

Westinghouse Savannah River Company

Final Report for Crucible Scale Vitrification of Pretreated C-106 Sludge Mixed with Secondary Wastes

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Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808



SAVANNAH RIVER SITE

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Westinghouse Savannah River Company

**Final Report for Crucible Scale Vitrification of Pretreated
C-106 Sludge Mixed with Secondary Wastes**

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Crucible Scale Vitrification of
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Terms and Abbreviations

AA	Atomic Absorption
AAS	Atomic Absorption Spectroscopy
AES	Atomic emission spectroscopy
ADS	Analytic Development Section
ARG-1 (glass)	Analytic Reference Glass-1 (used as analytical standard)
ARM (glass)	Analytical Reference Material Glass (used as PCT testing protocol reference)
ASTM	American Society for Testing and Materials
cc	cubic centimeter
CUA	Catholic University of America
DOE	U.S. Department of Energy
dpm	disintegrations per minute
DWPF	Defense Waste Processing Facility
EA (glass)	Environmental Assessment Glass (used as PCT benchmark durability standard)
Ele.Wt%	element weight percent
g	gram
HLW	high level waste
IC	ion chromatography
ISE	ion selective electrode
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectroscopy
ITS	Immobilization Technology Section
IHLW	immobilized high-level waste
ILAW	immobilized low activity waste
L	liter
LAW	low activity waste
LIMS	laboratory information management system
MALLD	maximum allowable lower limit of detection
M&TE	Measuring and Test Equipment
μCi	micro Curie
mg	milligram
mL	milliliter
MDA	minimum detectable activity
NIST	National Institute of Standards and Technology
NM	not measured
ORP	Department of Energy-Office of River Protection
PCT	Product Consistency Test
PNNL	Pacific Northwest National Laboratory
QA	Quality Assurance
RCRA	Resource Conservation and Recovery Act
RPP-WTP	River Protection Project-Waste Treatment Plant
SEM	Scanning Electron Microscopy

SEM-EDAX	Scanning Electron Microscopy-Electron Dispersive Analysis by X-ray
SRTC	Savannah River Technology Center
TCLP	Toxicity Characteristic Leaching Procedure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
UTS	Universal Treatment Standards
Vac	vacuum
Vol%	volume percent
VSL	Vitreous State Laboratory
WAPS	Waste Acceptance Product Specifications
Wt	weight
Wt%	weight percent
WSRC	Westinghouse Savannah River Company
XRD	x-ray diffraction

I. Executive Summary

The U.S. DOE has established the Office of River Protection (ORP) in Richland, Washington to manage and oversee the design, construction and commissioning of a new Waste Treatment and Immobilization Plant (WTP) that will treat and immobilize the waste for ultimate disposal. To accomplish the ORP mission, DOE established the River Protection Project (RPP). The RPP Contractor is responsible for designing, constructing, commissioning, and supporting the transition of the WTP. The River Protection Project-Waste Treatment Plant (RPP-WTP) team is responsible for producing an immobilized (vitrified) high-level waste (IHLW) waste form. The Savannah River Technology Center (SRTC), has been contracted to produce and test a vitrified IHLW waste form, from Envelope D high-level waste (HLW) samples previously supplied to the RPP-WTP project by DOE. The primary objective of this task was the vitrification of actual waste in order to produce glass samples for subsequent product testing. Testing demonstrated the glass waste form satisfied the product requirements concerning: chemical composition, radiochemical composition, crystalline and non-crystalline phase determination, the Product Consistency Test (PCT), and the Toxicity Characteristic Leaching Procedure (TCLP).

SRTC produced and characterized the IHLW glass waste form from the HLW fractions derived from pretreated LAW cesium eluates (Tanks AN-103, AZ-102, and AN-102) and HLW filtered sludge powder (Tank C-106). The C-106 HLW sludge had been previously caustic leached at PNNL before vitrification. A simulated Sr-TRU precipitate stream was prepared along with a blend of glass formers recommended by Vitreous State Laboratory at Catholic University of America (VSL-CUA). These waste feeds (washed C-106 sludge, Cs-eluates and surrogate Sr/TRU precipitate) were combined with the blend of glass formers in a 650-mL platinum/gold crucible and vitrified inside a sealed quartz vessel. Vitrification was carried out in the shielded cells inside a high temperature electric furnace at 1150°C for four hours. The waste glass was cooled following the projected canister, centerline-cooling curve. The resulting glass was removed from the crucible, crushed to a granular form, and used to determine the glass chemical and radiochemical composition, the Product Consistency Test (PCT), and the Toxicity Characteristic Leaching Procedure (TCLP). A very small milligram-quantity sample of the glass powder was removed from the shielded cells for examination by scanning electron microscopy (SEM) and x-ray diffraction (XRD).

The D Envelope glass samples were dissolved by two independent methods, in triplicate along with two samples of the Analytical Reference Glass (ARG-1) standard and one blank. The dissolutions were either sodium peroxide (Na_2O_2) fusion with hydrochloric acid uptake, or heated digestion with a mixture of acids. Compositions of the ARG-1 glass standard, which is used to assess the accuracy of the glass dissolution and subsequent dissolved glass chemical content analyses, to the target compositions indicate agreement to within experimental analytical measurement precision, indicating that the glass dissolutions and analyses were acceptable. The final chemical composition of the radioactive waste glass was also within experimental analytical measurement precision to the predicted composition supplied by VSL-CUA. Comparison of the measured glass chemical composition to contract specifications indicated that the glass contained the minimum 12.5 wt% Fe_2O_3 based on the average and standard deviation measured elemental iron in the replicate dissolved glass solutions.

Radionuclide analyses performed on the C-106 Envelope D glass confirm that Cs-137 and Sr-90 were the major radionuclides present at levels near their targeted values in the glass. Comparison of the present crucible-scale Envelope D glass, prepared with C-106 radioactive sludge, a simulated Sr/TRU stream and concentrated Cs-137 radioactive eluates, to other previous HLW crucible-scale glasses made from flow-sheet quantities of Hanford radioactive waste streams showed that the present C-106 Envelope D glass

contains an order of magnitude higher Cs-137 in the glass. The higher Cs-137 loadings derive from the present study glass formulation that incorporated all of the Cs-137 eluate streams available from pretreatment of the Hanford Site Waste Tanks AN-102, AN-103 and AZ-102 obtained during past Hanford privatization pretreatment studies (formerly referred to as Part B1) at SRTC. Calculations also show that this glass would meet contract specifications on maximum heat loading (1,500 Watts/canister) within a HLW glass canister.

Durability tests using the Product Consistency Test indicate that the Envelope D glass has average normalized release values that were more than two standard deviations lower than the average normalized release rates for the Environmental Assessment (EA) glass which is the benchmark standard performance glass for PCT comparison. This criteria for comparison of normalized release rate for HLW glass to EA glass is suggested by the Waste Acceptance Product Specifications (WAPS) which are referenced in the present modification/version of the WTP Contract Specification. Thus the C-106 HLW glass meets the Contract Specifications. It should be noted that there is no specified numerical value for upper limit normalized PCT release for the HLW glass specification in the present modification/version WTP Contract Specifications at this time.

Because the Tank C-106 radioactive waste is a “listed” Mixed Waste, the resulting immobilized High-Level Waste (IHLW) must be considered to be listed Mixed Waste. Further efforts to dispose of the IHLW will undoubtedly require that the waste form be shown to retain the hazardous components and not release them to the environment. Therefore, in addition to analyzing the glass waste form directly, the waste form was characterized by a TCLP test. The TCLP was performed according to the standard EPA SW-846 procedure (Method 1311) with certain modifications that are described in this report. Analysis of extract from the Toxicity Characteristic Leaching Procedure (TCLP) tests of this Tank C-106 glass waste form showed the concentrations of Resource Conservation and Recovery Act (RCRA) metals were less than the Universal Treatment Standard (UTS) limits. Digestion of the TCLP extract prevented detection of Thallium to the UTS limit, but direct analysis of the extract showed that Tl was below the UTS limit. All target analyses (including RCRA metals) were determined to the maximum allowable lower limit of detection (MALLD) given in the task specification¹ for this study. X-ray Diffraction and Scanning Electron Microscopy/Energy Dispersive X-ray (SEM-EDX) Analyses were performed on Envelope D glass powders to show that a small amount of the spinel, trevorite (NiFe_2O_4) was present. Measurements of the surface area of crystalline spots on a representative glass piece show the amount of crystalline material present is ~ 1 to 2 vol%. These findings are both qualitatively and quantitatively similar to the crystalline spinels found in previous HLW glasses made from similar HLW glass formulations using radioactive Hanford feeds (HLW sludge slurry, Sr/TRU precipitates and Cs/Tc eluates) at both SRTC and recent PNNL results. This data allows for estimation or projection of the chemical content, crystalline phases and amount present for this waste type as specified by the WAPS referenced in the WTP Contract Specifications.

II. Background and Introduction

The U.S. Department of Energy (DOE) Office of River Protection (ORP) has acquired Hanford tank waste treatment services at a demonstration scale. The River Protection Project-Waste Treatment Plant (RPP-WTP) team is responsible for producing an immobilized (vitrified) high-level waste (IHLW) waste form.^{2,3} Savannah River Technology Center (SRTC), has been contracted to produce and test a vitrified IHLW waste form from Envelope D high-level waste (HLW) samples previously supplied to the RPP-WTP project by DOE.⁴

The primary objective of the present task was to generate HLW glass samples for subsequent product testing.¹ This testing included chemical analysis, radiochemical composition, crystalline and non-crystalline phase determination, Product Consistency Test (PCT), and Toxicity Characteristic Leaching Procedure (TCLP). These tests will help demonstrate the RPP-WTP projects ability to satisfy the product requirements concerning, chemical and radionuclide reporting, waste loading, identification and quantification of crystalline and non-crystalline phases.

In previous Tank Waste Remediation System (TWRS) studies the Immobilization Technology Section (ITS) of SRTC demonstrated with a crucible-scale furnace, the vitrification portion of the waste treatment process proposed for the Hanford privatization effort as of that time. That work carried out in the 1996 to 1998 timeframe produced three Immobilized Low-Activity Waste (ILAW) glasses and a single High Level Waste (HLW) glass.⁵ The past studies were referred to as the small active vitrifications, i.e., crucible-scale vitrifications, of Envelopes A(AW-101), B(AN-105 adjusted to resemble Env. B), C(AN-102), and D(C-106). The previous work for the C-106 HLW glass blended filtered, air-dried C-106 sludge with concentrated Cs/Tc eluate and Sr/TRU precipitate (derived from Sr/Fe nitrate precipitation) and glass former minerals.

The waste envelopes for Hanford Site HLW tank wastes have been defined in general as follows:⁶

- **Envelope A** – This constitutes the majority of the LAW that the facility will process. This envelope contains Cs and Tc at concentrations sufficient to require removal so that the LAW glass specification can be met.
- **Envelope B** - This envelope contains higher concentrations of Cesium than in envelope A or C. Both Cs and Tc will require removal to ensure that the LAW glass specification can be met. This envelope contains higher concentrations of Cl, Cr, F, SO₄, and PO₄, which may limit the waste loading in the glass.
- **Envelope C** – This envelope contains sufficient Cs, Tc, and organically complexed Sr and transuranic radionuclides (TRU) to require removal so that the LAW glass specification can be met. This envelope has twice the concentration limit for SO₄ contract maximum than Envelope A.

- **Envelope D** – This envelope is a sludge waste slurry, with the majority of its radionuclides in unwashed solid form. The slurry will also contain the supernatants, meeting the definitions of envelopes A, B, or C waste.

The Env. D C-106 glass discussed in this report derives from work that started in late 1998 referred to as Part B1 of the Hanford privatization program. Samples of waste representative of the first three feed envelopes (A (AN-103), B (AZ-102), and C (AN-102)) were pretreated and resulting decontaminated supernates vitrified to form ILAW glasses. Additionally, a HLW sludge sample representative of the Envelope D feed was vitrified (the subject of this report) along with the high activity portions separated from the Envelopes A, B, and C waste samples. All of these studies are referred to as “small” active vitrifications, i.e., all were crucible-scale vitrifications (approximately 1.5 L waste samples from Hanford) to distinguish them from an approximately 15 L large-scale melter vitrification study which was recently completed at SRTC for LAW Envelope C waste (Large C or LC). During the preparation of the feeds for the three small and one large LAW vitrifications, the high activity, secondary waste streams were removed prior to vitrification. The cesium eluates and the Sr/TRU precipitate slurry were targeted for inclusion with the HLW C-106 sludge in the present Envelope D glass. The technetium eluate streams were not included in the C-106 HLW glass formulation. One of the technetium eluate streams from the AZ-102 pretreatment was consumed by analytical work from evaporation testing.⁷ It was decided by the acting RPP team (CHG-Hanford, VSL and SRTC) that there was not enough technetium in the other technetium eluate streams to adequately represent the amount of technetium required by flowsheet calculations in the C-106 HLW glass. The targeted streams for the C-106 HLW glass included:

- the cesium eluates from the small Envelopes B (AZ-102),⁸ A (AN-103)⁹ and C (AN-102),¹⁰
- the cesium eluate from the Large C (AN-102) stream,¹⁰
- one precipitated Strontium / TRU stream from the C Envelope samples (large and small),¹¹

The C-106 sludge powder was produced by filtering the washed C-106 sludge slurry previously described.¹² Unfortunately, the actual radioactive strontium/TRU stream was inadvertently combined with other Sr/TRU residuals set aside for return to Hanford, making it necessary to replace this radioactive stream with a simulated, non-radioactive mixture of reagent chemicals. All or portions of these six streams (3 small-scale Cs IX eluates, 1 large-scale Cs IX eluate, 1 simulated Sr/TRU matrix and the radioactive C-106 sludge solids) were combined with selected glass former chemicals and vitrified to produce a HLW glass wasteform.

A simplified flowsheet of the RPP operations is shown in Figure 1. The quantities and specifics for the glass formers were to be supplied by VSL-CUA. The task technical requirements for the vitrification of the D Envelope and some of the feed preparations have been fully described in a

previous Task Plan report.¹³ A listing of the required analytic support for this task is presented as attached Table 1. Note that no technetium was analyzed in the condensate and concentrate analyses since none of the technetium eluates were used in the C-106 HLW glass formulation.

The preparation of the individual HLW feed streams (cesium eluates and sludge powder) was documented in a previous status report document,¹⁴ and all details concerning weights, compositions, and techniques were included. A basic review of these feed preparation steps will be presented in this report. The original data sheets and procedures can be found in WSRC Laboratory Notebooks.^{15,16,17}

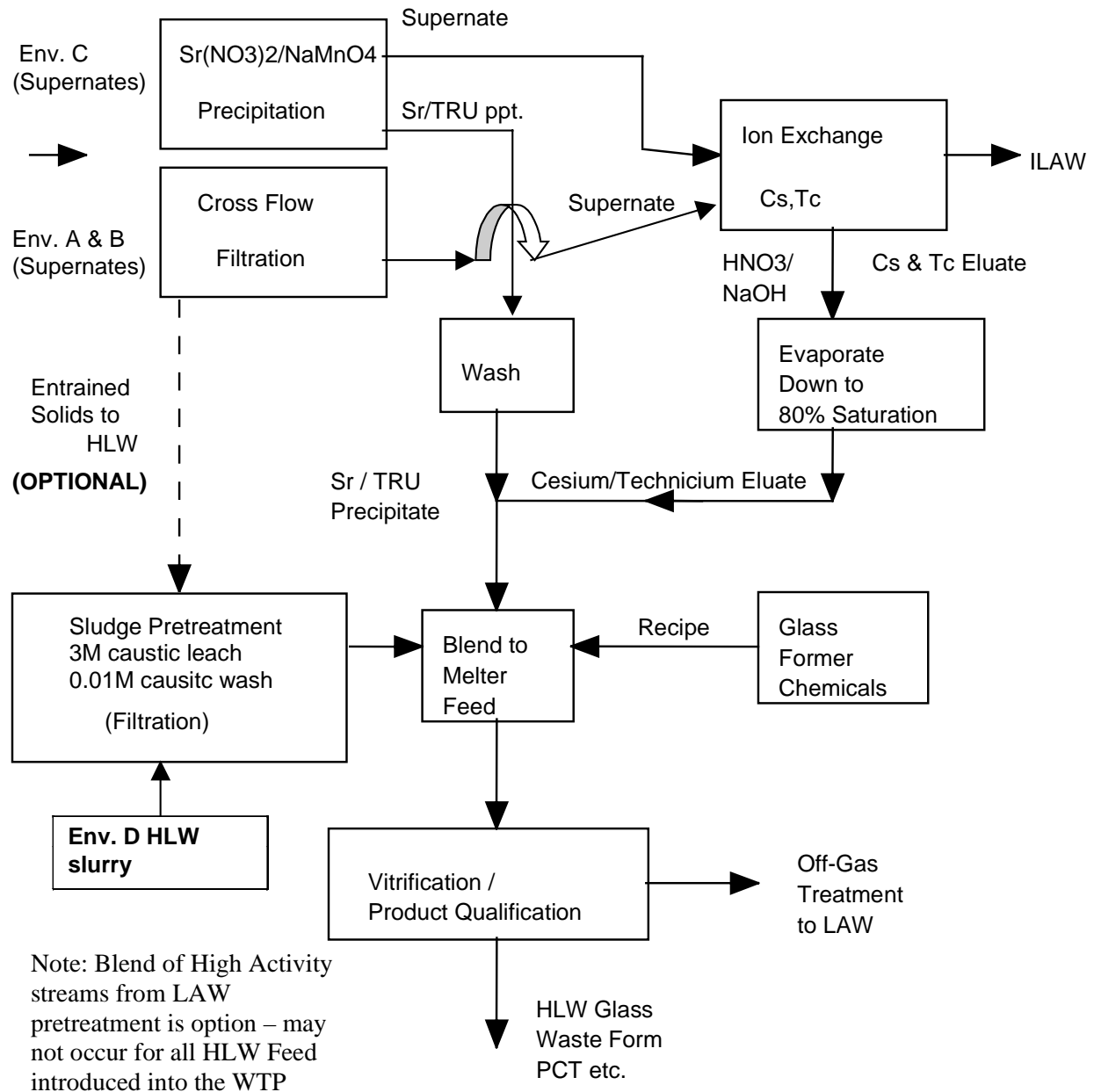


Figure 1. Hanford River Protection Project-Waste Treatment Plant Flow Sheet for Envelope D HLW Waste as of Approximate Timeframe 2000 – 2001.

Note that for this study, no Tc eluate was incorporated into the HLW glass (see text).

III. EXPERIMENTAL

A. C-106 Sludge Filtration

An objective of this work was to separate the HLW sludge from its supernate and chemically analyze both products. The sludge slurry (approximately 1 liter), which was originally labeled (BNF D002, Envelope D, Mike Hay, 10/13/99), was reported to be a 5 wt% solids slurry with inhibited water (0.01 Molar NaOH).¹² The C-106 sludge sample had previously been caustic leached using enhanced sludge washing and gravity settling at PNNL in 1996 and the sample sent to SRTC was an archive sub-sample from the PNNL-leached batch.^{18,19} This HLW sludge, D Envelope (C-106), was filtered through a 0.45 micron filter and dried overnight at 103 to 110°C. Both the sludge powder and the filtrate were analyzed chemically. The filtrate was analyzed in triplicate by dilution and analyzed along with two blanks. The dried sludge powder was analyzed in triplicate by two different chemical dissolutions: sodium peroxide fusion²⁰ and aqua regia dissolution.²¹ Each dissolution involved a dilution of approximately 0.25 grams of powder to 250 mL of water (nominal 1000:1). Two analytical glass standard samples (ARG-1) were included with each dissolution,²² as well as one blank. The dissolved sludge powders, the ARG-1 glass standards, and the blanks were submitted to ADS for chemical analysis. The final chemical results are summarized in Table 2 and a flow diagram of the processing is presented as Figure 2. Detailed data sets for the acid dissolution and peroxide fusion dissolutions of the sludge and analytical standard ARG-1 glass can be found in the earlier Status Report for this work issued by SRTC.¹⁴

B. Cesium Eluate Evaporation and Off-Gas System – Cell 15

Both the Large C cesium eluate (AN-102) and the residual cesium eluates from A (AN-103), B (AZ-102), and C (AN-102) were combined and evaporated down to about 100 mL for inclusion in the glass vitrification. The resulting condensates and concentrates were analyzed by ADS. A glass evaporator with a 1000-mL capacity was inserted into Cell 15 and the unit was heated by a laboratory hot plate with magnetic stirrer.²³ See Figure 3 for a schematic representation of this equipment. The temperature in the evaporator near the bottom was measured with a K type thermocouple and read out. A good (non-violent) boil was maintained, while an evaporation and collection rate of 2 to 9 mL per 15 minutes was considered adequate. The temperature for evaporating deionized water in Cell No. 15 was between 99 to 101°C.¹⁵ A water chiller, using dry ice to chill the water, was employed to condense the vapor. Dry ice was placed in a 4 inch, glass cylinder surrounded by water in an Igloo™ water cooler. The cold water was circulated through the condenser to form condensate in the condensate receiver. Periodically the condensate was drained into a one liter polybottle and held as the condensate. A running total of the condensate was maintained for each run and compared to the measured quantities. After the chill water condenser, a dry ice cold trap was installed, followed by two activated carbon filters. The carbon filters were connected to a vacuum line with the vacuum generated in the cell by a compressed air venturi (Air-Vac forced air vacuum transducer). A vacuum between 2 to 4 inches of water column was maintained throughout the runs.¹⁶

Initially, the large C cesium eluate was evaporated to approximately 100 mL (Condensate I). This concentrate was analyzed by ADS and the results reported in Reference,¹⁴ but this material was later combined with the small A, B, and C eluates and the combined eluates were evaporated down to approximately 100 mL. After this was accomplished samples of the condensate (Condensate II) and a 500:1 dilution of Concentrate II were removed from the cells. These samples were analyzed by ADS and the results presented in Table 3. Figure 4 shows a photograph of the evaporator set-up in cell 15 and Figure 5 provides a flow sheet for the evaporation process and the sampling.

