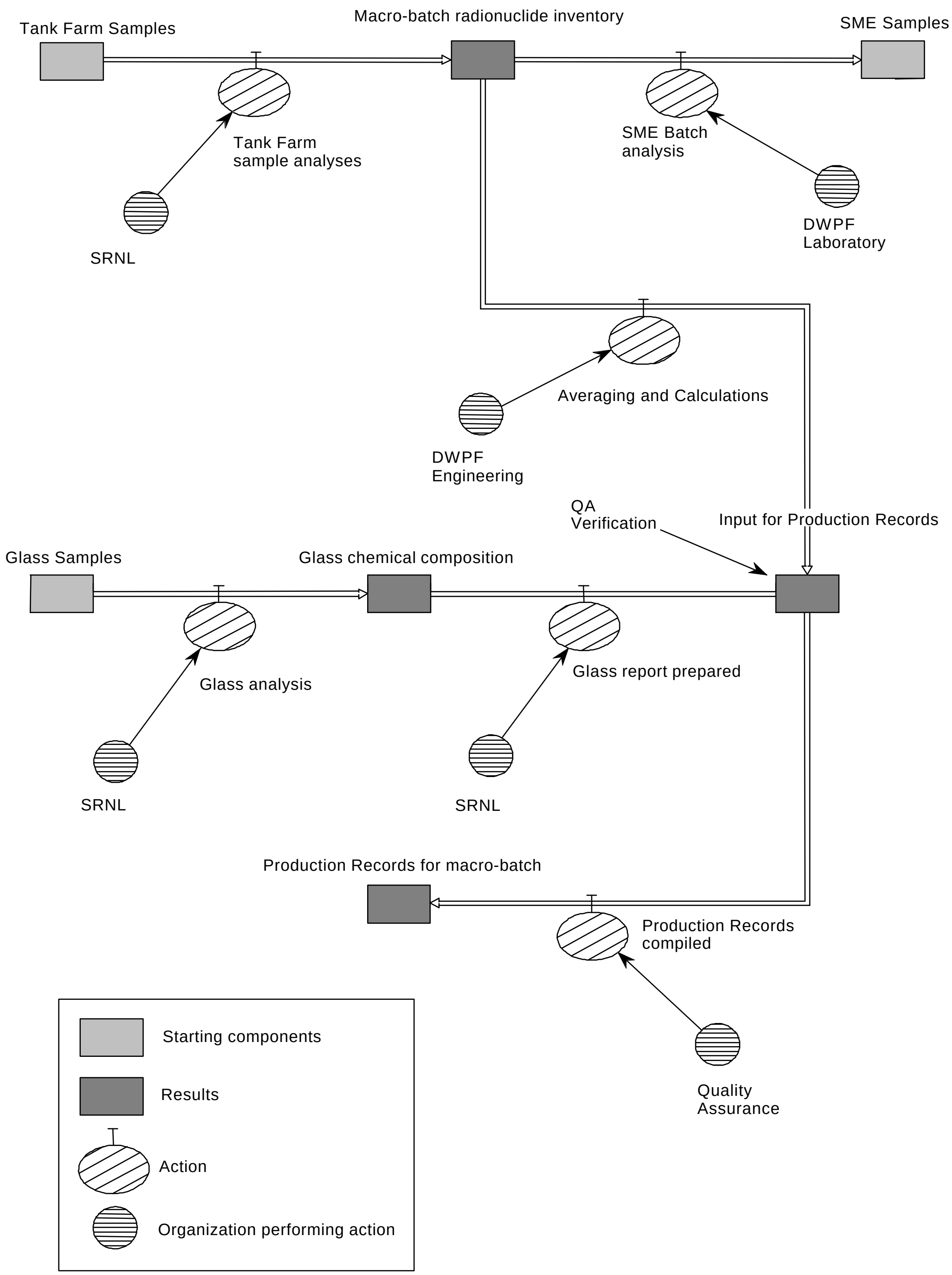


Table 6. Set of Radionuclides to be Reported in the Production Records for Macrobatch 1.