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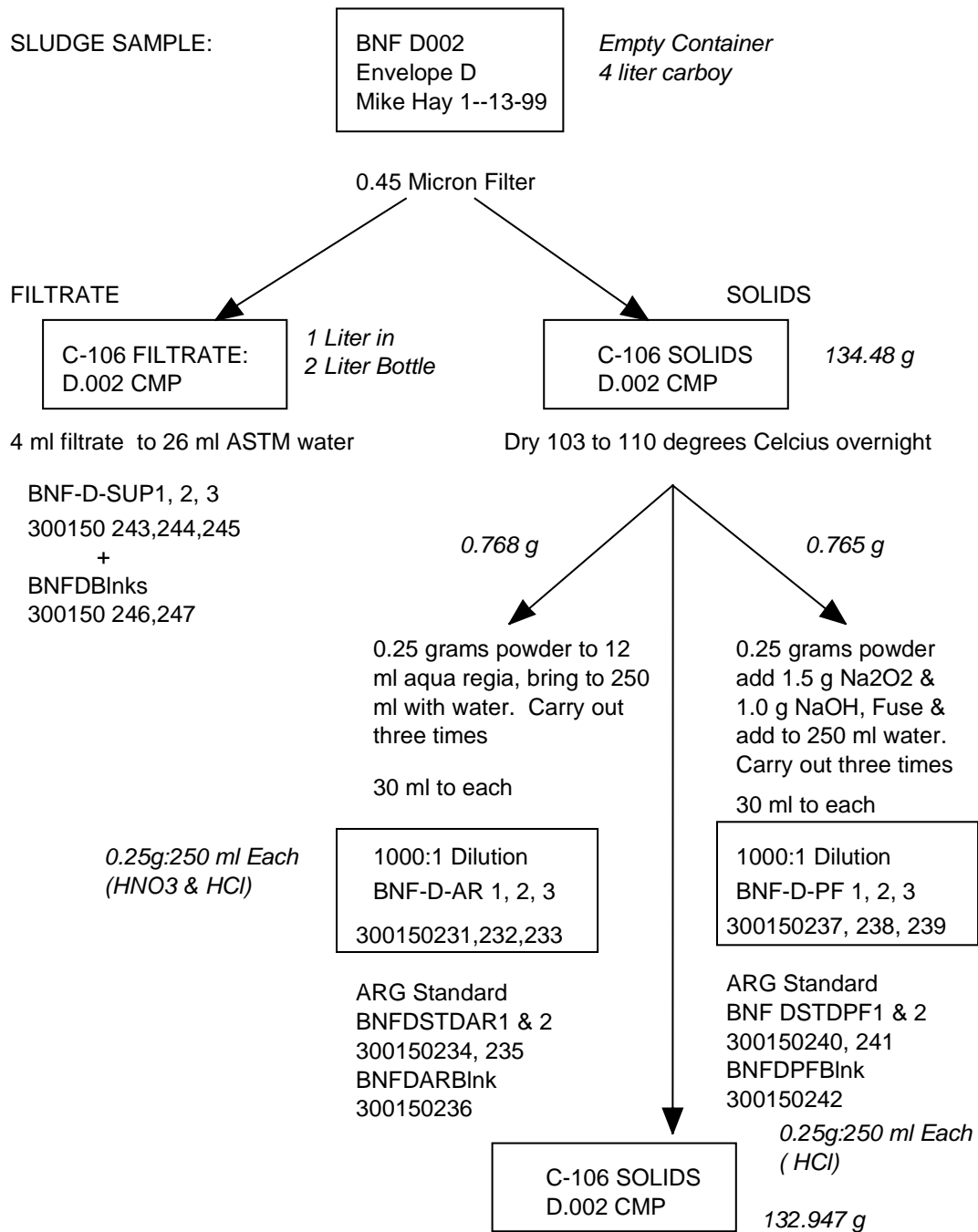


Figure 2. Filtration and Sampling of C-106 Filtrate and Solids

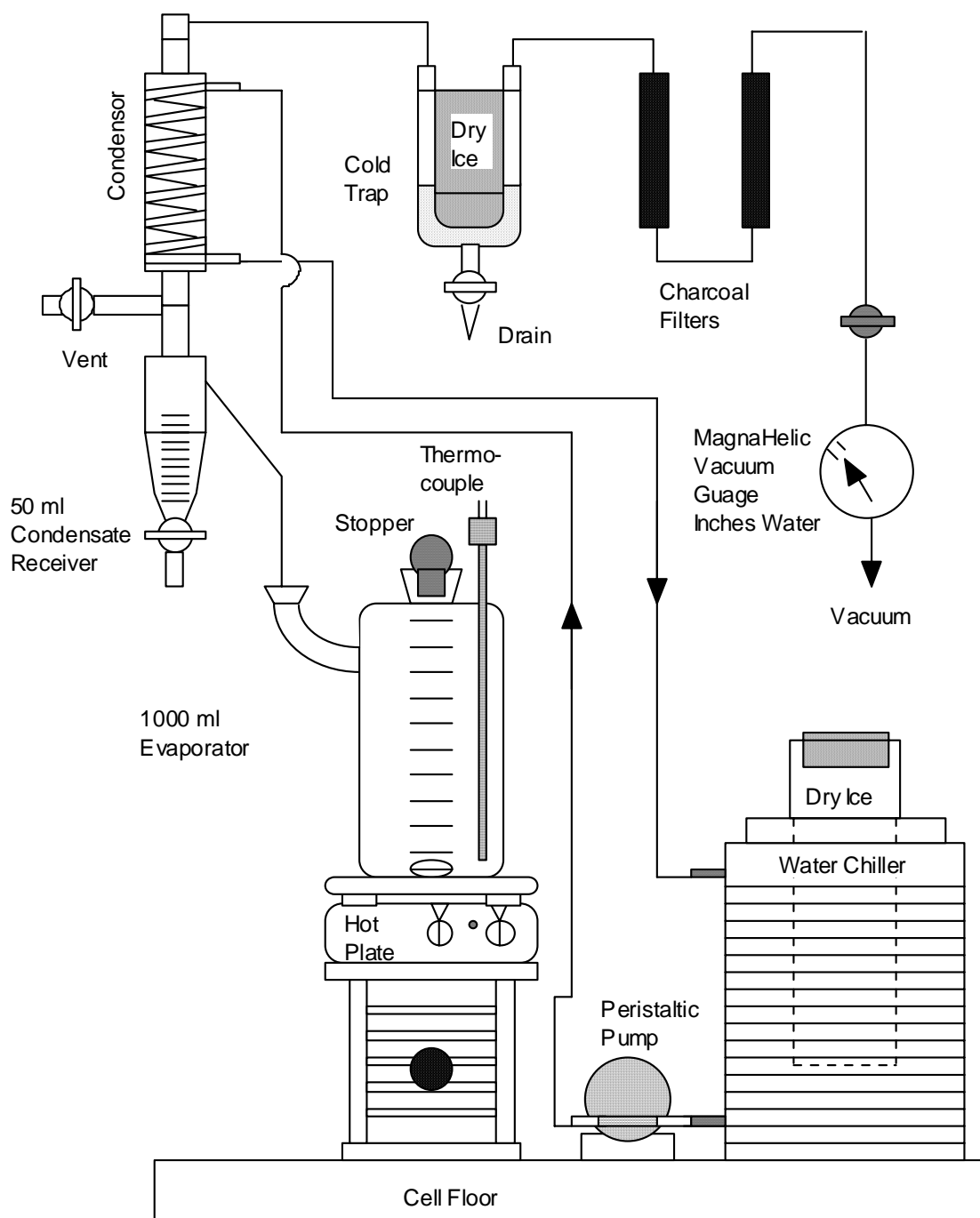


Figure 3 Simple Schematic of the Evaporation Equipment in Cell 15

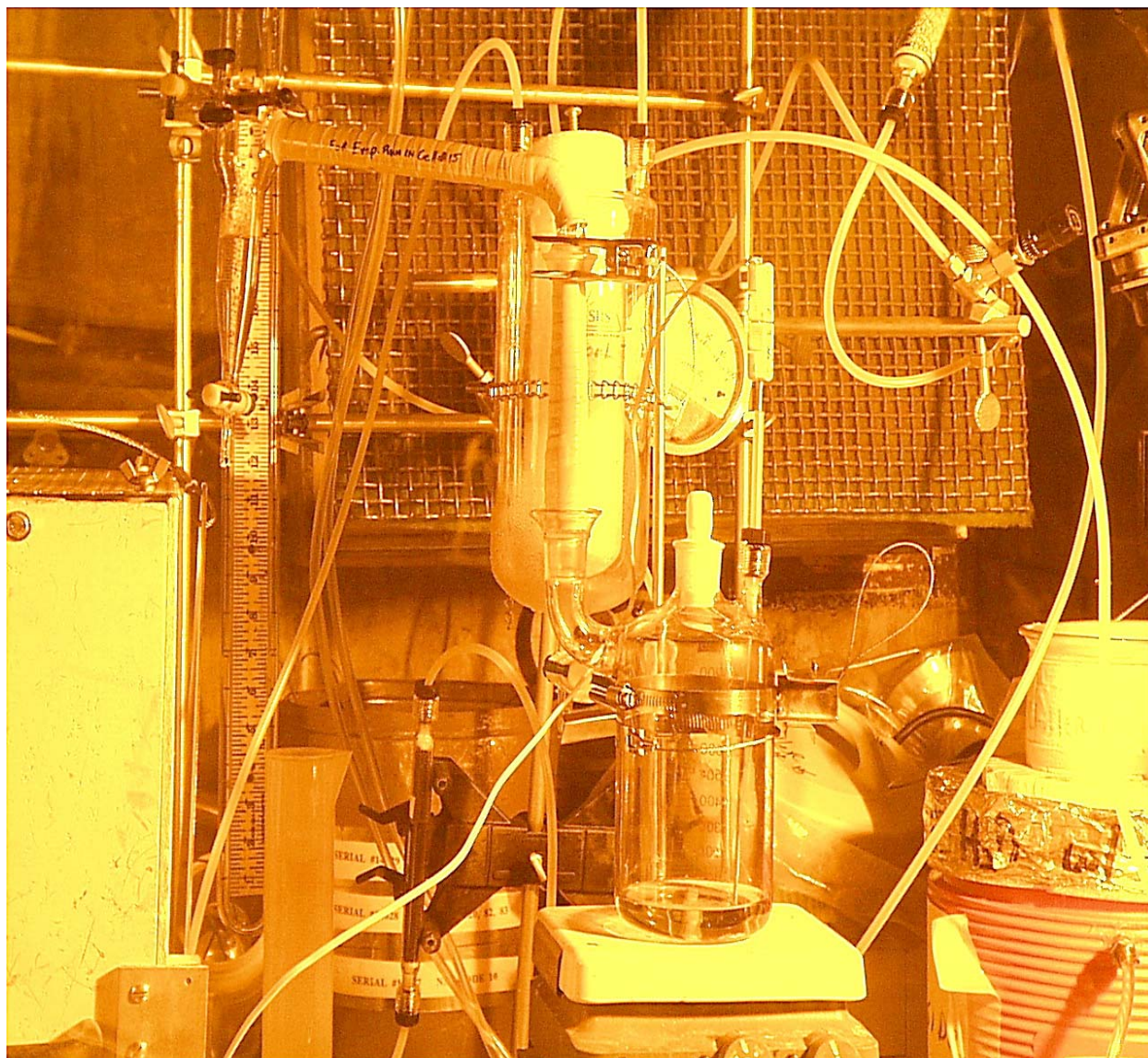


Figure 4. Evaporator Flask and Stirrer/Hot Plate in Cell No. 15

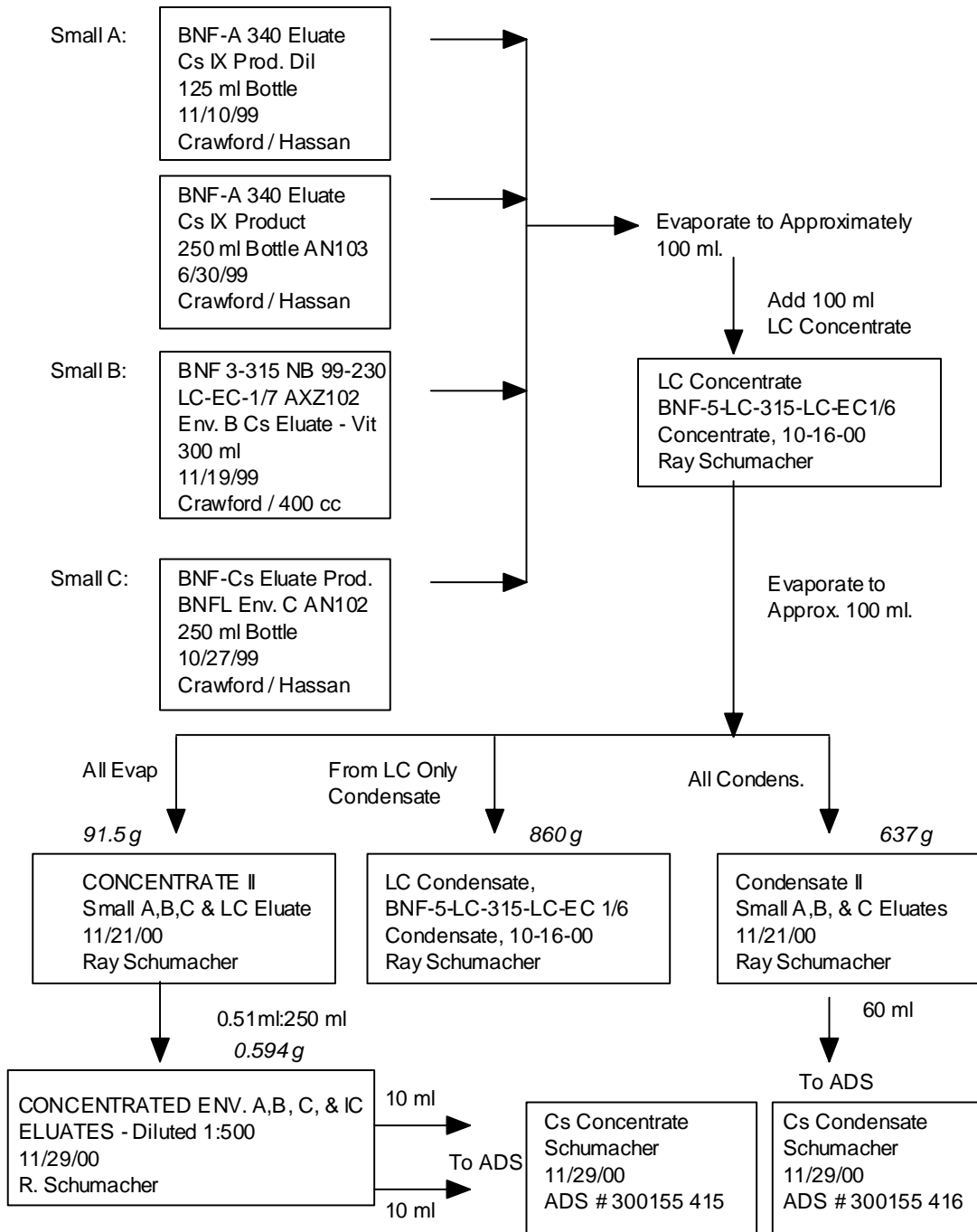


Figure 5. Evaporation of Cesium Eluates and Sampling

C. D Envelope Vitrification - Cell 15

Vitreous State Laboratory prepared a glass composition and glass former list based on analyses of the D Envelope HLW sludge, the composited and concentrated Cs eluate stream (consisting of Cs eluates from pretreatment of AN-103, AZ-102 and AN-102), and the radioactive Sr/TRU precipitate slurry from SRTC. The RPP-WTP team at the time of these activities consisted of Lead: CH2MHill Hanford Group (CHG), Subcontractor: VSL and Subcontractor: SRTC. This team decided to incorporate the entire ~ 100 mL of concentrated Cs eluate and the entire amount of radioactive Sr/TRU slurry (~ 150 mL slurry of ~ 5 wt% solids) into the product D glass. It was decided that all the radioactive strontium/TRU precipitate and cesium eluate concentrate should be used along with a portion (37.0 g) of C-106 dried sludge powder and a mixture of non-radioactive glass formers which would produce approximately 81 g of D Envelope glass. As mentioned previously in the Background and Introduction Section, the team also decided not to include any of the Tc-eluates in the C-106 HLW glass. The target amount of ~ 81 g of D Envelope glass was deemed enough glass to supply all of the necessary tasks following crucible vitrification, i.e., glass characterization, PCT and TCLP leach testing and crystalline determination by XRD and SEM/EDAX.

A copy of the VSL communication consisting of the glass formulation spreadsheet and the textual explanation e-mail of the glass formulation details are attached as Appendix A. Note from the VSL text explanation of the glass formulation that the specific quantities of secondary waste streams used resulted in a Sr/TRU products blending ratio that was slightly higher than the Sr/TRU products blending ratio in VSL C-106/AY-102 melter tests ongoing at that time in January of 2001. The VSL text explanation also indicates Cs elute products blending was lower than the eluate products blending in the VSL C-106/AY-102 melter tests. It should also be noted that the actual surrogate basis for the C-106 radioactive glass formulation used a surrogate formulation labeled 'HLW98-67' from VSL. This formulation resulted from previous studies involving a comprehensive treatment of various glass formulations and testing with RPP-WTP HLW Simulants,²⁴ that was issued by VSL in February of 2001. At this time, we are unaware of any VSL Technical Reports that describe the details of the actual HLW98-67 formulation. Finally, it should be noted from **Appendix A** that the iron content targeted in the HLW glass derives essentially all from the C-106 sludge at a targeted level of 12.5 wt% as oxide in the glass (See bottom-right corner of Total Waste Loading and Env. D Loading and Fe₂O₃ from Env. D data in VSL spreadsheet, Figure 1 of Appendix A). This target for iron in the glass is the only specification that was targeted to meet the numerous possible minimum glass waste loadings of the pretreated C-106 solids as specified in the RPP-WTP Contract Specifications,² Table TS-1.1, "Minimum Component Limits in High-Level Waste Glass".

For summary purposes to all the details discussed above, a listing of the pertinent data sets and communications are listed below as chronological record of the C-106 glass formulation development.

- **9/8/00, SRT-RPP-2000-00007** Communication from SRTC to VSL for waste stream analyses (See Appendix E of Reference 14)
- **11/14/00, Teleconference Call** between CHG, SRTC and VSL – Purpose to discuss C-106 glass formulation (See Reference 17)
- **1/11/01, SRT-RPP-2001-00007**, Communication for final Cs eluate analytical information from SRTC to VSL (See also Appendix E of Reference 14)
- **1/23/01, VSL transmit C-106 glass formulation to SRTC** (See **Appendix A** of this document)

Strontium / TRU Precipitate

While preparing for the vitrification run in Cell No. 15, it was determined that the strontium/TRU precipitate had inadvertently been combined with other samples of strontium/TRU precipitate for residual waste vitrification prior to their return to Hanford. After discussions with the RPP team (CHG, VSL and SRTC), it was decided to prepare a simulated Sr/TRU feed stream from reagent grade chemicals. The composition of this stream was determined during pretreatment¹¹ and this information had been passed on to VSL for inclusion in their glass formulation recipe. Some of the very small trace amounts in radioactive strontium/TRU composition were dropped from the simulated strontium/TRU composition. The development of a simulant permitted more accurate control of the oxide composition but lost the radioactive nature of the actual materials. Table 4 provides the initial composition of the radioactive strontium/TRU stream from **Appendix A** (Note: See the Sr/TRU Stream Element and Oxide data in the columns in the middle of the spreadsheet labeled 'Figure 1. Spreadsheet for Envelope D Glass Formulation Calculations', Appendix A) and compares it with the simulated stream calculated composition prepared from the reagent materials. In Table 4 the first several columns of data at left represent the radioactive Sr/TRU characterization, while the other several columns of data at right represent the simulated Sr/TRU characterization. The radionuclides Cs-137 and Sr-90 are also listed at the bottom left of Table 4 in units of $\mu\text{Ci/mL}$ for the radioactive Sr/TRU precipitate slurry. One can use these analyzed numbers to calculate the amounts of these radionuclides that would have been incorporated in the final glass if the actual radioactive Sr/TRU sample had been used. These values are used later in this report (See Section IV-C Results, Glass Radiochemical Analyses). The weighed amount of simulated strontium/TRU mixture was equal to 20.01 g while the goal was 19.98 g. It should be noted that while trace RCRA metals such as Ba and Ni were included in the surrogate Sr/TRU blend (See Table 4), the RCRA metal Pb was inadvertently left out of the recipe. However, Appendix A glass formulation data indicates that the C-106 sludge contributes about .21 PbO/.27 PbO ~ 78 % of the final Pb as oxide in the glass, while the Cs-eluate and radioactive Sr/TRU contribute each targeted 0.03 PbO/.27 PbO ~ 11 % of the final Pb as oxide in the glass. Thus by not including the trace Pb in the surrogate Sr/TRU mixture, only about 11% of the targeted Pb as oxide in the final glass was not included.

Glass Formers

The recipe for the glass formers was received from VSL and this communication is also shown in Appendix A. The recipe was presented as weight percent of the resulting glass oxides (B_2O_3 -4.99, Li_2O -4.50, and SiO_2 -34.00 wt%). The glass formers thus made up a total of 43.49 wt% of the final glass composition. Three glass former chemicals were selected and the glass former mixture calculations are shown in Table 5. The glass former chemicals used were selected to be typical of the RPP materials that were in use at VSL for developmental work with the HLW surrogate glass formulation development. Also shown in Table 5 are the ADS analyses of the three samples of the glass former material and the conversion to oxide weight percent for comparison. This analysis was accomplished by sodium peroxide fusion with hydrochloric acid uptake,²⁰ followed by ICP-emission spectroscopy of the resulting dissolved glass formers. The agreement between the VSL recipe and the analyzed composition was quite close. The technical data sheet for each of the three glass former materials is included as Appendix B. The required amount of this mixture (43.716 g.) was weighed into a bottle and moved into Cell 15.

D. Vitrification System – Cell 15

The vitrification system was first established in a “mock up cell” and evaluated.²⁵ The operation and the heating schedule were verified using NIST-traceable thermocouples and readout. After verification, the unit was transferred to Cell 15 and re-assembled. Vitrification of the D Envelope streams was carried out in Cell 15 using portions of the off-gas system from the evaporator runs. Figure 6 provides a photograph of Cell 15 from the operations side along with the Deltech furnace controller.

The schematic of the vitrification system in Cell 15 is presented as Figure 7 and a photograph of the set-up inside Cell 15 is shown as Figure 8. A custom-designed front-loading DELTECH Furnace (Deltech, Inc., Denver, CO, Model DT-22-FL-812-E2404) adapted to enclose a quartz glass container was installed as shown. Two thermocouples supplied the control temperature signals to a Del Tec controller outside the cell. Both thermocouples had been recently calibrated in the SRTC Standards Laboratory. The Measuring and Test Equipment (M&TE) labels were GT-1362 and GT-1363. A small amount of alumina powder was placed on the bottom of a large alumina crucible and the platinum crucible was lowered into the alumina crucible inside the quartz bottle. The alumina crucible and powder provided additional containment within the quartz crucible in case the glass melt foamed and flowed over the platinum crucible.

The off-gas exited the quartz bottle through PyrexTM glass tubing and then passed over a water-chilled condenser. The condensate droplets were collected in a graduated reservoir which required periodic draining. A running total of the condensate collection was maintained on the data sheets. A dry ice water chiller supplied the condenser via a peristaltic pump. The use of dry ice to chill the water reduced the amount of liquid waste generated. After the off-gas passed over the condenser, it flowed through a dry ice trap and then through two activated charcoal filters. The off-gas then entered a high-pressure vacuum generator (Air-Vac forced air vacuum transducer) and was released into the cell. A small amount of replacement dry air was drawn into the quartz bottle from a low-pressure air supply. Vacuum was maintained in the quartz bottle at

all times during the vitrification. This vacuum was greater than the small amount of air supplied to the quartz bottle. Procedure GTOP 3-147 was employed for the operation of this unit.²⁵

At start-up, the platinum crucible was removed from the furnace and filled with the cesium concentrate from the small A, B, C and Large C eluates (91.5 g). The dried sludge solids (C-106), Jar 1 (37.26 g), were poured into the solution and stirred with a small spatula. The simulated strontium/TRU powder (20.01 g) was added next. The material became very thick and difficult to stir. About 100 mL of the 500:1 dilution of the cesium eluate concentrate above was added to form the slurry. The prepared envelope D glass formers (43.72 g) were slowly added and allowed to react with the slurry. See Figure 9 for a schematic representation of the amounts and identification of materials vitrified. Note from Figure 9 that the material in the left part of the figure in the oval labeled as '150 mL MnO₂ Wash-4, BNF Residue #4, Env. C AN-102' represents the actual radioactive Sr/TRU product that was NOT added to the glass formulation due to inadvertent recombination of this material with other residues from the Sr/TRU tasks.

Approximately 308 g of material (Cs eluate concentrate, simulated Sr/TRU solids, radioactive C-106 dried sludge solids, glass formers and ~ 100 mL of 500:1 diluted concentrate) was added to the crucible. The crucible was returned to the alumina catch crucible, placed inside the quartz bottle, and placed into the Del Tec furnace. All the glass fittings were reconnected while the peristaltic chilled water pump started and the vacuum adjusted. The programmed controller for the Del Tec furnace was started under a special program to permit dehydration of the slurry and heating to 1150°C without excessive foaming. An approximation of the heating curve is presented as Figure 10. After the slurry had dried, a problem with the vacuum was discovered and it was decided to cool the Del Tec furnace back down to room temperature and replace the charcoal filters in the off-gas line. The quartz bottle was also checked for any leaks in the off-gas line. This seemed to improve the operation and heating was continued. It was later learned that the main problem had been pluggage of the fritted glass support in the charcoal filters.

Monitoring of the furnace around the clock was continued during the vitrification at 1150°C, until the glass was finally cooled to a temperature near the transition temperature, T_g. The total recorded condensate collected during the vitrification was approximately 154 mL. This was later confirmed by weighing the condensate (151.6 g). The actual furnace temperature profiles and controller outputs were recorded from the time the furnace had been shut-down to change the charcoal filters, until the glass temperature approached the transition temperature T_g. These profiles are presented in the Appendix C. After the furnace cooled to room temperature a black or dark deposit on the inside of the dry ice trap was noted. This gradually disappeared after the vacuum to the off-gas system was turned off. It is believed that this dark deposit is a condensed form of nitrogen oxide. The charcoal filters were removed and weighed. The total weight gain for the three filters was 14.50 grams. The total weight balance for the vitrification is presented in Figure 9. A little over 50 g was not accounted for and may be due to nitrates, carbonates, etc. These gases would not be captured by the off gas system.

Using the waste stream and glass former analyses, the actual measured material amounts and the information from glass formulation presented in Appendix A, the composition (based on actual measured amounts of materials) of the Envelope D glass with secondary waste streams was

calculated and is presented in Table 6. The calculated glass composition based on actual mass measurements of the various waste streams and the composition supplied by VSL differ by only about 1 %, which indicates that all amounts of each waste stream actually used in preparing the HLW glass are very close to the amounts prescribed by the original VSL glass formulation.

The crucible containing the glass was moved from cell 15 to cell 14 for weighing. A large piece of the glass weighing 29.33 g was placed into a glass jar for archiving. The remainder of the glass was crushed by striking the glass inside a plastic bag with a hammer. This material weighed 53.58 g and was used to prepare samples for chemical composition and radiochemical analysis, TCLP analysis, and PCT analysis. Table 7 is a record of the glass preparation and how it was portioned for use.



Figure 6. Operator at Shielded Cell No. 15 with Del Tec Furnace Controller on Right

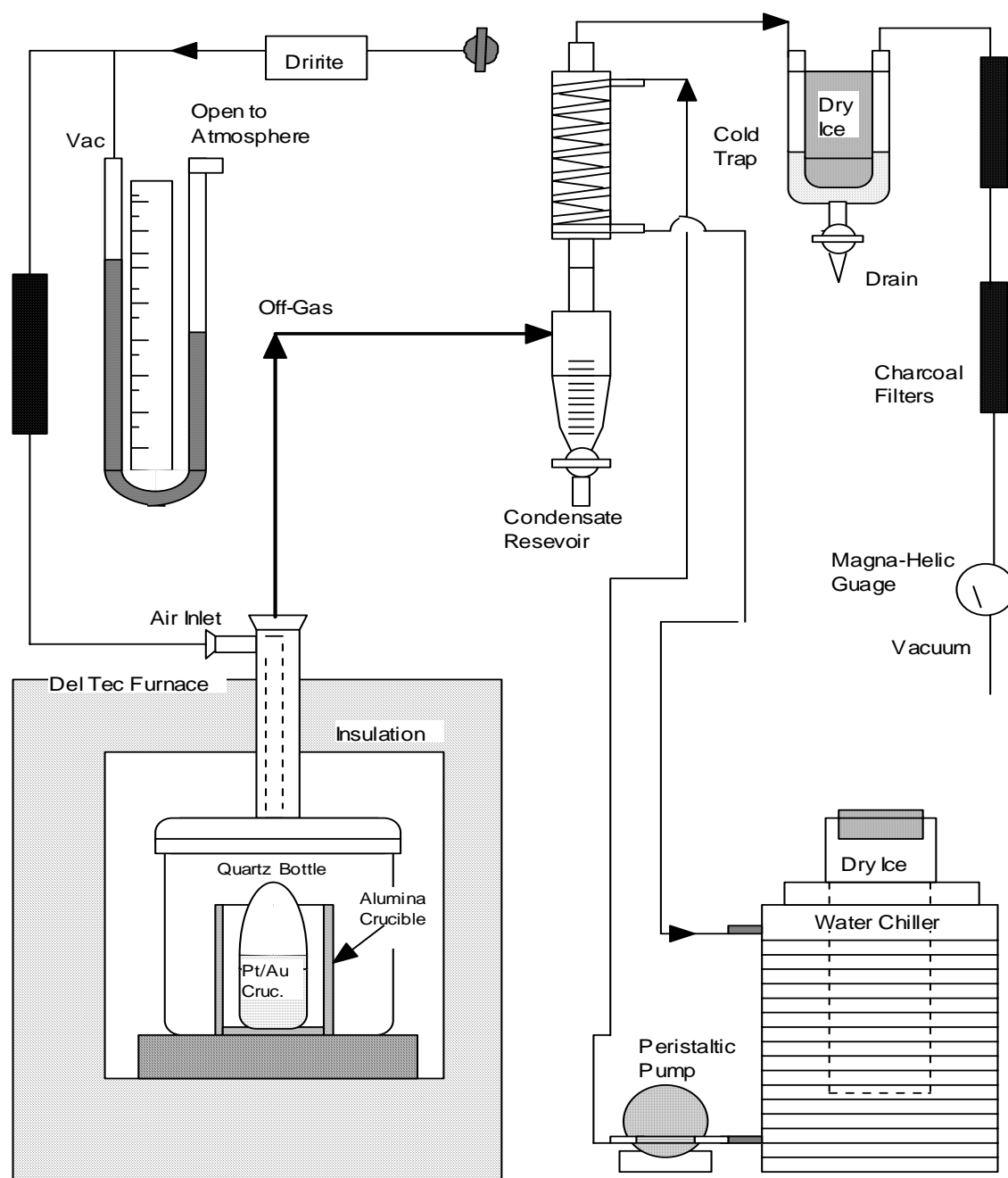


Figure 7. Schematic of Vitrification System in Cell 15

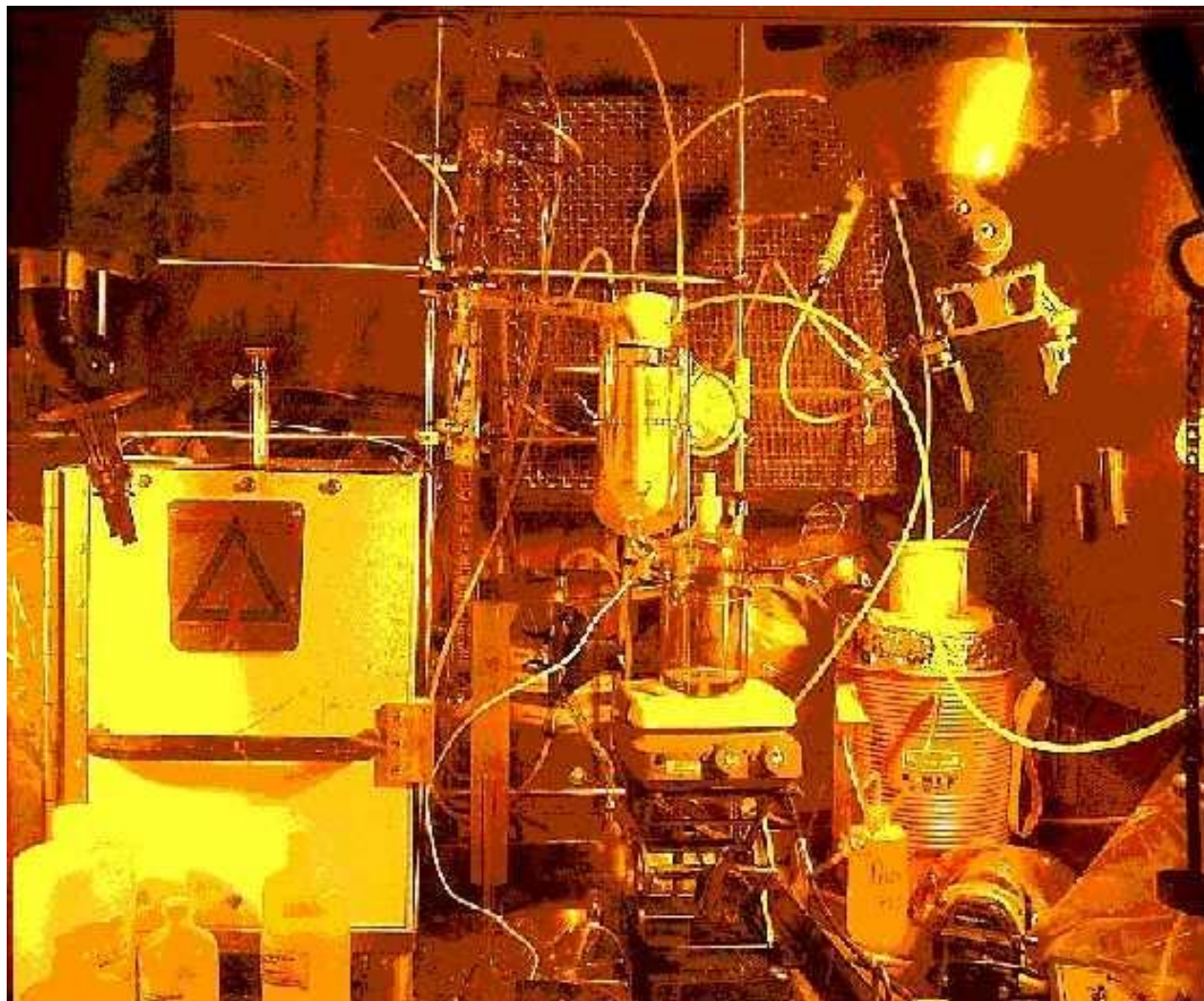


Figure 8. Del Tec Vitrification System, Evaporator, and Off-Gas System in Cell 15

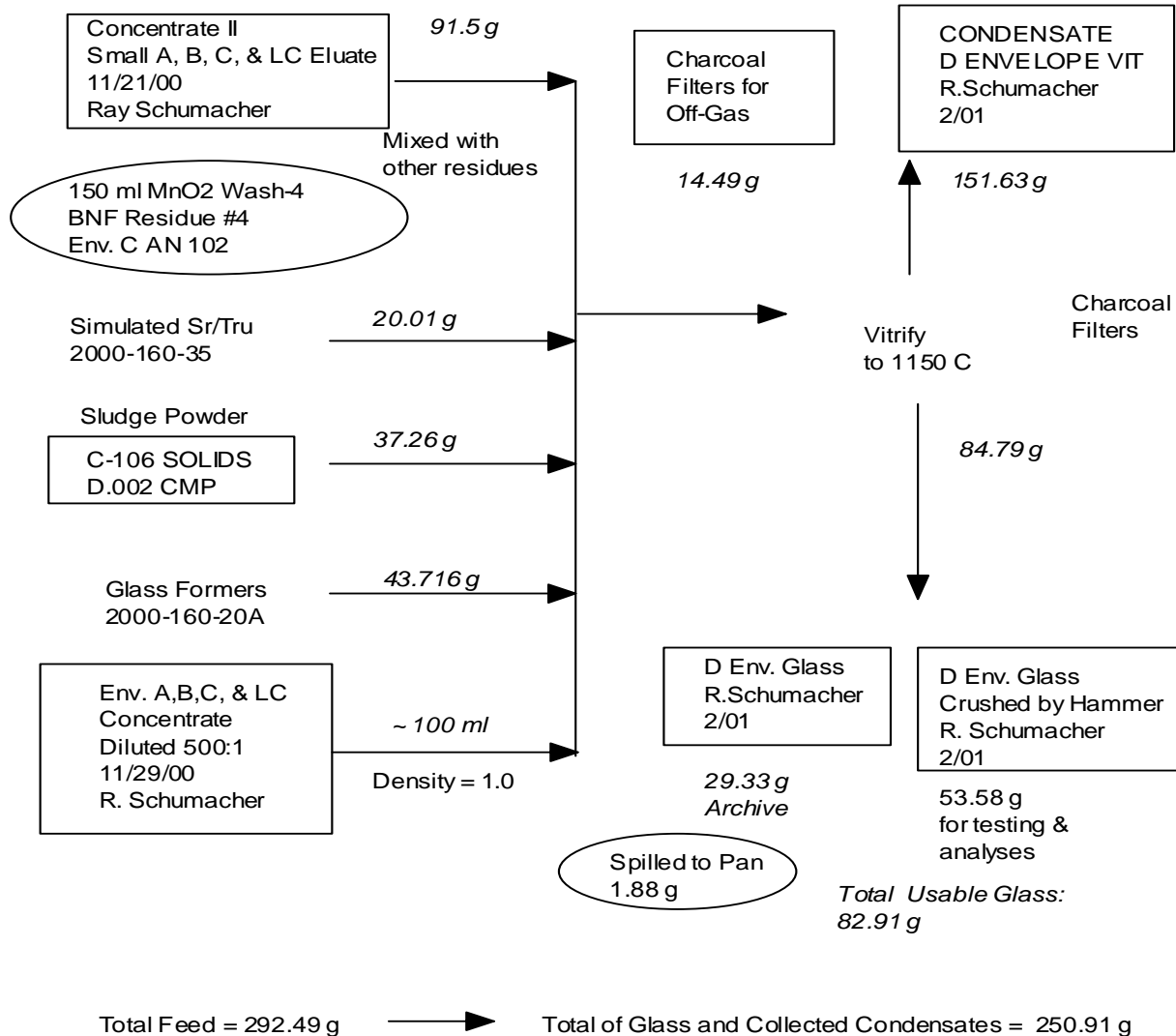


Figure 9. Components for the Vitrification of D Envelope Glass in Cell 15.

Note that the BNF Residue #4, MnO₂ Wash4, radioactive Sr/TRU material shown on left side of figure in oval was not actually used in radioactive glass feed. A simulated Sr/TRU stream was actually used.

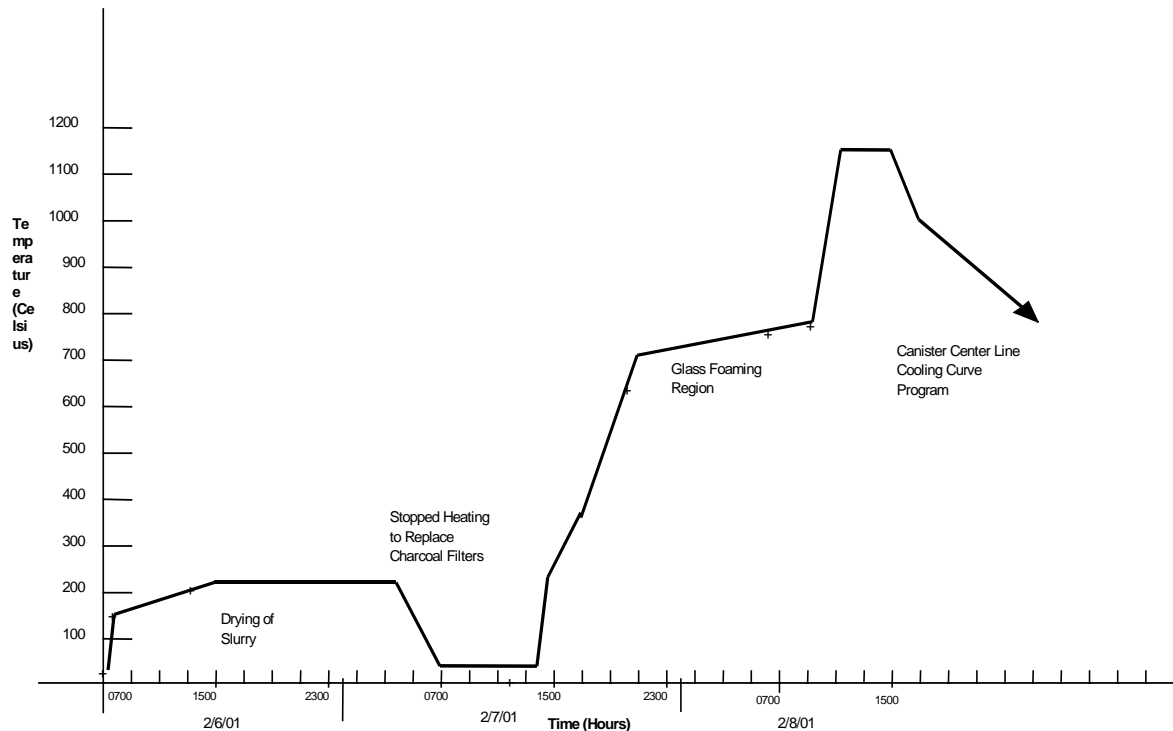


Figure 10. Summary of Del Tec Furnace Heating Schedule

E. Glass Chemical and Radiochemical Analysis

Written directions were employed to grind the D Envelope glass sample for the dissolutions. The glass was transferred to Cell 15 and ground in a SPEX 5300 CertiPrep grinder with agate (SiO_2) vial, cap and ball.¹⁶ The resulting glass powder was screened to -200 mesh (<75 micron). The glass powder samples were returned to Cell 14 for the dissolution procedures. A sample of ground analytical glass standard ARG-1 glass powder was also used to provide a comparison standard.