RADIONUCLIDES
Ni-59
Ni-63
Se-79
Sr-90
Zr-93/Nb-93m
Tc-99
Sn-126
Cs-137
Sm-151
Th-229
U-233
U-234
U-235
U-236
U-238
Np-237
Pu-238
Pu-239
Pu-240
Pu-241
Pu-242
Am-241
Am-243
Cm-244
Cm-245
Cm-246

Figure 13. Radionuclide Inventory and IAEA Safeguards Reporting.



The Production Records for the macrobatch will contain:

- The canister numbers and the dates the canisters were filled.
- The macrobatch number.
- The process batch numbers.
- Each radionuclide, and its calculated content.
- The total uranium and plutonium content per canister and the mass ratios of the required uranium and plutonium isotopes.
- The total plutonium content per canister expressed as grams/m^3 and comparison with the IAEA limit of 2,500 grams/m^3.
- The results of determining the radionuclide content of actual production samples of glass (see Section 8.1.4).

8.1.3 Product Consistency

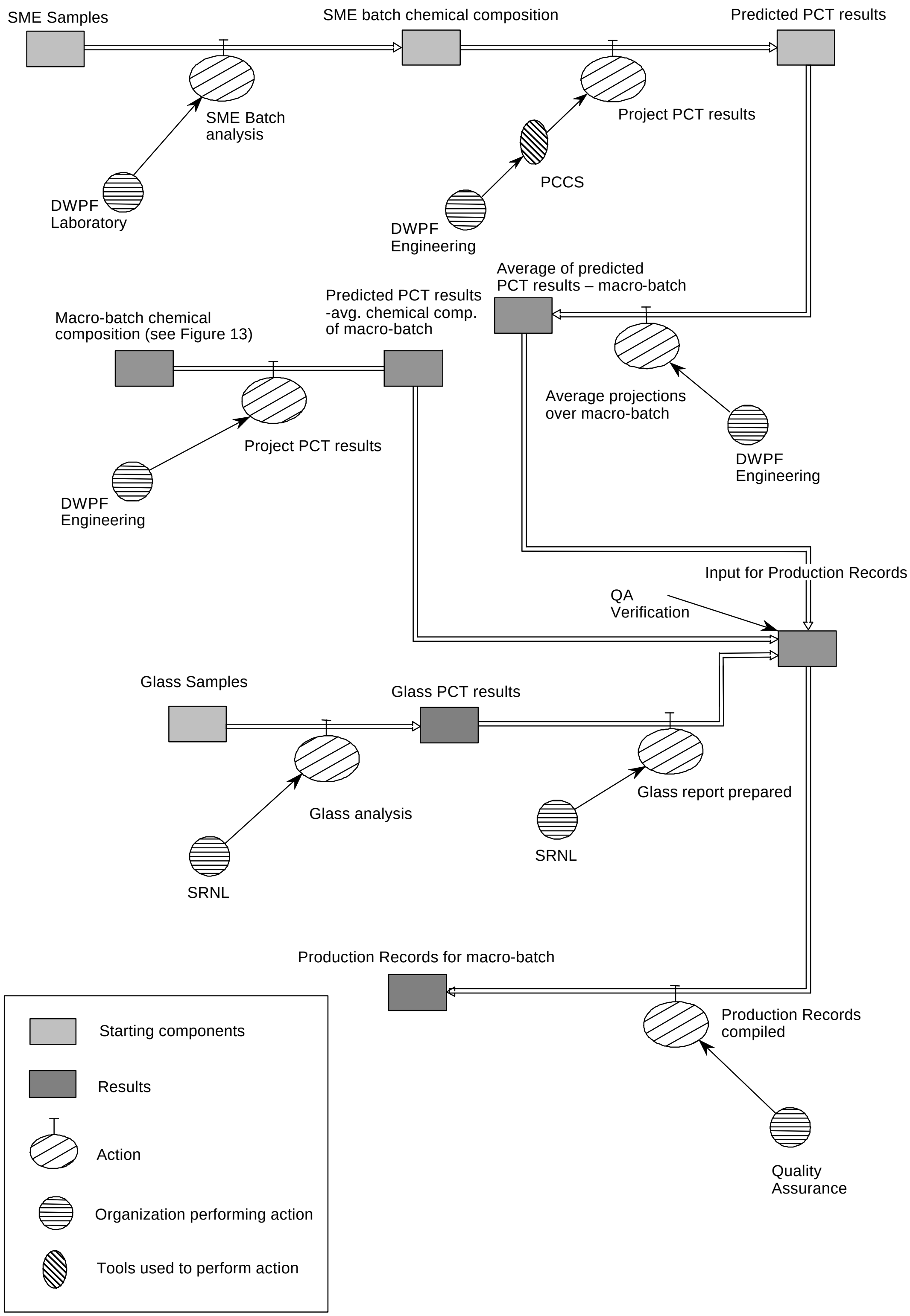
The DWPF will predict the normalized lithium, sodium and boron concentrations of the PCT for each process batch. These values will be average and reported as the average concentrations of lithium, sodium, and boron for each macrobatch. Thus, the predicted PCT results will be the same for every canistered waste form in the macrobatch. The DWPF will also report the PCT results predicted from the average macrobatch composition using the PCT/chemical composition correlation. If these two sets of results are outside the range expected due to compositional uncertainties the results will be reconciled. The DWPF will also certify in the appropriate canister/batch wallet that these values are at least two standard deviations below the mean of the PCT results for the benchmark glass of specification 1.3. In addition, the results of performing the PCT on production samples of glass will also be reported. See Figure 14 for a summary.