Peroxide Fusion Dissolution – Initial Dissolution of the C-106 HLW Glass with Nitric Acid Uptake

The first dissolution of the D Envelope glass was by sodium peroxide fusion and followed a modification of Procedure ADS-2502.²⁰ This procedure used 0.25 gram of the glass powder to 1.5 gram of sodium peroxide plus 1.0 gram of sodium hydroxide in nickel crucibles. The mixture was heated for about 10 minutes in a calcining oven at 675°C. The resulting fused mixture was then dissolved with water and 25 mL of concentrated nitric acid. The acid normally used in this procedure was hydrochloric. However nitric acid was felt to be more compatible with

the ADS waste treatment capabilities. The resulting solution was dark or black in color. This color was suspected of being caused by some of the nickel oxide from the crucible. The dissolved material was poured into a 250-mL volumetric flask and diluted with deionized water to the 250-mL mark. The resulting dissolution was placed in a polybottle and was approximately 1000:1 dilution. This was repeated three times for the D Envelope glass and twice for the ARG-1 standard glass. A 250-mL blank containing the sodium peroxide and sodium hydroxide was also prepared. All the samples were dark in color. Duplicate (A & B) 10 mL samples of each of the 1000:1 dilutions were removed from the cells. The whole body dose from these samples was about 4 mrem/hr at 30 cm whole body and 250 mrem/hr extremity. The samples were sent to ADS for analyses with the A samples submitted for radiolytic measurements and the B samples for the ICP-ES, ICP-MS, and AA analyses. The ADS sample numbers were 300160285 to 300160290 (A&B). Samples # 285 – 287 were radioactive, samples # 288 and 289 were standard glasses and sample # 290 was a reagent blank. The chemical analyses for these samples are presented in Appendix D. This analysis indicated incomplete dissolution of the D Envelope glass although both the analytical reference standard ARG-1 and Blank appeared to be acceptable.

Peroxide Fusion Dissolution – Surrogate Glass from VSL using Nitric Acid Uptake (Initial Trial) and Hydrochloric Acid Uptake (Second Trial)

The problem of incomplete dissolution by the above peroxide fusion with nitric acid uptake was investigated initially by obtaining a sample of similar non-radioactive glass (HLW98-67) from VSL-CUA. This glass had been chemically analyzed by VSL-CUA. The glass was initially analyzed at SRTC in duplicate with the same procedure previously employed in Cell 14. A slightly higher furnace temperature (694°C) was employed for the fusion but the nickel crucibles and nitric acid were used. Dilution was a nominal 1000:1. A blank was also prepared. A similar black solution was obtained and the liquids were filtered prior to analysis. The ADS Laboratory Information Management System (LIMS) numbers were #300163442 - 300163444. Only ICP-ES analysis was requested. The data is also presented as Appendix E. Most of the analyses compared well with the VSL-CUA analysis, except the MnO was low by about 3 wt% and the Fe₂O₃ low by about 0.5 wt%. After discussions with ADS, it was decided to repeat the dissolution in a second attempt returning to the original hydrochloric acid uptake procedure. It was believed this would be more effective in bringing the MnO into solution. Again using the glass from VSL, duplicate dissolutions were repeated along with duplicate analytical reference glass ARG-1 standards and one blank. The LIMS numbers for these analyses were 300 163-834 to 838. Dilution was again a nominal 1000:1. The results are included in Appendix E. The agreement with the VSL-CUA analysis of HLW98-67 was very good. No major disagreements were detected.

Peroxide Fusion Dissolution – Second Dissolution of the C-106 HLW Glass with Hydrochloric Acid Uptake

A second peroxide fusion of the actual D Envelope glass using hydrochloric acid uptake was initiated some time later. Fresh D glass was crushed with the SPEX 5300 CertiPrep grinder. See

Table 7 for accountability of the Envelope D glass. Approximately 0.8 grams was prepared for the three dissolutions.

The glass samples were prepared with the hydrochloric acid uptake and diluted to approximately 1000:1, i.e., nominally 0.25 g of glass was dissolved in total 250 mL. The radioactive D-glass samples were further diluted by combining 3 mL of the 1000:1 dilution with 6 mL of ASTM water before removing from the high level cells. This provided a dilution of a nominal 3000:1. The measured whole body dose was 4 mrem/hr and the extremity dose was 94 to 130 mrem/hr at contact. The ARG-1 analytical reference standard samples were prepared in duplicate and a single blank was prepared. The LIMS numbers for all of these samples were from #300163910 to #300163915 (A samples) and #300163916 to #300163921 (B samples). The A samples #910 - 915 were again used for the radiochemical determinations and the B samples #916 - 921 for the ICP-ES, ICP-MS, and AA analyses. The analysis was calculated and is presented Appendix F. The silicon level of the ARG-1 glass was about 2 elemental wt% high, or about 10% of the total elemental silicon amount, and this was used to bias-correct the D Glass silicon levels, i.e., lower the measured silicon levels in the D glass. The iron level of the ARG-1 glass was about 0.9 elemental wt% low, or about 10% of the total elemental iron amount, and this was used to bias-correct the D glass iron levels upward.

Acid Dissolution of the C-106 HLW Glass

For the acid dissolutions, procedure ADS- 2227 was used as written.²⁶ This procedure uses a mixture of the acids: nitric, hydrofluoric and ultra pure boric acid solution, to digest the glass in pressure vessels at 115°C for two hours. Hydrochloric acid is added and the vessel is reheated for another one half-hour. The dissolution is then diluted 1000:1 in a 250 mL flask. The material is then sampled and diluted further by removing 3 mL of the material and adding 6 mL of ASTM water for a nominal dilution of 3000:1. The D glass was dissolved in triplicate along with two ARG-1 analytical reference standard glasses and one blank. The standard glasses and blanks were not diluted 3:1 but removed as 1000:1 samples. The resulting samples were wiped down and passed on to ADS for analysis.

The LIMS numbers for the samples were 300161691A&B to 300161696A&B. Samples # 691, 692 and 693 were the radioactive D glass, samples # 694 and 695 were the standard ARG-1 glass and the final sample # 696 was a reagent blank containing no dissolved glass powder. The samples were sent for radiochemical measurements and the B samples for ICP-ES, ICP-MS, and AA analyses. The complete analyses are presented in Appendix G at the end of this report. Dilution 1 refers to the initial 0.25 gram glass to 250 ml water (radioactive and standard glasses), while the second dilution is the 3 ml of sample to 6 ml of water for the radioactive D glass samples only. The blank appeared to be somewhat high in silicon and boron as would be expected. The silicon is derived from the instrument fused silica parts and the boron is from the boric acid addition to the blank. The average of the two ARG glass standards was compared to the accepted target values. Good agreement was obtained for all elements except boron and silicon. The boron number was not used since boron was used to stabilize the acid solution. The silicon was about 2 elemental wt% high compared to the standard and this correction was used to bias-correct the silicon for the D Envelope glass, i.e., lower the measured values of silicon in the

D glass. The iron values measured for the standard glass were only slightly higher by 0.1 elemental wt% iron vs. the target, and this slight ~ 1% correction was applied to the measured D glass iron amount, i.e., the measured iron levels in the D glass were lowered by about 1%.

Radiochemical Analyses

Radiochemical analyses on the dissolved glasses (peroxide fusion and acid dissolution) were performed on all samples (radioactive, standard and reagent blanks) to investigate the levels of radionuclides in the radioactive glasses and to obtain information on possible contamination of the standard glass dissolutions and reagent blanks that were all prepared inside of the SRTC shielded cells facility.

Pu Separation and Analysis: An aliquot of the sample was subjected to a thenoyltrifluoroacetone (TTA) separation. An aliquot of the sample was initially spiked with a Pu-239 tracer. A second aliquot of straight sample was analyzed along with the spiked sample. In addition, a third aliquot was used for determining the Pu-241 concentration via liquid scintillation analysis. All of the plutonium in the samples was reduced once using hydroxylamine. An anion complexing reagent (aluminum nitrate) was then added, and the solutions were oxidized with 4M sodium nitrite. The plutonium was then extracted from the matrix using a TTA solution. The TTA layer was mounted on a counting dish, the mount was then analyzed by alpha spectroscopy. A blank sample was run with the sample set. All analysis results for the plutonium separations were yielded using the Pu-239 recoveries from the Pu-239 traced sample separation.

Gross Alpha/Beta Analysis (TK50): In order to complete the radiological screening analyses for gross alpha and gross beta constituents, an aliquot of the sample was added to liquid scintillation cocktail. In order to monitor and correct for any alpha/beta spill-over events, an identical sample spiked with alpha activity was analyzed in sequence with each unspiked sample. The samples were counted on a Packard Instruments liquid scintillation counter along with an instrument blank. The instrument blank was counted first and was used to establish an instrument background that was subtracted from the count results for the samples.

Gamma Analysis: An aliquot of the sample was analyzed by gamma spectroscopy analysis using a high purity germanium detector. Results were background subtracted. Only Cs-137 was detected as gamma emitter in the samples. All other gamma emitter radionuclides were reported at below minimal detectable activities.

Sr-90 Separation and Counting: Each sample was analyzed for Sr-90 using an Eichrom Sr-Spec based extraction procedure. A Sr-90 spiked blank and a duplicate sample spiked with Sr-90 were analyzed with each sample batch to establish Sr-90/Y-90 counting efficiencies and Sr chemical recoveries. Once the extractions were complete, the extracts were given at least one week to reach equilibrium. After this in-growth period, aliquots of the resultant Sr-90/Y-90 containing extracts mixed with liquid scintillation cocktail were counted in the ADS Radiochemistry Counting Room. The samples were counted on a Packard Instruments Model 2550 TR/AB liquid scintillation counter along with an instrument blank. The instrument blank was counted first and was used to establish an instrument background that was subtracted from the count results for the samples.

Americium-241 and Curium-244 both decay primarily by alpha decay with respective energies of 5.48 Mev/disintegration and 5.80 Mev/disintegration. Dissolved glass samples were treated to separate Am-241 and Cm-244, followed by alpha counting to determine the levels of Am-241 and Cm-244.

Mass spectroscopy data was obtained for the actinide masses and the fission fragment masses at mass > 81. The actinide data in units of $\mu\text{g/L}$ were converted to $\mu\text{Ci/g}$ glass values by assigning each actinide mass number from mass 231 to 244 with the most probable actinide radionuclide and converting the mass/volume values using standard equations involving each radionuclide half-life and mass number. Fission fragment data from mass spectroscopy was not converted from mass/volume to curie/g glass (except for Tc-99) due to the various radioactive and nonradioactive contributing analytes to each mass number. In certain cases, the fission fragment mass spectroscopy data can be used to estimate upper limit values for various radionuclides.

F. Glass Leaching (PCT and TCLP) and Phase Determination

The experimental procedures and results for the PCT and TCLP testing are included separately as Appendix H and Appendix I, respectively. The preparation and experimental data pertaining to XRD and SEM/EDAX are included as Appendix J.

IV. Results

A. Vitrification

The D Envelope flow sheet was carried out in the shielded cells with samples of Hanford RPP radioactive waste streams: Envelopes A, B, C, and D. The waste streams were combined with glass formers and a simulated strontium/TRU stream to form slurry feed for vitrification in an electric resistance furnace. During the vitrification the glass was maintained in the platinum crucible and upon cooling at the canister cooling rate and removal, nearly 85 g of black glass was obtained. This compared quite well with the expected weight which was 81 g. The mass balance shown in Figure 9 indicates a total of 292.49 g of feed and a total weight for the glass product, collected condensate, and activated charcoal adsorption as 250.91 g. This accounted for nearly 86% of the feed material. The remaining 14% of feed material is attributed to the nitrate and

carbonate salts from the Cs-eluate (See Table 3), the surrogate Sr/TRU powder solids (See Table 4), and the carbonate included in the glass former minerals (See Table 5). These salts would have left the evaporation, calcine and vitrification system as CO_x and NO_x species not captured by the offgas. Inclusion of these salts totaling about 32 grams into sum of measured products, i.e., 250 grams product plus 32 gram non-captured salt, allows for accounting of total feed mass (~ 292 grams) vs. total measured product (~ 282 grams) to about 97%.

B. Glass Chemical Analyses

Two analytical procedures were employed for the dissolution of the D Envelope glass for subsequent chemical analyses. These two procedures were sodium peroxide fusion and acid dissolution. The peroxide fusion procedure required the use of hydrochloric acid (replacement for nitric acid) in order to maintain the manganese in solution. The acid dissolution procedure was used as written. One of the three replicate analysis sets resulting from acid dissolution of the glass appeared unusually low and was dropped. Both analytic methods required a bias correction for SiO₂ based on comparison of the analytical reference standard glass measured Si vs. target Si. In both cases, the measured silicon in the analytical reference standard glasses were higher than target, thus the measured silicon values for the D glass samples were bias-corrected, i.e., lowered by the correct ratio. The measured iron levels in the D glass samples were also bias-corrected vs. the measured values compared to target for the analytical reference standard glass. The average of the remaining two analyses was combined with the average from the three peroxide fusion analyses to form the best estimate chemical composition for the D Envelope glass. These results are presented in Table 8. Table 8 is a summary table for the glass dissolution data previously discussed and shown in Appendix F (peroxide fusion with hydrochloric acid uptake) and Appendix G (acid dissolution). It should be noted that Table 8 is a summary table for the D Envelope glass while the specific data sets for each dissolution including the dissolved analytical standard glasses are presented in their entirety in Appendix F and Appendix G. The analyzed glass was quite similar to the calculated composition supplied by VSL as each measured elemental species was typically within about +/- 5% of target. This +/- 5% range is typical of the spread in analytical measurements for dissolved glasses. The last column of Table 8 shows that the following elements were present in the glass as their oxides at above the 0.5 wt% level: Al, B, Ca, Fe, Li, Mn, Na, Si and Sr. This minimum reporting level of 0.5 wt% is specified in the WAPS²⁷ for reporting chemical composition of vitrified HLW glass components.²⁷ The next to last column in Table 8 indicates that all of these elements targeted above were measured to be above the 0.5 wt% on oxide basis. Two other minor elements were also measured to be above this 0.5 wt% oxide basis – P and Zr. Values shown in the next to last column of Table 8 are targeted values for Cl, F and SO₃ in the glass as these specific elements were not measured in the C-106 HLW glass.

The chemical composition data for the Envelope D glass can be used to determine that the product glass meets the RPP-WTP Contract Specifications, Table TS-1.1, “Minimum Component Limits in High-Level Waste Glass,” gives several different criteria for acceptance.² The summary data in Table 8 show that the Envelope D glass has measured elemental iron (when bias-corrected for analytical standard reference glass data) in the range of 8.79 to 9.01 wt%. When the average and one-sigma standard deviation of these values is converted to iron on an

iron oxide bases (Fe_2O_3), the measured range of Fe_2O_3 in the glass is in the range of 12.72 ± 0.22 wt%, compared to the minimum specification value of 12.5 wt% Fe_2O_3 . As noted earlier in Section III-C, this specification concerning iron content in the HLW glass from pretreated C-106 sludge solids was the only specification targeted vs. the list of possible minimum component limits listed in the Contract Specification Table TS-1.1.

C. Glass Radiochemical Analyses

Summary radiochemical analyses of the Envelope D glass are also shown in Table 8. These data result from the various radiochemical analysis techniques applied to the dissolved Envelope D glasses. Cesium-137 measured by gamma spectroscopy is shown to be the highest radioisotope present in the glass. The Cs-137 derives from Cs ion exchange eluates added to the glass formulation with a minor contribution from the C-106 sludge. Cs-137 decays to Barium-137m that is a metastable decay product of Cs-137 and is in secular equilibrium with Cs-137. The activity of Ba-137m is 95% of that for Cs-137 since 5% of the Cs-137 decays directly to stable Ba-137.

All other radioisotopes shown in Table 8 derive primarily from the C-106 radioactive sludge added to the glass formulation. The primary radioisotope from the C-106 sludge is Sr-90 (determined by separation and beta-scintillation counting) that is present in the glass at ~ 2 orders of magnitude lower ($\sim 400 \mu\text{Ci/g}$) relative to Cs-137 ($\sim 40,000 \mu\text{Ci/g}$). Yttrium-90 (half-life = 2.671 days) is a beta decay product of Sr-90 (half-life = 28.5 years) and is in secular equilibrium with Sr-90. Thus the concentration of this short-lived Y-90 daughter-product is equal to Sr-90. As mentioned previously, the radioactive Sr-90 in this product glass is lower than would be present if actual radioactive Sr/TRU precipitate slurry had been used as feed to the glass formulation.

Dissolved glass samples were screened for total alpha and total beta counts. Table 8 shows the total alpha counts to be in the range of 40 to 120 $\mu\text{Ci/g}$ and the total beta counts are much higher at about 40,000 $\mu\text{Ci/g}$. The alpha activity in these dissolved glasses derives from the transuranic sludge components added from the powdered C-106 sludge (since no radioactive Sr/TRU precipitate was actually added in these tests). Actual measurements involving separation techniques and radiochemical counting show the sum of individual transuranic radionuclides (Am-241, Cm-244, Pu-238, Pu-239+240) to total about 4.4 $\mu\text{Ci/g}$ or 4,400 $\eta\text{Ci/g}$ for the acid dissolved Envelope D glass. Similar measurements for the peroxide fusion dissolved glasses showed a low bias for Am-241 and Cm-244.

Total beta counts can be compared to the Cs-137 values to show that all of the measured gross beta counts essentially derive from Cs-137. The total beta analyses are in the range of (3 to 4) $\text{E}+04 \mu\text{Ci/g}$ compared to the Cs-137 values in the range of (3.5 to 3.9) $\text{E}+04 \mu\text{Ci/g}$. The Sr-90 and Y-90 beta decays are likely not significantly represented in this total gross beta counting since Sr-90 is present at levels that are 2 orders of magnitude lower than Cs-137 (again, since no radioactive Sr/TRU precipitate was actually added in these tests).

Gamma spectroscopy minimal detectable activity data were determined for all the dissolved radioactive glasses, standard glasses and reagent blanks. These data are shown in Appendix F (peroxide fusion dissolved glasses with hydrochloric acid uptake) and Appendix G (acid dissolved glasses). Cs-137 was detected at trace levels in some of the nonradioactive dissolved glasses and reagent blank. No other detectable gamma emitters were detected in these analyses. Therefore it should be noted that all minimal detectable activity gamma spectroscopy levels shown in the gamma spectroscopy data tables of Appendix F and Appendix G are upper limit values – actual values may be considerably lower.

Other radiochemical data can be obtained from the mass spectroscopy results for the dissolved glasses. The ICP-MS data is shown in Appendix F for the peroxide fusion dissolved glass and Appendix G for the acid dissolved glass. Reported values from ICP-MS analyses in $\mu\text{g/L}$ were converted to wt% of analyte in the glass using the appropriate dilution factors. These dilution factors were nominally 1000X for the 0.25 grams of glass dissolved in 250 mL of solution, another ~ 3X dilution that involved diluting the dissolved radioactive glass products in order to remove them from the shielded cells facility up to the analytical labs, and finally certain ICP-MS instrument-specific dilutions of typically 10X to 100X. Certain masses of the actinide isotopes were also converted to $\mu\text{Ci/g}$ values using the half lives shown in the tables. This conversion was also performed for mass-99 for Tc-99. This conversion associated with mass-99 to Tc-99 assumes all mass-99 identified by ICP-MS derives from technetium-99 with no contribution from ruthenium.