If the predicted PCT results or the actual PCT results do not satisfy the specification, the group of glass-filled canisters will be identified as nonconforming items, to be dispositioned as outlined in Part 6, Item 300 of the WCP.

The Production Records for the macrobatch will contain:

- The canister numbers and the dates the canisters were filled.
- The macrobatch number.
- The process batch (i.e., SME) numbers.
- The normalized concentrations of lithium, sodium and boron, (PCT results) predicted using the PCT/chemical composition correlation from the average composition of the macrobatch.
- The averages and standard deviations of the normalized concentrations of lithium, sodium and boron, (PCT results) predicted (using the PCT/chemical composition correlation) from all of the process batches in the macrobatch.

Figure 14. PCT Release Reporting.



- The results of performing the PCT on actual production samples of glass (see Section 8.1.4). The reported results will include the EA glass results and the standard deviation of the glass sample results (see WQR Volume 5).

Also, as requested by the DOE Office of Civilian Radioactive Waste Management, the predicted PCT results determined using the PCT/chemical composition correlation for each SME batch will be provided in the Production Records.

8.1.4 Glass Samples

SRNL reports the results of glass sample characterization (see Section 7.2) to DWPF in a report keyed to the number of the canister being filled during sampling. Copies of this glass sample report will be included in the appropriate canister wallet and a macrobatch wallet. The report, as well as a DWPF Engineering acceptance memorandum, will be attached to the Production Record for the particular canister from which the sample was obtained.

8.2 Review and Approval

Input to the Production Records will come from several areas of the process such as those shown in Figures 12-14. In addition, input to the Production Records will come from other areas of the process to satisfy specifications not discussed in this document (see WCP). Most of this information will be generated from operating manuals/procedures, canister supplier information, and PCCS. QA will verify this supporting information which will be collected in canister and batch wallets. DWPF Operations will turn over the procedures, etc. to QA for insertion into the wallet. Analysis of production glass samples, from SRNL, will also be placed in the appropriate canister wallet. QA will develop the Production Records from the canister and batch wallets and will attach the glass pour stream sample results. The Production Records will be reviewed and discrepancies reconciled before the records are provided to DOE. DWPF Engineering and Quality Assurance will review and concur with the Production Records. The DWPF Facility Manager will then approve the Production Records.

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