Mass spectrometry values for the various actinides and Tc-99 in units of $\mu\text{g/L}$ were converted to specific activity values via Equation #1,

Equation #1:

$$a_i/g_g = 3.5778\text{E}+05 \times (g_i / (t_y \times M))$$

Where:

a_i = activity of isotope in Curies (Ci)

g_g = mass of glass

$3.5778\text{E}+05$ = constant (Ci*years/g)

g_i/g_g = mass of isotope in grams/ mass glass

= $\mu\text{g/L} \times (1\text{g}/1\text{E}+06\mu\text{g}) \times .25\text{L}/0.25\text{g glass}$

t_y = $1/2$ life of isotope in years

M = mass number of isotope

The ICP-MS data for the actinides with mass numbers in the 230 to 247 range indicate that measurable levels of the actinides Th-232, U-235, U-236, Np-237, U-238, Pu-239, Pu-240 and Am-241 were detected in the acid dissolved glasses. The acid dissolved glasses were diluted 10X before analyses by ICP-MS. The ICP-MS data for the sodium peroxide fusion dissolved

glasses are not as useful due to the higher dilution (~ 100X) used in running these samples in the ICP-MS. Only Th-232, U-235, Np-237, U-238 and Pu-239 were detectable for these samples.

ICP-MS data for mass-99 indicates that the radioactive glasses, the standard glasses and the reagent blanks all contained measurable levels of mass-99 at slightly above the instrument detection limits that were in the range of 0.001 to 0.009 $\mu\text{g/L}$. The absence of any measurable Tc-99 in the Envelope D glass is expected since no Tc eluates were used in the glass formulation.

The radionuclides measured in this Envelope D, C-106 sludge mixed with secondary wastes HLW glass can be compared to previous vitrification studies involving Hanford HLW glass from SRTC and PNNL that also used HLW sludge mixed with secondary waste streams. Table 9 summarizes the feed streams in Hanford HLW glasses previously made at SRTC⁵ and PNNL²⁸ during earlier Hanford Waste Treatment Plant privatization studies. The HLW glass previously made at SRTC used the pretreated C-106 sludge as feed along with radioactive Sr/TRU precipitate derived from previous Sr/Fe precipitate methodology and concentrated Cs-137 and Tc-99 eluates. The two PNNL HLW glasses were produced in recent Hanford WTP work that was reported in February 2001.²⁸ One of the PNNL HLW glasses was made from Hanford Tank C-104. The other PNNL HLW glass was made from Hanford Tank AZ-102. Table 9 gives pertinent details for the blended streams from all of the HLW glass studies for comparison.

Comparison of the curie loadings in these glasses from Table 9 is shown in Table 10. These data show that the total curie loading in the first three glasses produced were all in the 2,000 to 10,000 $\mu\text{Ci/g}$ range with both Sr-90 and Cs-137 being the predominant radionuclides present. The present study C-106 glass contains an order of magnitude higher level of Cs-137 and thus a glass with total curie loading of about 47,000 $\mu\text{Ci/g}$. This present study C-106 glass also contains the lowest Sr-90 loading due to the use of simulated Sr/TRU material in the glass formulation. This is also the reason why the sum of transuranic radioisotopes measured in the present C-106 glass (Pu-238, Pu-239 + Pu-240, Am-241) are lower than previously measured TRU activities in earlier HLW glasses formulated with actual radioactive Sr/TRU precipitate slurry.

Radiochemical data for the two radioactive feed streams used in the Envelope D glass can be used along with the amounts of these feed streams added, to calculate the expected specific activities in the product glass. Table 11 shows feed data for Cs-137, Sr-90 and total alpha measured in the C-106 sludge analyses and the concentrated Cs eluates (Cs-137 only). The glass formulation used 37.26 grams of dry C-106 sludge powder and ~83 mL of concentrated Cs eluate. Target product glass was 80 grams and a value of 84.7 grams was determined from actual measurements of final glass product mass. All of this data in Table 11 was taken from previous Tables 2, 3 and 6. Table 11 shows the calculated amounts of Cs-137, Sr-90 and total alpha based on these data. The calculated Cs-137 is in the range of $3.12\text{E}+04$ to $3.27\text{E}+04$ $\mu\text{Ci/g}$ glass. The calculated Sr-90 is 532 to 558 $\mu\text{Ci/g}$ glass and total alpha is 4.6 to 4.8 $\mu\text{Ci/g}$ glass. These data are then compared to actual measured values from the dissolved radioactive glasses taken from Table 8. The last three columns in Table 11 show that calculated Cs-137 feed to measured Cs-137 glass ratios are around 85% while the Sr-90 feed/glass ratios are about 130%. Comparing total alpha calculated values in the feed to measured values of actual alpha emitting transuranics (Am-241, Cm-244, Pu-238 and Pu-239+240) gives excellent agreement of 105 to 109%.

Comparison of gross total alpha values from the C-106 sludge to the gross total alpha values measured in the glass indicates only 6% feed/glass ratio.

Radiochemical data measured for the present C-106 glass presented in Table 11 can be used to estimate the total heat loading resulting from loading a HLW glass canister with this glass by using Equation #2 below. One can assume that the nominal HLW glass density is ~ 2.76 g/cc, and a HLW glass cylinder would be nominally 4.5 m height and 0.61 m diameter with 100% glass fill of ~ 1.27 m³.^{2,28}

Equation #2:

Watts/canister

$$\begin{aligned} &= ((\text{Sr-90} + \text{Y-90 Ci/g glass}) \times \text{heat load (W/Ci) for Sr-90/Y-90}) \\ &\quad + ((\text{Cs-137} + \text{Ba-137m Ci/g glass}) \times \text{heat load (W/Ci) for Cs-137/Ba-137m}) \\ &\quad \times \text{grams glass/canister} \end{aligned}$$

With,

Sr-90 Ci/g = Y-90 Ci/g = 413 E-6 Ci/g from Table 8

Cs-137 = 37000 E-6 Ci/g from Table 8

Ba-137m = 0.95 x Cs-137

Heat load for Sr-90/Y-90 = 0.00691 W/Ci

Heat load for Cs-137/Ba-137m = 0.00445 W/Ci

Watts/canister =

$$\begin{aligned} &((2 \times 413\text{E-6 Ci/g}) \times 0.00691 \text{ W/Ci}) + \\ &\quad ((37000\text{E-6 Ci/g} + 0.95 \times 37000\text{E-6 Ci/g}) \times 0.00445 \text{ W/Ci}) \\ &\quad \times 2.76 \text{ g/cm}^3 \times (100 \text{ cm/1m})^3 \times 1.27 \text{ m}^3/\text{canister} \end{aligned}$$

$$\begin{aligned} \text{Watts/canister} &= [(8.26\text{E-04 Ci/g} \times 0.00691 \text{ W/Ci}) + (7.21\text{E-02 Ci/g} \times 0.00445 \text{ W/Ci})] \\ &\quad \times 3.50\text{E+06 g/canister} \end{aligned}$$

$$\text{Watts/canister} = [(5.71\text{E-6 W/g}) + (3.21\text{E-04 W/g})] \times 3.50\text{E+06 g/canister}$$

$$\text{Watts/canister} = 1,145 \text{ W/canister}$$

The HLW glass product specifications for the RPP-WTP specify that the heat loading on any single canister is limited to 1500 W/canister and an average canister loading of 300 W/canister.² The estimated heat loading calculated above for the C-106 glass shows the total heat loading due to Sr-90/Y-90 and Cs-137/Ba-137m (1,145 W/canister) to be less than the upper limit of 1,500 Watts/canister. The radioactive Sr-90 measured in the C-106 glass derived exclusively from the radioactive C-106 sludge feed since the present vitrification study used simulated Sr/TRU feed stream.

One can use the actual values for measured Cs-137 and Sr-90 in the radioactive Sr/TRU slurry to estimate how much Cs-137 and Sr-90 would have been added to the glass had the actual radioactive Sr/TRU slurry been used. From earlier Table 4 data, the total Cs-137 possible from radioactive Sr/TRU would be $\sim 13 \mu\text{Ci/mL} \times 150 \text{ mL} = 1,950 \mu\text{Ci}$ of Cs-137. These 1,950 μCi of Cs-137 would have yielded an additional $1.95 \text{ E-3 Ci} / 84.7 \text{ g glass} = 23 \text{ E-6 Ci of Cs-137/g glass}$. Similarly, from Table 4 data, the total Sr-90 possible from radioactive Sr/TRU would be $\sim 106 \mu\text{Ci/mL} \times 150 \text{ mL} = 15,900 \mu\text{Ci}$ of Sr-90. These 15,900 μCi of Sr-90 would have yielded an additional $1.59 \text{ E-2 Ci} / 84.7 \text{ g glass} = 1.88 \text{ E-4 Ci of Sr-90/g glass}$. One can add these amounts to the actual measured values in the C-106 HLW glass and recalculate (using the previous Equation #2) the expected heat loading of the glass if all of the radioactive Sr/TRU had been used. This calculation shown below shows that the estimated canister heat loading would only be 1,155 W/canister vs. the previously calculated value (based on actual glass measurements) of 1,145 W/canister.

Equation #2:

Watts/canister (if radioactive Sr/TRU had been added)

$$= ((\text{Sr-90} + \text{Y-90 Ci/g glass}) \times \text{heat load (W/Ci) for Sr-90/Y-90}) \\ + ((\text{Cs-137} + \text{Ba-137m Ci/g glass}) \times \text{heat load (W/Ci) for Cs-137/Ba-137m}) \\ \times \text{grams glass/canister}$$

With,

Sr-90 Ci/g = Y-90 Ci/g = 413 E-6 Ci/g from Table 8 plus 188 E-6 Ci/g from Sr/TRU = $601 \text{ E-6 Ci/g total}$
Cs-137 = 37000 E-6 Ci/g from Table 8 plus 23 E-6 Ci/g from Sr/TRU = 37023 Ci/g total
Ba-137m = $0.95 \times \text{Cs-137}$

Heat load for Sr-90/Y-90 = 0.00691 W/Ci

Heat load for Cs-137/Ba-137m = 0.00445 W/Ci

Watts/canister =

$$((2 \times 601 \text{ E-6 Ci/g}) \times 0.00691 \text{ W/Ci}) + \\ ((37023 \text{ E-6 Ci/g} + 0.95 \times 37023 \text{ E-6 Ci/g}) \times 0.00445 \text{ W/Ci}) \\ \times 2.76 \text{ g/cm}^3 \times (100 \text{ cm/1m})^3 \times 1.27 \text{ m}^3/\text{canister}$$

$$\text{Watts/canister} = [(1.20 \text{ E-03 Ci/g} \times 0.00691 \text{ W/Ci}) + (7.22 \text{ E-02 Ci/g} \times 0.00445 \text{ W/Ci})] \\ \times 3.50 \text{ E+06 g/canister}$$

$$\text{Watts/canister} = [(8.30 \text{ E-6 W/g}) + (3.21 \text{ E-04 W/g})] \times 3.50 \text{ E+06 g/canister}$$

$$\text{Watts/canister} = 1,155 \text{ W/canister}$$

D. Product Consistency Test

The durability of the HLW C-106 glass was measured using the ASTM C-1285 standard leach test referred to as the Product Consistency Test (PCT). This test is required by the RPP-WTP contract as specified by the WAPS. Information on the PCT, the results and the measured leachate data are all presented in Appendix H of this document. The PCT results indicate that normalized released B, Si and Na were well below those for the Environmental Assessment (EA) glass even after two standard deviations are subtracted from the average for the normalized EA glass.

E. Toxicity Characteristic Leaching Procedure

The Hanford Site Tank C-106 radioactive waste is a 'listed' Mixed Waste and thus the immobilized HLW glass must be considered to be listed Mixed Waste. The toxicity characteristic leaching procedure, or TCLP, is required per the RPP-WTP Contract in the Standard 2, Research, Technology and Modeling section relating to Immobilized High-Level Waste Qualification Testing. The TCLP test was carried out at SRTC on the C-106 HLW glass using the EPA SW-846 procedure Method 1311 with some modifications. Information on the TCLP, modifications and the test results are all presented in Appendix I of this document. TCLP testing and analysis on the C-106 HLW glass proved that all RCRA metal concentrations in the TCLP extract were below the Universal Treatment Standard (UTS) limits.

F. Microscopy and Crystallinity Phase Determination

The RPP-WTP Contract Specifications for HLW glass reference the WAPS, which refer to the requirement of identifying crystalline phases expected to be present and projection of the amount of each crystalline phase expected to be present in each waste type. Powered glass samples were analyzed by XRD-spectroscopy for crystalline phase identification and by Energy Dispersive X-ray Analysis to determine the qualitative identification of crystals and estimate the quantitative amount present in the C-106 HLW glass. These analyses and results are presented in Appendix J of this document. Results show that trevorite (NiFe_2O_4) is present in the C-106 HLW glass at estimated 1.5 %.

V. Conclusion/Summary

A. Glass Chemical Analyses

The RPP-WTP, D Envelope flow sheet as it existed during the approximate timeframe of calendar years 2000 through early 2001 was carried out in the SRTC shielded cells with samples of the Hanford Site radioactive waste streams from Envelopes A, B, C, and D. The amount of

glass produced agreed well with the predicted, calculated value. The chemical composition of the Envelope D glass was very similar to the calculated composition supplied by VSL. The chemical composition for the glass was found to meet the RPP-WTP specifications under the criteria involving minimal iron loading from the pretreated C-106 sludge.

B. Glass Radiochemical Analyses

Radiochemical analyses on the dissolved Envelope D glass show that as expected, Cs-137 is the predominant radionuclide present in the glass from the concentrated Cs eluates. Strontium-90 and various transuranic radionuclides were also measured in the glass at levels expected from the radioactive C-106 sludge waste feed stream. Calculations estimating the expected heat loading of this Envelope D glass indicate the total heat loading to be below the specified 1,500 Watt/canister limit. Comparison of the present Envelope D, C-106 glass with previous HLW glasses vitrified in the WTP research studies indicates that this glass contains ~ 10X higher loading of Cs-137 and contains relatively lower levels of Sr-90 and transuranics due to use of a nonradioactive simulated Sr/TRU manganese precipitate.

C. Product Consistency Test

The ASTM standard Product Consistency Test (PCT) was performed at 90°C on the C-106 radioactive glass and the Environmental Assessment (EA) glass. The PCT results indicate that normalized released for B, Si, and Na were well below those for the EA glass even after two standard deviations are subtracted from the average for the EA glass.

D. Toxicity Characteristic Leaching Procedure

The TCLP was performed according to the standard EPA SW-846 procedure (Method 1311) with certain modifications as discussed in Appendix G. TCLP analysis was used successfully to characterize the Tank C-106 glass waste to provide the following information:

- Concentrations of all target analytes to below the maximum allowable lower limit of detection.
- Proof that all RCRA metal concentrations were below the UTS limits in the TCLP extract.
- Results for metal analytes were similar regardless of whether an acid-digestion was performed.
- Low extraction temperatures caused 4 analytes to be biased slightly low in the TCLP standard.
- Recoveries were greater than 50 % for all target analytes and greater than 75 % for most.

E. Crystalline Phase Identification

XRD data obtained from analyses of the powdered C-106 HLW glass produced in this study indicate that trevorite (NiFe_2O_4), a crystalline phase of the spinel family of crystals ($(\text{Fe},\text{Ni},\text{Mn})(\text{Fe},\text{Cr})_2\text{O}_4$) is present in the glass. Energy Dispersive X-ray Analyses (EDAX)

supports the identification of these crystals to contain the elements Fe and Ni. Calculations involving the surface area of these crystals on a SEM image vs. the surface area of the background amorphous glass in the same SEM image indicate about 1.5 % crystallinity in the glass.

VI. Controls and Quality Assurance

This quality assurance program for this task was in compliance with the applicable elements of the DOE/RW/0333P "Quality Assurance Requirements and Description" for work affecting the immobilized high-level waste form development, qualification, characterization, and certification.

All work was conducted under written procedures and/or written directions. Major equipment was controlled under SRTC's M&TE controls and/or calibrated prior to use. Measurement readings, data log sheets, and associated task information was maintained in SRTC laboratory notebooks.^{15,16,17} The analyses were carried out under routine QA and QC as employed by the ADS. This included use of analytic standards, calibration of analytical instrumentation. All training records for personnel were maintained as required. QA audits were carried out and documented in the laboratory notebooks.

VII. Acknowledgements

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VIII. Tables

Table 1. Required Analytical Support

Table 2. C-106 Sludge Supernate and Sludge Solids – Chemical Analysis Results

Table 3. Small A,B,C, and Large C Eluate Evaporation – Chemical Analyses, Concentrate and Condensate II

Table 4. Radioactive and Simulated Strontium/TRU Material

Table 5. Glass Former Mixture

Table 6. Envelope D Waste Streams, Glass Formers and Calculated Glass Composition

Table 7. Envelope D, Glass Weights and Accountability

Table 8. Measured D Glass Chemical Composition from Peroxide Fusion & Acid Dissolution

Table 9. High-Level Waste Glasses Mixed with Secondary Products Comparison

Table 10. High-Level Waste Glass Mixed with Secondary Wastes Radionuclide Comparison

Table 11. Comparison of Expected Specific Activities in the Glass to Measured Values

Table 1. Required Analytical Support

Technique	Characterize Sludge C-106 & Supernate	Characterize Conc. & Cond.	Preparation Glass Formers	PCT / TCLP	Glass Analysis
Sample Digestion	Aqua Regia			TCLP	
Na ₂ O ₂ Fusion	X		X		X
HNO ₃ Dissolution			X		X
Analyses					
ICP-ES	X	X	X	PCT/TCLP	X
AA (Na/K)	X	X		PCT	X
CV-AA (Hg)				TCLP	
AA (Se/As)				TCLP	
ICP-MS	X >84				Actinides and M>84
IC	X	X			
Total alpha/beta					X
Gamma-PHA	X	X			X
Am-241/Cm-244					X
Pu separation and counting					X
Sr-90	X				X
Tc-99 (ICP-MS)					X
TIC/TOC	X	X			
XRD					X
SEM/EDAX					X

Table 2. C-106 Sludge Supernate and Sludge Solids – Chemical Analysis Results

Analyte	Sludge Supernate (Average of Three) (mg/L)	Sludge Solids (Average) (Element %)	
Ag	1.460	0.033	
Al	4.631	7.611	
B	<0.385	N.D.	
Ba	<0.152	0.065	
Ca	<0.076	0.636	
Cd	<0.228	0.013	
Co	<0.380	0.007	
Cr	1.458	0.094	
Cu	0.637	0.022	
Fe	0.731	19.144	
La	<0.836	0.022	
Li	0.211	0.003	
Mg	0.945	0.139	
Mn	<0.076	0.399	
Mo	<0.474	0.007	
Na	5562	8.998	
Ni	1.535	0.200	
P	227.4	0.255	
Pb	2.451	0.411	
Si	5.437	9.99	
Sn	<1.141	N.D.	
Sr	<0.173	0.007	
Ti	<0.173	0.157	
V	0.454	0.004	
Zn	0.917	0.014	
Zr	0.583	0.079	
K	3.918	0.212	
NO3-	45.62		
NO2-	98.84		
PO4	<76.03		
SO4	22.81		
Formate	129.25		
Oxalate	6839.1		
Cl /IC	4.058		
F / IC	<15.207		
TIC (Blnks<0.5)	293.70		
TOC (Blnks<5.3)	3371.0		
	dpm/mL	μCi/mL	μCi/g
Alpha	4.45 E+04	2.01 E-02	1.04 E+01
Beta	1.35 E+06	6.10 E-01	2.98 E+03
Cs-137		4.58 E-01	5.83 E+02
Sr-90			1.21 E+03
Total Ci		1.1 E-03	6.36E-01
Total Na (grams)		5.56	12.1
Total Quantity	~1000 mL		134.6 g

**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

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Table 3. Small A,B,C, and Large C Eluate Evaporation – Chemical Analyses, Concentrate and Condensate II

Cs Concentrate II (Average of Two)		Cs Condensate II (Average of Two)		Conc./Cond.
Analyte	mg/L		mg/L	Factor
Al	64.6		0.016	4,169
B	4.4		0.009	520
Ba	12.2	<	0.002	
Ca	< 0.4	<	0.001	
Cd	36.4	<	0.003	
Co	2.7	<	0.005	
Cr	248.1	<	0.007	
Cu	310.9	<	0.002	
Fe	36.2		0.006	6,582
La	< 4.6	<	0.011	
Li	< 0.8	<	0.002	
Mg	3.8	<	0.001	
Mn	44.4	<	0.001	
Mo	< 2.5	<	0.006	
Na	32,353		0.059	548,361
Ni	983.9	<	0.007	
P	< 10.9	<	0.026	
Pb	312.5	<	0.028	
Si	39.6		0.245	162
Sn	< 6.3	<	0.015	
Sr	21.5	<	0.001	
Ti	< 0.8	<	0.002	
V	< 1.5	<	0.003	
Zn	10.7	<	0.003	
Zr	< 1.7	<	0.004	
U				
K				
Total(mg/L)	34,515		0.463	
Total(g/L)	34.51		0.000463	
Analyte	mg/L		mg/L	Conc./Cond. Factor
NO3/IC	241,403		94.5	2,555
NO2/IC	273.60		5	55
PO4/IC	< 420.93	<	1	
SO4/IC	420.93		0.7	601
Form/IC	420.93		17	25
Oxalate/IC	126.28	<	1	
Cl/IC	420.93		2	210
F/IC	84.19		0.3	281
	$\mu\text{Ci/mL}$		$\mu\text{Ci/mL}$	
Cs-137	3.15E+04		8.21E-03	3.8E+06

**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

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Table 4. Radioactive and Simulated Strontium/TRU Material

Element	Sr-TRU Stream (g/L)	Oxide	Per VSL – W.Kot] **		Simulated Sr/TRU (g)	[Sr/TRU Simulant Recipe]		
			Convert Oxide Wt (g/L)	X 0.15 L Oxide Wt (g)		X 0.15L Ox. Wt (g)	Chemical Reagent (g)	Mixture Weight (g)
Ag	0.000	Ag ₂ O	0.000	0.000				
Al	1.037	Al ₂ O ₃	1.959	0.294*	1.960	0.294*	Al ₂ O ₃	1.96
B	0.006	B ₂ O ₃	0.019	0.003				
Ba	0.026	BaO	0.029	0.004*	0.029	0.004*	BaCO ₃	0.04
Ca	0.717	CaO	1.003	0.150*	1.003	0.150*	CaCO ₃	1.79
Cd	0.003	CdO	0.003	0.001				
Co	0.002	CoO	0.003	0.000				
Cr	0.303	Cr ₂ O ₃	0.443	0.066*	0.443	0.066*	Cr ₂ O ₃	0.89
Cu	0.004	CuO	0.005	0.001				
Fe	0.121	Fe ₂ O ₃	0.173	0.026*	0.173	0.026*	Fe ₂ O ₃	0.17
K	N.A.	K ₂ O	N.A.	N.A.				
La	0.048	La ₂ O ₃	0.056	0.008				
Li	0.001	Li ₂ O	0.002	0.000				
Mg	0.001	MgO	0.002	0.000				
Mn	13.728	MnO	17.723	2.659*	17.720	2.658*	MnO ₂	21.71
Mo	0.002	MoO ₂	0.003	0.000				
Na	10.461	Na ₂ O	14.103	2.116*	14.100	2.115*	Na ₂ CO ₃	23.23
Nd	N.A.	Nd ₂ O ₃	N.A.	N.A.				
Ni	0.073	NiO	0.093	0.014*	0.090	0.014*	NiO	0.09
P	0.259	P ₂ O ₅	0.594	0.089*	0.590	0.089*	Na ₂ HPO ₄	2.24
Pb	0.140	PbO	0.151	0.023				
Si	3.681	SiO ₂	7.870	1.181*	7.870	1.181*	SiO ₂	7.87
Sr	30.306	SrO	35.839	5.376*	35.840	5.376*	Sr(NO ₃)	73.19
Ti	0.001	TiO ₂	0.002	0.000				
U	N.A.	U ₃ O ₈	N.A.	N.A.				
V	0.001	V ₂ O ₅	0.002	0.000				
Zn	0.019	ZnO	0.024	0.004				
Zr	0.023	ZrO ₂	0.031	0.005*	0.030	0.005*	ZrO ₂	0.03
60.963 Sum			80.132	12.020	79.848	11.9772		133.21

Cs-137 = 13 uCi/mL

Sr-90 = 106 uCi/mL

* Agreement with VSL and
Pretreatment Measurements

** See **Appendix A**

133.21 x 0.15= 19.980

(Amount Added to Crucible)

WSRC-NB-2000-160-35

Sr/TRU Simulant

Table 5. Glass Former Mixture

Based on 100 grams of glass Additives from Wing Kot - VSL			Based on 81 grams of glass vitrification Glass Former Mixture (NB-2000-160-20)				
Glass (Wt%)	Oxide (Wt.%)		Glass Former Material *	Weight (grams)	Weight factor	Oxide Weight (grams)	Oxide (Wt.%)
B2O3	4.99	11.47	Boric Acid	7.131	0.5652	4.03	11.47
Li2O	4.50	10.35	Lithium Carbonate	9.062	0.4011	3.63	10.35
SiO2	34.00	78.18	SCS-75	27.544	0.997	27.46	78.18
Total	43.49	100.00		43.737		35.13	100.00
ADS Analyses							
LIMS No.	158192	158192	158192	AVG.			
Element mg/g	mg/g	mg/g	mg/g	(3)			
B	26.20	25.50	28.00	26.57			
Li	35.10	35.80	34.60	35.17			
Si	272.00	264.00	281.00	272.33			
ADS Analyses to Oxide Percent							
Oxide	Factor Ox./Ele.	Ele.Avg. mg/g	Oxide mg/g	Oxide Wt.%			
B2O3	3.218	26.57	85.49	11.50			
Li2O	2.153	35.17	75.70	10.18			
SiO2	2.138	272.33	582.25	78.32			
			743.45	100.00			
* Glass Formers:							
Boric Acid			US Borax, Technical Granular				
Lithium Carbonate			Cyprus-Foote, Crystal				
Silica			US Silica, SCS 75, Mill Creek				

* See **Appendix B**

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0

Table 6. Envelope D Waste Streams, Glass Formers and Calculated Glass Composition

Oxide	Sim. Si-TRU Oxide Wt Calc. Value	g/L	x0.15L	Ele Wt% Analysis	g/100g	Sludge C-106 Dry Oxide Wt		Oxide Wt x37.256 g	C s Ele Wt g/L	1.155g/l Vol=79ml x0.079		Sum Radioactive Components	Oxide wt%	GFC Oxide grams	Oxide	GFC Additives GI Form.	Weight	Glass Wt grams	GLASS		
						Oxide Wt g/100g	Oxide Wt g/L			Ele Wt g	Conc. II Ele Wt								Oxide Wt gr	Glass Comp..	Kot Comp.
						g/100g	g/L			g	g/L								gr	Comp..	Comp.
Ag2O	0	0	0	0.033	0.035	0.013	0	0	0	0	0.013	0.03%			AgO			0.013	0.02%	0.03%	
Al2O3	1.96	0.294	0	7.611	14.379	5.357	0.065	0.005	0.01	0.001	5.661	12.40%			Al2O3			5.661	7.06%	6.97%	
B2O3	0	0	0	0	0	0	0.004	0	0.001	0	0.001	0.00%	4.03		B2O3	BoricAc.	7.131	0.032	5.03%	5.00%	
BaO	0.029	0.004	0	0.065	0.073	0.027	0.012	0.001	0.001	0.001	0.032	0.07%			BaO			0.032	0.04%	0.04%	
CaO	1.003	0.15	0	0.636	0.89	0.332	0	0	0	0	0.482	1.06%			CaO			0.482	0.60%	0.59%	
CdO	0	0	0	0.013	0.015	0.006	0.036	0.003	0.003	0.003	0.009	0.02%			CdO			0.009	0.01%	0.01%	
CoO	0	0	0	0.007	0.009	0.003	0.003	0	0	0	0.004	0.01%			CoO			0.004	0.00%	0.00%	
Cr2O3	0.443	0.066	0	0.094	0.137	0.051	0.248	0.02	0.029	0.029	0.146	0.32%			Cr2O3			0.146	0.18%	0.19%	
CuO	0	0	0	0.022	0.028	0.01	0.311	0.025	0.031	0.031	0.041	0.09%			CuO			0.041	0.05%	0.06%	
Fe2O3	0.173	0.026	0	19.144	27.367	10.196	0.036	0.003	0.004	0.003	10.226	22.41%			Fe2O3			10.226	12.76%	12.58%	
K2O	0	0	0	0.212	0.255	0.095	0	0	0	0	0.095	0.21%			K2O			0.095	0.12%	0.12%	
La2O3	0	0	0	0.022	0.026	0.01	0	0	0	0	0.01	0.02%			La2O3			0.01	0.01%	0.02%	
Li2O	0	0	0	0.003	0.006	0.002	0	0	0	0	0.002	0.01%	3.63		Li2O	LiCarb	9.062	3.637	4.54%	4.50%	
MgO	0	0	0	0.139	0.23	0.086	0.004	0	0	0	0.086	0.19%			MgO			0.086	0.11%	0.11%	
MnO	17.72	2.658	0	0.399	0.515	0.192	0.044	0.004	0.005	0.004	2.854	6.25%			MnO			2.854	3.56%	3.53%	
MoO2	0	0	0	0.007	0.009	0.003	0.003	0	0	0	0.004	0.01%			MoO2			0.004	0.01%	0.01%	
Na2O	14.1	2.115	0	8.998	12.131	4.52	32.363	2.556	3.446	0	10.08	22.09%			Na2O			10.08	12.57%	13.11%	
Nd2O3	0	0	0	0	0	0	0	0	0	0	0	0.00%			Nd2O3			0	0.00%	0.00%	
NiO	0.09	0.014	0	0.2	0.254	0.095	0.984	0.078	0.099	0	0.207	0.45%			NiO			0.207	0.26%	0.28%	
P2O5	0.59	0.089	0	0.255	0.584	0.218	0	0	0	0	0.306	0.67%			P2O5			0.306	0.36%	0.36%	
PbO	0	0	0	0.411	0.443	0.165	0.313	0.025	0.027	0.027	0.192	0.42%			PbO			0.192	0.24%	0.27%	
SiO2	7.87	1.181	0	9.99	21.36	7.958	0.04	0.003	0.007	0.003	9.145	20.04%	27.46		SiO2	SCS75	27.544	36.607	45.66%	45.26%	
SiO	35.84	5.376	0	0.007	0.008	0.003	0.021	0.002	0.002	0	5.381	11.79%			SiO			5.381	6.66%	6.66%	
TiO2	0	0	0	0.157	0.262	0.098	0	0	0	0	0.098	0.21%			TiO2			0.098	0.12%	0.12%	
U3O8	0	0	0	0	0	0	0	0	0	0	0	0.00%			U3O8			0	0.00%	0.00%	
V2O5	0	0	0	0.004	0.007	0.003	0	0	0	0	0.003	0.01%			V2O5			0.003	0.00%	0.00%	
ZnO	0	0	0	0.014	0.017	0.006	0.011	0.001	0.001	0.001	0.008	0.02%			ZnO			0.008	0.01%	0.01%	
ZrO2	0.03	0.005	0	0.079	0.107	0.04	0.002	0	0	0	0.044	0.10%			ZrO2			0.044	0.06%	0.05%	
SO3/SO4	0	0	0	0	0	0	0.421	0.033	0.028	0.033	0.028	0.06%			SO3/SO4			0.028	0.04%	0.04%	
Cl	0	0	0	0	0	0	0.421	0.033	0.033	0.033	0.033	0.07%			Cl			0.033	0.04%	0.05%	
F	0	0	0	0	0	0	0.084	0.007	0.007	0.007	0.007	0.01%			F			0.007	0.01%	0.01%	
NO3							[241.403]	[19.071]	[19.071]	[19.071]	[19.071]	[41.79]			[NO3]			[19.071]	[23.789%]		
Sum	79.848	11.977	48.522	79.149	29.488	35.415	2.798	3.733	45.198	99.00%	35.127	Sum			Sum			43.737	80.325	100.19%	
Oxide Wt. Kot	12.02				29.3													43.49	80.77	From Kot	
Add. Wt. Crucible	20.014				37.256													43.716	84.753	Actual Measured	
Curies (Ci)	0				0.18				2.49												
Ref. Ele-Ox																				2.67Ci	

Table 7. Envelope D, Glass Weights and Accountability

Actual Measurements From Notebook		Split Sample for TCLP		Samples for PCT		Samples for Dissolutions	
Remove Glass from Crucible 2/13/01		5.0 +/- 0.10 gram		Ref. 2001-00031+25		RFS Diss.	
Glass + Plastic Bag	grams	TCLP-1	29.875	Glass in Jar After PCT PCT Samples	NEB PCT	Glass in Vial Vial Wt. Glass to Grinder 2.131	<200M PF
Plastic Bag	21.307	Bottle + Glass	24.963				
Glass Weight	84.643	Glass Weight	4.912				
(Rough Measurement)							
Glass Jar + Glass	197.114	TCLP-2	29.826	PCT Samples	16.352	AD	0.238
Glass Jar	112.323	Bottle Weight	24.928				
Glass Weight	84.791	Glass Weight	4.898				
Archive Sample		TCLP-3	30.125	Crushed by Hammer D Env. Glass in Jar Jar Wt + Cap etc. Glass Sample	15.06	PF	0.245
D Env. Glass in Jar	155.067	Bottle + Glass	25.137				
Jar Wt.+Cap etc.	125.737	Glass Weight	4.988				
Glass Wt(Archive)	29.33						
Crushed by Hammer		TCLP-4	29.954	Crushed by Hammer Glass Sample	15.06	PF	0.252
D Env. Glass in Jar	178.975	Bottle Weight	25.004				
Jar Wt + Cap etc.	125.398	Glass Weight	4.95				
Glass Sample	53.577	TCLP Samples	19.748				
Spilled in Pan	1.884	Sample Jar	158.894	Crushed by Hammer D Env. Glass in Jar Jar Wt + Cap etc. Glass Sample	17.158	PF	0.994
		D Env. Glass in Jar	125.398				
		Jar Wt + Cap etc.	33.496				
		Glass Sample	33.496				
Total:	84.791	Total Glass	53.244	Total Glass	33.510		

Table 8. Measured D Glass Chemical Composition from Peroxide Fusion & Acid Dissolution

Note: The analytes Cl, F and S were not measured in the dissolved glasses and are thus not included in this table.

Element	PerFus Average(3) Ele Wt%	Acid Dis. Average(2) Ele Wt%	Best Combined Analysis (or average) EleWt%	Oxide	As Analyzed Oxide Wt%	VSL Glass Comp. Oxide Wt%
Ag	N.D.	0.019	0.019	AgO	0.022	0.02
Al	3.728	3.828	3.778	Al ₂ O ₃	7.137	6.97
B	1.353	N.D.	1.353	B ₂ O ₃	4.354	5.00
Ba	0.038	0.055	0.046	BaO	0.051	0.04
Ca	0.571	0.442	0.507	CaO	0.709	0.59
Cd	0.024	0.013	0.018	CdO	0.021	0.01
Co	0.143	0.052	0.097	CoO	0.123	0.00
Cr	0.189	0.199	0.194	Cr ₂ O ₃	0.284	0.19
Cu	0.162	0.203	0.182	CuO	0.228	0.06
Fe	* 9.008	* 8.791	8.900	Fe ₂ O ₃	12.722	12.58
K	0.322	0.235	0.264	K ₂ O	0.318	0.12
La	< 0.033	< 0.03	< 0.03	La ₂ O ₃	< 0.035	0.02
Li	1.841	1.962	1.901	Li ₂ O	4.092	4.50
Mg	0.092	0.089	0.090	MgO	0.149	0.11
Mn	2.651	2.593	2.622	MnO	3.385	3.53
Mo	0.007	< 0.009	0.007	MoO ₂	< 0.009	0.01
Na	N.D.	9.382	9.382	Na ₂ O	12.649	13.11
Ni	N.D.	0.231	0.231	NiO	0.294	0.28
P	0.237	0.293	0.265	P ₂ O ₅	0.607	0.38
Pb	0.323	0.308	0.315	PbO	0.339	0.27
Si	* 18.402	* 22.991	20.697	SiO ₂	44.250	45.26
Sn	< 0.133	0.05	0.050	SnO	0.030	0.00
Sr	4.890	5.519	5.204	SrO	6.154	6.66
Ti	0.116	0.093	0.104	TiO ₂	0.173	0.12
V	0.008	0.017	0.012	V ₂ O ₅	0.021	0.00
Zn	0.430	0.035	0.233	ZnO	0.289	0.01
Zr	0.339	0.409	0.374	ZrO ₂	0.505	0.05
Sum			56.844	Sum	98.95	99.89
Isotope	uCi/g	uCi/g	uCi/g			
Am-241	3.25E-01	1.65E+00	9.88E-01			
Cm-244	6.42E-02	4.90E-01	2.77E-01			
Pu-238	7.78E-01	5.76E-01	6.77E-01			
Pu-239-40	2.14E+00	1.67E+00	1.91E+00			
Pu-241	4.11E+00	3.33E+00	3.72E+00			
Sr-90	4.06E+02	4.20E+02	4.13E+02			
Alpha	3.77E+01	1.17E+02	7.74E+01			
Beta	3.10E+04	4.73E+04	3.92E+04			
Cs-137	3.49E+04	3.91E+04	3.70E+04			
Total uCi/g	3.53E+04	3.95E+04	3.74E+04			
Glass Wt. (g)	84.8	84.8	84.8			
Total (Ci)	2.99E+00	3.35E+00	3.17E+00			

Ref. Ele toWt%Dglass.xls

* PerFus Fe and Acid Dis Fe Bias correct per Appendix F and G, respectively.

* PerFus Si and Acid Dis Si Bias correct per Appendix F and G, respectively.

Table 9. High-Level Waste Glasses Mixed with Secondary Products Comparison

SRTC C-106 HLW Glass (Jan. 1998)^a	PNNL C-104 HLW Glass (Feb. 2001)^b	PNNL AZ-102 HLW Glass (Feb. 2001)^b	SRTC C-106 HLW Glass
C-106 pretreated sludge solids, filtered and air- dried 58.5 g	C-104 pretreated sludge slurry (20 wt% solids) waste 400.5 g	AZ-102 pretreated sludge slurry (9.5 wt% solids) waste 499.02 g	C-106 pretreated sludge solids, filtered and oven- dried 37.3 g
Radioactive AN-102 Sr/TRU precipitate (Sr/Fe process) 5.8 g	Radioactive AN-107 Sr/TRU precipitate 25.55 g	Simulant AN-107 Sr/TRU precipitate 14.37 g	Simulant AN-102 Sr/TRU precipitate (Sr/Mn process) 19.98 g
Cs and Tc eluate concentrates (AW-101, AN-102), ~ 50 mL	Cs eluate composite (AW- 101, AN-107), 529.0 g	High nitrate Tc eluate, 11.37 g Low nitrate Tc eluate, 69.38 g Cs eluate composite (AW- 101, AN-107), 157.29 g	Cs eluate concentrate (AN-103, AZ-102, AN- 102), ~ 100 mL
Glass Formers: Boric acid, calcium hydroxide, lithium hydroxide and silica sand, 24.7 g batch	Glass Formers: borax, sodium carbonate, lithium hydroxide, silica sand and zinc oxide, 219.53 g batch	Glass Formers: borax, sodium carbonate, sodium metasilicate, lithium hydroxide and silica sand, 111.36 g batch	Glass Formers: Boric acid, lithium carbonate and silica sand, 43.7 g batch
<u>60 gram</u> product glass, Formulation ^c	<u>239.84 gram</u> product glass, Formulation HLW98-51R	<u>115.07 gram</u> product glass, Formulation HLW98-61	<u>80 gram</u> product glass, Formulation HLW98-67

a) See Reference 5-d.

b) See Reference 28.

c) See Reference ²⁹.

Table 10. High-Level Waste Glass Mixed with Secondary Wastes Radionuclide Comparison

Radionuclide	SRTC C-106 HLW Glass (Jan. 1998)^a	PNNL C-104 HLW Glass (Feb. 2001)^b	PNNL AZ-102 HLW Glass (Feb. 2001)^b	SRTC C-106 HLW Glass (this study)*
	($\mu\text{Ci/g glass}$)	($\mu\text{Ci/g glass}$)	($\mu\text{Ci/g glass}$)	($\mu\text{Ci/g glass}$)
Sr-90	1150	519	8900	413
Cs-137	2000	1160	705	37100
Pu-239 + Pu-240	3.2	3.3	3.4	1.7
Pu-238	Not Reported	0.4	0.4	0.6
Am-241	4.2	4.8	71.5	1.6
Co-60	Not Reported	0.1	2.7	< (10 - 30)
Sb-125	Not Reported	<3	20.9	< (250-575)
Eu-154	Not Reported	2.7	26.2	< (30-100)
Eu-155	Not Reported	1.3	46.6	< (100-250)
Sum	3,157.4	1,694.7	9,776.7	47,293.6**

- a) “Vitrification of Four Radioactive Hanford Waste Samples”, D.M. Ferrara, C.L. Crawford, B. C. Ha, N. E. Bibler and A. S. Choi, **SRTC-BNFL-023, Rev. 2, Jan. 5, 1998** and “Hanford High Level Waste Glass – Additional Results”, N. E. Bibler, D. M. Ferrara and C. L. Crawford, **SRTC-BNFL-026, Rev. 0, Jan. 19, 1998**.
- b) “Vitrification and Product Testing of C-104 and AZ-102 Pretreated Sludge Mixed with Flowsheet Quantities of Secondary Wastes”, G. L. Smith et al., **PNNL-13452, WTP-RPT-006, Rev. 0, Formerly BNFL-RPT-042, Feb. 2001**.

*Sr-90 by separation & beta counting, Cs-137 by gamma spectroscopy, Pu isotopes by separation and counting, Am-241 by separation and counting, Co-60, Sb-125, Eu-154/155 from minimal detectable activity values from gamma spectroscopy.

** Total not inclusive of Co/Sb/Eu minimal detection limits.

Table 11. Comparison of Expected Specific Activities in the Glass to Measured Values

						Feed	Feed	Measured				
	C-106 Sludge	Amount Added	Cs Eluate	Amount Added*	Glass Amount	μCi/g (@80g)	μCi/g (@84.7g)	in Glass Cs-137				
	(μCi/g)	(g)	(μCi/mL)	(mL)	(g)			(μCi/g)		Feed/Glass		Feed/ Glass
Cs-137	5.83E+02	37.26	3.15E+04	83	80.77	3.27E+04		3.74E+04		0.87**		
					84.753		3.12E+04			0.83***		
								Sr-90 (μCi/g)				
Sr-90	1.21E+03	37.26	(#)	83	80.77	5.58E+02		4.13E+02				1.35**
					84.753		5.32E+02					1.29***
								Sum Am/Pu/Cm (μCi/g)	Total Alpha (μCi/g)	Feed/Glass Using Am/Pu/Cm	Feed/Glass Using Total Alpha	
Total Alpha	10.4	37.26	(#)		80.77	4.8		4.39	77.35 ^(##)	1.09**	0.06**	
					84.753		4.57			1.05***	0.06***	

*The 91.5 g of concentrated cesium eluate assumed to have nominal density of 1.1 g/cc for ~ 3M nitric acid solution. Trace Cs-137 added from the 100 mL of 1/500 dilution Cs eluate rinse (see text) also included in calculations

** Using target 80 gram basis.

*** Using measured 84.7 gram basis.

(#) Sr-90 and Total Alpha were not analyzed for in the concentrated Cs eluates.

(##) This value for total alpha measured in glass had low precision with a range of 37 to 117 μCi/g.

IX. Appendices

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Figure 1. Spreadsheet for Envelope D Glass Formulation Calculation

Figure 1. Spreadsheet for Envelope D Glass Formulation Calculation

Assume Density
of Concentrate = 1 g/ml
(Components below Detection
Limits NOT included)

Appendix A. Figure 2. Email from VSL Explanation of the Envelope D Glass Formulation

All,

VSL has completed formulating and testing glasses for the C-106 sludge, which we discussed during a teleconference in 11/00. The attached spreadsheet contains information on both waste blending and glass composition. The blending of wastes is such that all the available Sr/TRU products and Cs-eluate concentrate will be used up (to produce about 81 g of glass). The resulting Sr/TRU products blending ratio is slightly higher than that found in C-106/AY-102 melter tests at VSL, but for the case of Cs-eluate, it is lower.

Please note that there is a 10%-15% difference between the analytical data for the Cs-eluate before and after concentration. There is consequently a small compositional difference between the test glass, which was based on pre-concentration data, and the glass found in the spreadsheet, which is based on post-concentration data. The spreadsheet also assumes the concentrate density, which was not provided, to be 1 g/ml, likely to be lower than the actual value. Finally, as discussed during the teleconference, substantial amount of nitrate is present and this possibly will cause foaming in the melt.

Should there be questions about the spreadsheet, please feel free to reach me. Thanks.

W.K. Kot
Vitreous State Laboratory
The Catholic University of America
(202)319-5321

The following section of this message contains a file attachment prepared for transmission using the Internet MIME message format. If you are using Pegasus Mail, or any another MIME-compliant system, you should be able to save it or view it from within your mailer. If you cannot, please ask your system administrator for assistance.

----- File information -----
File: C106_SRTC_01_23.xls
Date: 23 Jan 2001, 16:32
Size: 40960 bytes.
Type: Excel-sheet

Appendix B. Technical Data Sheets for Glass Formers – Boric Acid, Lithium Carbonate
and Silica



Boric Acid

Technical Granular

Orthoboric Acid
 H_3BO_3
CAS No. 10043-35-3

Product Specification B-0310-U

February 1, 1998

Boric Acid Technical Granular is a free-flowing, white,
crystalline product manufactured in the USA by U.S. Borax Inc.

Chemical specification

	Guarantee	
B_2O_3 %	56.3-56.8	
Equivalent H_3BO_3 %	99.9-100.9	
SO_4 ppm	≤350	0 0 350
Cl ppm	≤18	
Fe ppm	≤6	

Sieve specification

U.S. Standard Sieve No.	% Retained Guarantee
20	≤2.0

Note:
All data in the above specifications are determined by U.S. Borax analytical
methods.

Packaging

Boric Acid Technical Granular is available in bulk, in 2500 lb.
IBCs and in 50 lb. multiwall paper sacks.



Issued by:
U.S. Borax Inc.
26877 Tourney Road
Valencia, CA 91355-1847
USA

Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0



Cyprus Foote Mineral Company
Silver Peak Operations Hwy 265
Silver Peak, Nevada 89047
(702) 937-2222
FAX (702) 937-2250

PAT
Li2CO3

CERTIFICATE of ANALYSIS
LITHIUM CARBONATE
(CRYSTAL)

ANALYSIS
DATE: 04/08/95

CHEMICAL ANALYSIS	LOT NO. 503-14 %	REQUIRED SPEC'S %
Li2CO3	99.2	MIN 99.0
Cl	0.017	
SO4	0.12	MAX .40
H2O	0.02	
Ca	0.045	
Mg	0.007	
Na	0.09	
K	0.037	
B	0.03	
Fe2O3	0.001	MAX .004
Insol	0.018	
LOI	0.65	
Reflectance: Blue	93	
Green	93	
SIEVE ANALYSIS		
+10	0.0	MAX 0.0
+20	0.3	MAX 1.5
+30	0.8	
+40	2.8	
+60	19.1	
+100	29.0	
+140	16.0	
+200	10.9	
-200	21.1	MAX 60.0

WE CERTIFY THAT THIS
LOT MEETS THE MINIMUM
REQUIREMENTS FOR SP01000. Rev 1.

David H. Miller 4/10/95
QA ANALYST DATE

FOR SHIPPING USE ONLY	
CUSTOMER	RAY SCHUMACHER
CUSTOMER PO#	VERBAL
CUSTOMER CODE#	
CFM ORDER#	1003490



Crucible Scale Vitrification of
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Secondary Wastes

WSRC-TR-2001-00252
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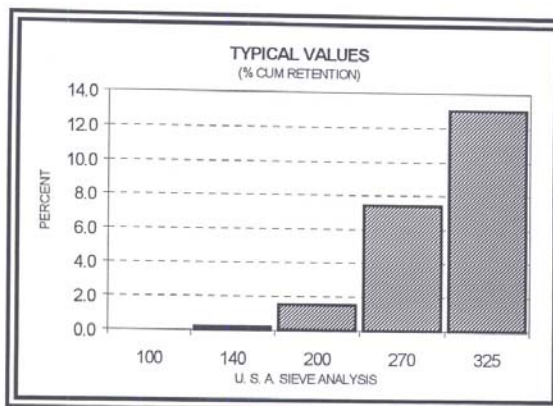


PRODUCT DATA

SIL-CO-SIL® 75

GROUND SILICA

PLANT: MILL CREEK, OKLAHOMA



USA STD SIEVE SIZE	MILLIMETERS	% RETAINED		CUMULATIVE
		INDIVIDUAL	CUMULATIVE	% PASSING
100	0.150	0.0	0.0	100.0
140	0.106	0.2	0.2	99.8
200	0.075	1.3	1.5	98.5
270	0.053	5.9	7.4	92.6
325	0.045	5.6	13.0	87.0

TYPICAL PHYSICAL PROPERTIES

HARDNESS (Mohs) 7.0
MELTING POINT (Degrees F) 3100
MINERAL..... QUARTZ
pH..... 7

REFLECTANCE (%) 89.5
YELLOWNESS INDEX 3.42
SPECIFIC GRAVITY (g/cm³) 2.65

TYPICAL CHEMICAL ANALYSIS, %

SiO₂ (SILICON DIOXIDE) 99.7
Fe₂O₃ (IRON OXIDE) 0.016
Al₂O₃ (ALUMINUM OXIDE) 0.135
TiO₂ (TITANIUM DIOXIDE)..... 0.008
CaO (CALCIUM OXIDE) 0.008

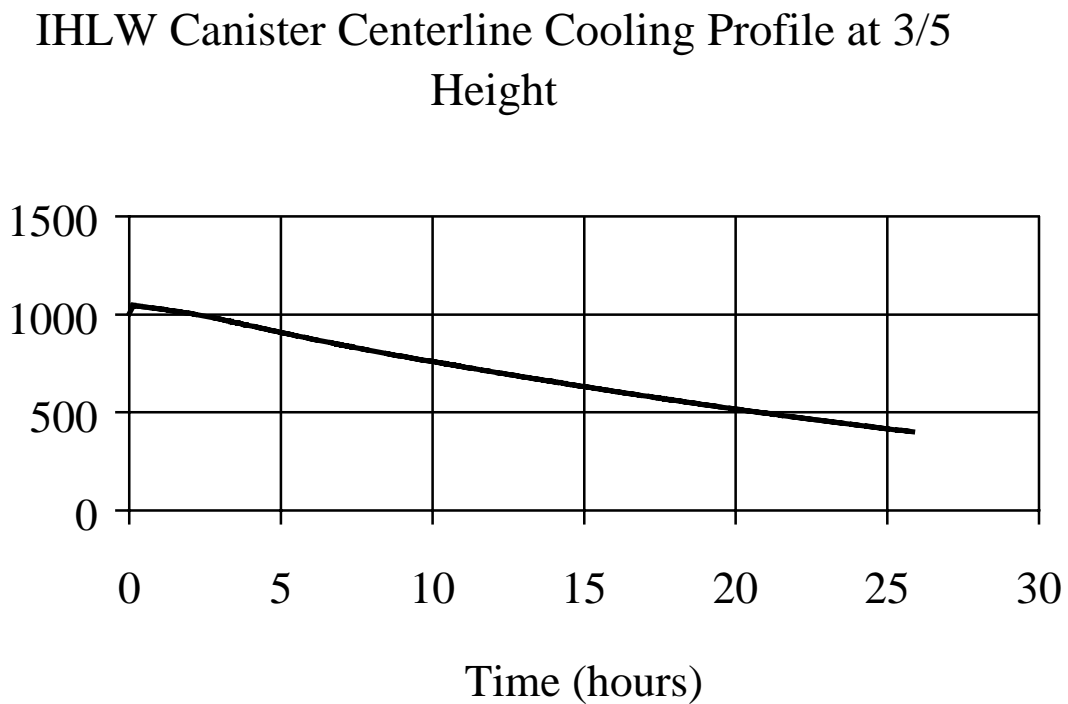
MgO (MAGNESIUM OXIDE)..... 0.008
Na₂O (SODIUM OXIDE)..... 0.002
K₂O (POTASSIUM OXIDE) 0.017
LOI (LOSS ON IGNITION) 0.07

05-29-98

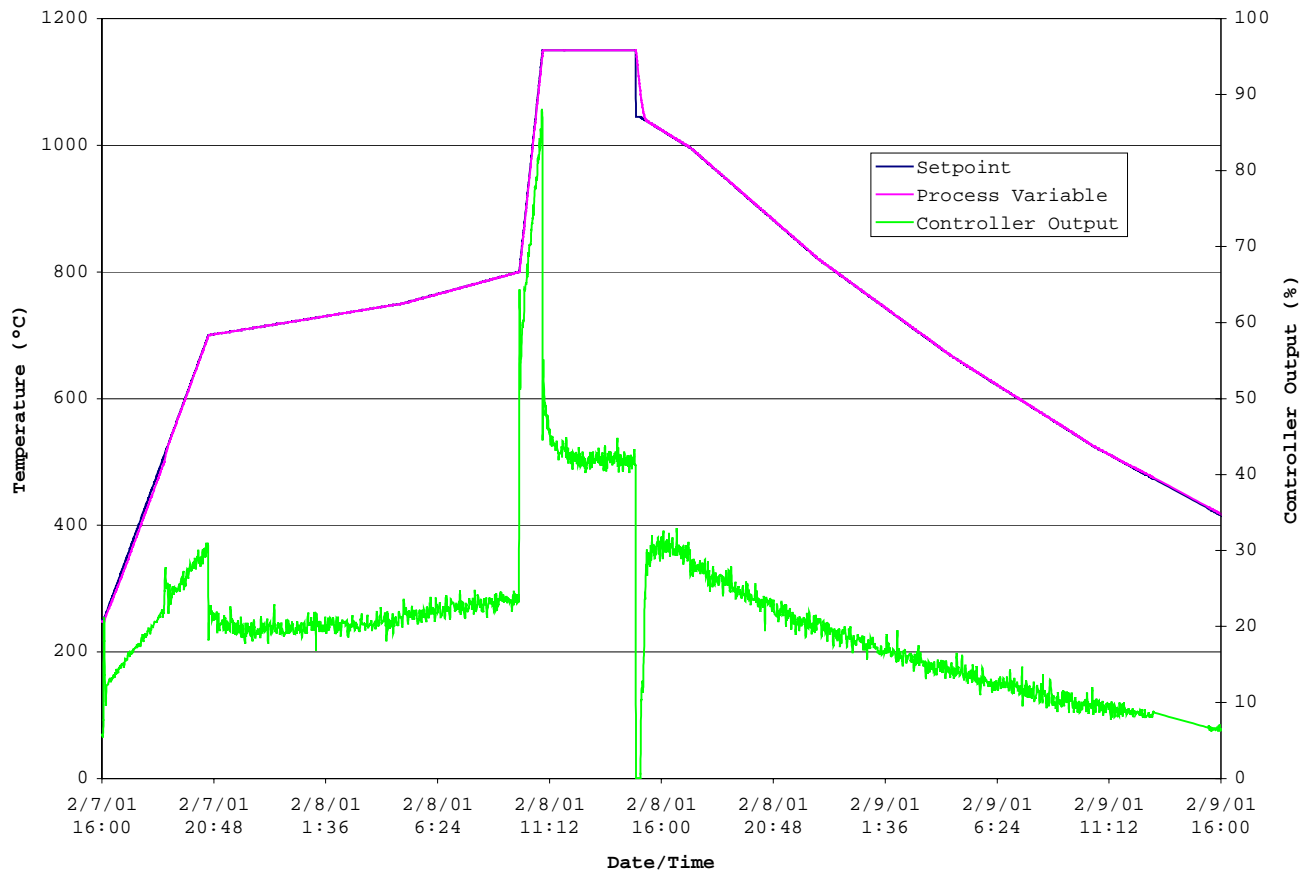
U.S. Silica Company • P.O. Box 187, Berkeley Springs, WV 25411 • (800) 243-7500

Appendix C. Temperature Profiles during Vitrification

Appendix C, Figure 1. HLW Glass Cooling Curve Provided by BNFL, Inc.



Appendix C, Figure 2. Actual Heatup, Calcine, Melting and Simulated Canister Cooling
Curve Used in Envelope D Glass Crucible Vitrification Study



**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

**WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0**

Appendix D. Peroxide Fusion Analysis –Nitric Acid Uptake

Spread Scheet - Peroxide Fusion Results - No Bias Correction																								
LIMS No.	160285				160286				160287				160288				160289				160290			
Ident.	DG-PF1				DG-PF2				DG-PF3				DG-STD1				DG-STD2				ARG-1			
Weight	0.241				0.231				0.236				0.243				0.236				Average			
Dilution	1037.344				1082.251				1059.322				1028.807				1059.322				Target			
Element	mg/L				mg/L				mg/L				mg/L				mg/L				mg/L			
	Ele Wt%				Ele Wt%				Ele Wt%				Ele Wt%				Ele Wt%				Ele Wt%			
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Appendix E. Peroxide Fusion Analyses - VSL-CUA HLW98-67Glass

	Data		VSL/w	VSL/w				
	W.Kot		HNO3	HCl				
	HLW98-67		Oxide Wt	Oxide Wt	Formula	Element		Ele Wt
Oxide	%		%	%	Weight	Weight	Element	%
AgO	0.03	<	0.03	na	123.88	107.88	Ag	0.03
Al2O3	7.02		6.89	6.97	101.94	26.98	Al	3.645
B2O3	4.5		4.41	4.46	69.64	10.82	B	1.37
BaO	0.04		0.13	0.15	153.36	137.36	Ba	0.12
CaO	0.71		0.99	0.99	56.08	40.08	Ca	0.705
CdO	0.01	<	0.00	< 0.00	128.41	112.41	Cd	0.001
CoO	0	<	0.04	0.02	74.94	58.94	Co	0.03
Cr2O3	0.19		0.15	0.16	152.02	52.01	Cr	0.105
CuO	0.05		0.08	0.08	79.57	63.54	Cu	0.065
Fe2O3	12.57		11.99	12.22	159.68	55.85	Fe	8.385
K2O	0.32		na	na	94.19	39.1	K	na
Li2O	4.5		4.54	4.65	29.88	6.94	Li	2.11
MgO	0.11		0.13	0.13	40.32	24.32	Mg	0.08
MnO	3.53		0.25	3.71	70.93	54.94	Mn	0.195
MoO2	0.01	<	0.01	< 0.01	127.95	95.95	Mo	0.01
Na2O	13.72		na	na	61.99	22.99	Na	na
Nd2O3	na		na	na	336.54	144.27	Nd	na
NiO	0.27		na	na	74.69	58.71	Ni	na
P2O5	0.44		0.45	0.48	141.96	30.975	P	0.195
PbO	0.25		0.24	0.27	223.21	207.21	Pb	0.22
SiO2	44.77		43.72	43.08	60.06	28.09	Si	20.45
SrO	6.66		6.73	6.39	103.63	87.63	Sr	5.695
TiO2	0.12		0.13	0.18	79.9	47.9	Ti	0.08
U3O8	0		na	na	842.21	238.07	U	na
V2O5	0	<	0.02	< 0.02	181.9	50.95	V	0.013
ZnO	0.02	<	0.05	< 0.05	81.37	65.38	Zn	0.037
ZrO2	0.13		0.07	0.11	123.22	91.22	Zr	0.055
Sum	99.97		81.07	84.14				43.475
Ref. VSL-CUA.xls								

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Sodium Peroxide with Nitric Acid Dissolution					Sodium Peroxide with Hydrochloric Acid Dissolution									
LIMS No.	300	300		300	LIMS No.	300	300		300	300			300	
	163442	163443		163444		163834	163835		163836	163837			163838	
Sample	VSL-1	VSL-2	Average VSL 1&2	Blank	Sample	VSL-1	VSL-2	Average VSL 1&2	ARG1	ARG2	Average ARG1&2	ARG-1 Target	Blank	
Element	ELE Wt%	ELE Wt%	ELE Wt%		Element	ELE Wt%	ELE Wt%	ELE Wt%	ELE Wt%	ELE Wt%	ELE Wt%	ELE Wt%	ELE Wt%	
Ag	< 0.03	< 0.03	< 0.03	< 0.03	Ag									
Al	3.66	3.63	3.645	< 0.025	Al	3.68	3.7	3.69	2.42	2.61	2.52	2.500	0.11	
B	1.34	1.4	1.37	< 0.07	B	1.34	1.43	1.385	2.6	2.88	2.74	2.690	< 0.022	
Ba	0.12	0.12	0.12	< 0.001	Ba	0.13	0.13	0.13	0.08	0.08	0.08	0.079	0.01	
Ca	0.69	0.72	0.705	0.15	Ca	0.7	0.71	0.705	1.16	1.24	1.20	1.090	0.16	
Cd	< 0.001	< 0.001	< 0.001	< 0.001	Cd	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001		0.064	< 0.001	
Co	< 0.03	< 0.03	< 0.03	< 0.03	Co	0.02	0.01	0.015	0.03	0.03	0.03		0.09	
Cr	0.1	0.11	0.105	< 0.005	Cr	0.11	0.11	0.11	0.07	0.07	0.07		0.01	
Cu	0.06	0.07	0.065	< 0.006	Cu	0.07	0.06	0.065	0.01	0.02	0.02	0.003	0.03	
Fe	8.37	8.4	8.385	0.05	Fe	8.55	8.55	8.55	9.84	9.89	9.87	9.790	0.2	
K	na	na	na	na	K	na	na	na	na	na	na	na	< 0.07	
Li	2.11	2.11	2.11	< 0.02	Li	2.1	2.22	2.16	1.47	1.48	1.48	1.490	< 0.011	
Mg	0.08	0.08	0.08	< 0.008	Mg	0.08	0.08	0.08	0.44	0.47	0.46	0.520	0.01	
Mn	0.13	0.26	0.195	0.06	Mn	2.9	2.84	2.87	1.7	1.7	1.70	1.460	0.43	
Mo	< 0.01	< 0.01	< 0.01	< 0.01	Mo	< 0.011	< 0.011	< 0.011	< 0.011	< 0.011	< 0.01		< 0.011	
Na	na	na	na	na	Na	na	na	na	na	na	na		na	
Nd	na	na	na	< na	Nd	na	na	na	na	na	na	8.520	na	
Ni	na	na	na	< na	Ni	na	na	na	na	na	na	0.827	na	
P	0.2	0.19	0.195	< 0.07	P	0.23	0.19	0.21	< 0.1	< 0.2	< 0.02	0.110	< 0.1	
Pb	0.22	0.22	0.22	< 0.07	Pb	0.24	0.26	0.25	< 0.07	< 0.07	< 0.07		< 0.07	
Si	20.4	20.5	20.45	< 0.04	Si	20.1	20.2	20.15	22.05	22.14	22.10	22.400	0.07	
Sr	5.68	5.71	5.695	< 0.001	Sr	5.36	5.45	5.405	< 0.01	< 0.01	< 0.01	0.003	< 0.01	
Ti	0.08	0.08	0.08	< 0.014	Ti	0.11	0.1	0.105	0.72	0.72	0.72	0.690	0.05	
U				<	U									
V	< 0.013	< 0.013	< 0.013	< 0.013	V	< 0.014	< 0.014	0.014	< 0.014	< 0.014	< 0.01		< 0.014</	

**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

**WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0**

Appendix F. Peroxide Fusion Analysis– Hydrochloric Acid Uptake

Sample	RPP-DG PF4A	RPP-DG PF4B	RPP-DG PF4B	RPP-DG PF5A	RPP-DG PF5B	RPP-DG PF5B	RPP-DG PF6A	RPP-DG PF6B	RPP-DG PF6B		RSD
LIMS	300163	300163	300163	300163	300163	300163	300163	300163	300163		
	910	916	916	911	917	917	912	918	918		
Weight	0.245			0.244			0.252			PerFus	
Dilution	250	ADS		250	ADS		250	ADS		Average	
Total Dil.	2843.64	3000.00	2826.47	2838.27	3000	2860.65	2962.34	3000	2885.29	4,5,6	
Correction	2.902X	0.942	2.884X	2.908X	0.954	2.931X	2.939	0.962	2.862		
Element	mg/L	Ele Wt%	Ele Wt%	mg/L	Ele Wt%	Ele Wt%	mg/L	Ele Wt%	Ele Wt%	Ele Wt%	%
Ag		N.D.	N.D.		N.D.	N.D.		N.D.	N.D.	N.D.	
Al		3.9763	3.746		3.7447	3.571		4.0213	3.868	3.728	4.0%
B		1.4460	1.362		1.3702	1.307		1.4440	1.389	1.353	
Ba		0.0397	0.037		0.0379	0.036		0.0417	0.040	0.038	5.3%
Ca		0.6007	0.566		0.5787	0.552		0.6197	0.596	0.571	4.0%
Cd		0.0231	0.022		0.0230	0.022		0.0294	0.028	0.024	15.5%
Co		0.1305	0.123		0.1352	0.129		0.1841	0.177	0.143	20.7%
Cr		0.2040	0.192		0.1883	0.180		0.2025	0.195	0.189	4.3%
Cu		0.1745	0.164		0.1751	0.167		0.1600	0.154	0.162	4.3%
Fe		8.7512	8.245		8.2937	7.908		8.8152	8.478	8.211	3.5%
La		< 0.0345	< 0.033		< 0.0345	0.033		< 0.0345	< 0.033	< 0.033	
Li		1.9633	1.850		1.8728	1.786		1.9614	1.886	1.841	2.8%
Mg		0.0948	0.089		0.0904	0.086		0.1044	0.100	0.092	8.1%
Mn		2.7929	2.631		2.6716	2.548		2.8836	2.773	2.651	4.3%
Mo		0.0072	0.007		0.0066	0.006		0.0091	0.009	0.007	
Na		N.D.	N.D.		N.D.	N.D.		N.D.	N.D.	N.D.	
Ni		N.D.	N.D.		N.D.	N.D.		N.D.	N.D.	N.D.	
P		0.2477	0.233		0.2423	0.231		0.2577	0.248	0.237	3.8%
Pb		0.3352	0.316		0.3182	0.303		0.3627	0.349	0.323	7.3%
Si		21.9279	20.660		20.8095	19.843		22.1504	21.303	* 18.402	3.6%
Sn		< 0.1401	< 0.132		< 0.1401	< 0.134		< 0.1401	< 0.135	< 0.133	
Sr		5.2362	4.933		4.9757	4.745		5.1892	4.991	4.890	2.6%
Ti		0.1182	0.111		0.1130	0.108		0.1326	0.128	0.116	9.1%
V		0.0074	0.007		0.0073	0.007		0.0093	0.009	0.008	15.0%
Zn		0.3928	0.370		0.4023	0.384		0.5583	0.537	0.430	21.5%
Zr		0.3624	0.341		0.3442	0.328		0.3604	0.347	0.339	2.8%
		mg/L			mg/L			mg/L			
K		1.0955	0.322		0.9895	0.297		1.0115	0.287	0.302	6.0%
Total			46.494			44.709			48.060		
										* -0.022	
	dpm/ml	dpm/g	uCi/g	dpm/ml	dpm/g	uCi/g	dpm/ml	dpm/g	uCi/g	uCi/g	RSD
Am-241	7.00E+02	2.07E+06	9.34E-01	1.22E+01	3.64E+04	1.64E-02	1.90E+01	5.54E+04	2.50E-02	3.25E-01	162.2%
Cm-244	1.35E+02	4.00E+05	1.80E-01	3.62E+00	1.08E+04	4.86E-03	< 5.93E+00	< 1.73E+04	< 7.79E-03	6.42E-02	156.2%
Pu238	5.34E+02	1.58E+06	7.12E-01	3.69E+02	1.10E+06	4.95E-01	8.57E+02	2.50E+06	1.13E+00	7.78E-01	41.2%
Pu239-40	1.64E+03	4.86E+06	2.19E+00	1.06E+03	3.16E+06	1.42E+00	2.14E+03	6.24E+06	2.81E+00	2.14E+00	32.5%
Pu-241	3.09E+03	9.15E+06	4.12E+00	1.95E+03	5.81E+06	2.62E+00	4.26E+03	1.24E+07	5.59E+00	4.11E+00	36.2%
Sr-90	3.15E+05	9.33E+08	4.20E+02	3.04E+05	9.06E+08	4.08E+02	2.97E+05	8.66E+08	3.90E+02	4.06E+02	3.7%
Alpha	2.00E+04	5.92E+07	2.67E+01	2.87E+04	8.55E+07	3.85E+01	3.64E+04	1.06E+08	4.78E+01	3.77E+01	28.1%
Beta	1.77E+07	5.24E+10	2.36E+04	2.30E+07	6.85E+10	3.09E+04	2.93E+07	8.54E+10	3.85E+04	3.10E+04	24.0%
	uCi/ml			uCi/ml			uCi/ml				
Cs-137	1.91E+00	(drop)	(drop)	1.06E+01		3.15E+04	1.31E+01		3.83E+04	3.49E+04	13.7%
Second Peroxide Fusion Analysis D Glass										Dilution	
Sample	RPP-DG PF4A		RPP-DG PF4B	RPP-DG PF5A		RPP-DG PF5B	RPP-DG PF6A		RPP-DG PF6B		
LIMS	300163		300163	300163		300163	300163		300163		
	910		916	911		917	912		918		
Weight	0.245			0.244			0.252				
Dilution	250.000			250.000			250.000				
Dil Factor-1	980.000			976.000			1008.000				
Empty Wt	88.035		87.315	87.461		88.829	86.029		89.421		
Sample Wt.	91.147		90.457	90.572		91.901	89.086		92.604		
3ml Sample	3.112		3.142	3.111		3.072	3.057		3.183		
6ml Water	97.065		96.377	96.508		97.833	95.013		98.532		
Water	9.030		9.062	9.047		9.004	8.984		9.111		
Dil Factor-2	2.902		2.884	2.908		2.931	2.939		2.862		
Tot. Dil	2843.638		2826.467	2838.275		2860.646	2962.340		2885.293		
Ref. Dglass-pfll.xls											

**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

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Appendix F. Peroxide Fusion Analysis– Hydrochloric Acid Uptake, Continued

Sample	RPP-DG	RPP-DG	RPP-DG	RPP-DG	RPP-DG	RPP-DG					RPP-DG	RPP-DG
	STD3A	STD3B	STD3B	STD4A	STD4B	STD4B					BlnkA	BlnkB
LIMS	300163	300163	300163	300163	300163	300163					300163	300163
	913	919	919	914	920	920					915	921
Weight	0.236			0.263								
Dilution	250	ADS		250	ADS						ADS	ADS
Tot. Dil	944.00	1000	944.00	1052.00	1000	1052.00	Average	ARG-1	Difference		1000	1000
Correction			0.944			1.052						
Element	mg/L	Ele. Wt%	Ele. Wt%	mg/L	Ele. Wt%	Ele. Wt%	Ele. Wt%	Ele. Wt%	Ele. Wt%		mg/L	Ele. Wt%
Ag		N.D.	N.D.		N.D.	N.D.	N.D.					N.D.
Al		2.6019	2.456		2.8048	2.951	2.703	2.5	0.203			0.1099
B		2.3412	2.210		2.6175	2.754	2.482	2.69	-0.208			0.0085
Ba		0.0714	0.067		0.0795	0.084	0.076	0.079	-0.003			0.0010
Ca		1.0010	0.945		1.1102	1.168	1.056	1.09	-0.034			0.1192
Cd		0.0150	0.014		0.0151	0.016	0.015	0.064	-0.049			0.0172
Co		0.1150	0.109		0.1084	0.114	0.111					0.1611
Cr		0.0688	0.065		0.0824	0.087	0.076					0.0109
Cu		0.0238	0.022		0.0237	0.025	0.024	0.003	0.021			0.0288
Fe		8.4033	7.933		9.4234	9.913	8.923	9.79	-0.867			0.1903
La	<	0.0115	< 0.011		< 0.0115	< 0.012					<	0.0115
Li		1.4282	1.348		1.6129	1.697	1.522	1.49	0.032		<	0.0008
Mg		0.4648	0.439		0.5180	0.545	0.492	0.52	-0.028			0.0151
Mn		1.4375	1.357		1.6570	1.743	1.550	1.46	0.090			0.3604
Mo		0.0043	0.004		0.0824	0.087	0.045					0.0048
Na		N.D.	N.D.		N.D.	N.D.		8.52				N.D.
Ni		N.D.	N.D.		N.D.	N.D.		0.827				
P		0.1457	0.138		0.1678	0.177	0.157	0.11	0.047			0.0401
Pb		0.0781	0.074		0.0754	0.079	0.077					0.1020
Si		23.1453	21.849		25.9885	27.340	24.595	22.4	2.195	*		0.1051
Sn	<	0.0467	< 0.044		< 0.0467	< 0.049					<	0.0467
Sr		0.0071	0.007		0.0174	0.018	0.013	0.003	0.010			0.0056
Ti		0.6482	0.612		0.7204	0.758	0.685	0.69	-0.005			0.0475
V		0.0165	0.016		0.0177	0.019	0.017					0.0062
Zn		0.3520	0.332		0.3321	0.349	0.341	0.016	0.325			0.5030
Zr		0.1050	0.099		0.1231	0.130	0.114	0.096	0.018			0.0314
K		mg/L						Si Bias	-2.20%			
Total		22.9140	2.427		22.9980	2.186	2.307				0.826	0.8260
			42.578				47.380	52.348	-4.968			
	dpm/ml	dpm/g	uCi/g	dpm/ml	dpm/g	uCi/g	uCi/g				dpm/ml	uCi/ml
Am-241	1.97E+01	2.09E+04	9.40E-03	2.32E+01	2.21E+04	9.93E-03	9.67E-03			<	4.02E+01	
Cm-244	2.92E+01	3.09E+04	1.39E-02	6.62E+01	6.29E+04	2.83E-02	2.11E-02				3.26E+01	
Pu238	2.12E+02	2.25E+05	1.01E-01	3.95E+02	3.75E+05	1.69E-01	1.35E-01				1.79E+02	
Pu239-40	3.91E+01	4.14E+04	1.87E-02	5.58E+01	5.30E+04	2.39E-02	2.13E-02				2.46E+01	
Pu-241	7.15E+01	7.57E+04	3.41E-02	1.33E+02	1.26E+05	5.69E-02	4.55E-02				1.17E+02	
Sr-90	2.57E+03	2.72E+06	1.23E+00	5.41E+03	5.14E+06	2.32E+00	1.77E+00				2.94E+03	
Alpha	< 1.80E+03	< 1.91E+06	< 8.59E-01	< 1.78E+03	1.69E+06	7.62E-01	8.11E-01			<	1.68E+03	
Beta	1.30E+04	1.38E+07	6.20E+00	7.14E+03	6.79E+06	3.06E+00	4.63E+00				1.16E+04	
	uCi/ml		uCi/ml								uCi/ml	
Cs-137	2.73E-03		2.89E+00	3.81E-03		3.62E+00	3.26E+00				4.47E-03	
Sample	RPP-DG		RPP-DG	RPP-DG		RPP-DG	Average				RPP-DG	RPP-DG
	STD3A		STD3B	STD4A		STD4B					BlnkA	BlnkB
LIMS	300163		300163	300163		300163					300163	300163
	913		919	914		920					915	921
Weight	0.236			0.263								
Dilution	250.000			250.000								
Dil fFactorI	944.000		944.000	1052.000		1052.000						
Empty Wt	7.461		7.665	7.569		7.563					7.643	7.517
Sample Wt.	17.539		17.831	17.800		17.661					17.808	17.466
3ml Sample	10.078		10.166	10.231		10.098					10.165	9.949
6ml Water												
Water												
Dil FactorII												
Tot. Dil	944.000		944.000	1052.000		1052.000						

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Appendix F. Peroxide Fusion Analysis– Hydrochloric Acid Uptake, Continued

Gamma Spectroscopy	Rad1			Rad2				Rad3				ARG1			ARG2			ReagBlank
	(uCi/mL)		(uCi/g)	(uCi/mL)		(uCi/g)		(uCi/mL)		(uCi/g)		(uCi/mL)		(uCi/g)	(uCi/mL)		(uCi/g)	(uCi/mL)
K-40	< 1.46E-01		< 4.3E+02	< 1.71E-01		< 5.1E+02		< 1.46E-01		< 4.1E+02		< 3.57E-03		< 3.8E+00	< 3.12E-03		< 3.0E+00	< 3.96E-03
Co-60	< 3.25E-03		< 9.6E+00	< 7.90E-03		< 2.4E+01		< 1.11E-02		< 3.2E+01		< 6.37E-05		< 6.7E-02	< 1.55E-04		< 4.5E-01	< 2.53E-04
Zn-65	< 2.18E-02		< 6.4E+01	< 2.65E-02		< 7.9E+01		< 2.43E-02		< 6.9E+01		< 2.92E-04		< 3.1E-01	< 4.76E-04		< 4.5E-01	< 2.92E-04
Rb-84	< 3.23E-03		< 9.5E+00	< 1.43E-02		< 4.3E+01		< 1.57E-02		< 4.5E+01		< 2.81E-04		< 3.0E-01	< 2.81E-04		< 2.7E-01	< 2.17E-04
Sr-85	< 2.09E-02		< 6.2E+01	< 4.81E-02		< 1.4E+02		< 4.93E-02		< 1.4E+02		< 2.51E-04		< 2.7E-01	< 2.25E-04		< 2.1E-01	< 2.48E-04
Nb-94	< 8.78E-03		< 2.6E+01	< 1.22E-02		< 3.6E+01		< 9.80E-03		< 2.8E+01		< 2.25E-04		< 2.4E-01	< 1.48E-04		< 1.4E-01	< 2.10E-04
Zr-95	< 9.67E-03		< 2.8E+01	< 2.04E-02		< 6.1E+01		< 1.85E-02		< 5.3E+01		< 3.38E-04		< 3.6E-01	< 2.77E-04		< 2.6E-01	< 2.39E-04
Tc-99m	< 1.10E-02		< 3.2E+01	< 2.22E-02		< 6.6E+01		< 2.37E-02		< 6.7E+01		< 8.20E-05		< 8.7E-02	< 9.89E-05		< 9.4E-02	< 1.235E-04
Ru-106	< 1.53E-01		< 4.5E+02	< 3.84E-01		< 1.1E+03		< 3.93E-01		< 1.1E+03		< 1.69E-03		< 1.8E+00	< 2.18E-03		< 2.1E+00	< 2.00E-03
Sb-125	< 8.07E-02		< 2.4E+02	< 1.72E-01		< 5.1E+02		< 1.92E-01		< 5.5E+02		< 5.83E-04		< 6.2E-01	< 5.65E-04		< 5.4E-01	< 7.39E-04
Sb-126	< 5.95E-03		< 1.8E+01	< 1.14E-02		< 3.4E+01		< 9.71E-03		< 2.8E+01		< 1.70E-04		< 1.8E-01	< 1.90E-04		< 1.8E-01	< 1.90E-04
Sn-126	< 2.93E-02		< 8.6E+01	< 6.33E-02		< 1.9E+02		< 6.31E-02		< 1.8E+02		< 2.28E-04		< 2.4E-01	< 2.61E-04		< 2.5E-01	< 3.27E-04
Cs-134	< 9.45E-03		< 2.8E+01	< 1.24E-02		< 3.7E+01		< 1.32E-02		< 3.7E+01		< 1.66E-04		< 1.8E-01	< 1.60E-04		< 1.5E-01	< 1.77E-04
Cs-137	< 1.91E-00		< 5.6E+03	< 1.06E+01		< 3.2E+04		< 1.31E+01		< 3.7E+04		< 2.73E-03		< 2.9E+00	< 3.81E-03		< 3.6E+00	< 4.47E-03
Ce-144	< 8.13E-02		< 2.4E+02	< 1.89E-01		< 5.7E+02		< 1.94E-01		< 5.5E+02		< 7.93E-04		< 8.4E-01	< 9.81E-04		< 9.3E-01	< 9.66E-04
Eu-152	< 7.00E-02		< 2.1E+02	< 7.16E-02		< 2.1E+02		< 5.88E-02		< 1.7E+02		< 1.17E-03		< 1.2E+00	< 7.88E-04		< 7.5E-01	< 1.23E-03
Eu-154	< 2.35E-02		< 6.9E+01	< 3.12E-02		< 9.3E+01		< 3.12E-02		< 8.9E+01		< 2.32E-04		< 2.5E-01	< 2.80E-04		< 2.7E-01	< 1.94E-04
Eu-155	< 3.31E-02		< 9.7E+01	< 7.22E-02		< 2.2E+02		< 7.25E-02		< 2.1E+02		< 2.61E-04		< 2.8E-01	< 3.15E-04		< 3.0E-01	< 3.53E-04
Tl-208	< 1.94E-02		< 5.7E+01	< 1.74E-02		< 5.2E+01		< 2.42E-02		< 6.9E+01		< 2.29E-04		< 2.4E-01	< 2.39E-04		< 2.3E-01	< 1.86E-04
Pb-212	< 3.42E-02		< 1.0E+02	< 7.19E-02		< 2.2E+02		< 8.13E-02		< 2.3E+02		< 3.31E-04		< 3.5E-01	< 3.42E-04		< 3.3E-01	< 3.02E-04
Bi-214	< 5.64E-02		< 1.7E+02	< 3.44E-02		< 1.0E+02		< 9.89E-02		< 2.8E+02		< 5.11E-04		< 5.4E-01	< 6.62E-04		< 6.3E-01	< 6.62E-04
Pb-214	< 6.41E-02		< 1.9E+02	< 1.33E-01		< 4.0E+02		< 1.49E-01		< 4.2E+02		< 5.16E-04		< 5.5E-01	< 5.99E-04		< 5.7E-01	< 6.34E-04
Ra-224	< 3.89E-01		< 1.1E+03	< 7.74E-01		< 2.3E+03		< 8.89E-01		< 2.5E+03		< 3.38E-03		< 3.6E+00	< 4.19E-03		< 4.0E+00	< 3.38E-03
Ra-226	< 4.03E-01		< 1.2E+03	< 8.60E-01		< 2.6E+03		< 9.25E-01		< 2.6E+03		< 4.03E-03		< 4.3E+00	< 3.51E-03		< 3.3E+00	< 3.83E-03
Ac-228	< 3.49E-02		< 1.0E+02	< 4.25E-02		< 1.3E+02		< 3.01E-02		< 8.5E+01		< 6.85E-04		< 7.3E-01	< 8.34E-04		< 7.9E-01	< 8.97E-04
Pa-233	< 5.02E-02		< 1.5E+02	< 9.55E-02		< 2.9E+02		< 1.04E-01		< 3.0E+02		< 3.67E-04		< 3.9E-01	< 3.67E-04		< 3.5E-01	< 4.65E-04
Pa-234	< 4.11E-02		< 1.2E+02	< 5.47E-02		< 1.6E+02		< 7.32E-02		< 2.1E+02		< 4.56E-04		< 4.8E-01	< 4.66E-04		< 4.4E-01	< 5.76E-04
Th-234	< 4.10E-01		< 1.2E+03	< 7.94E-01		< 2.4E+03		< 9.05E-01		< 2.6E+03		< 4.38E-03		< 4.6E+00	< 4.40E-03		< 4.2E+00	< 5.04E-03
U-234	< 2.72E+01		< 8.0E+04	< 6.47E+01		< 1.9E+05		< 6.74E+01		< 1.9E+05		< 2.59E-01		< 2.7E+02	< 2.79E-01		< 2.7E+02	< 2.23E-01
U-235	< 2.45E-02		< 7.2E+01	< 5.22E-02		< 1.6E+02		< 5.53E-02		< 1.6E+02		< 2.36E-04		< 2.5E-01	< 2.20E-04		< 2.1E-01	< 2.27E-04
Np-237	< 5.02E-02		< 1.5E+02	< 9.55E-02		< 2.9E+02		< 1.04E-01		< 3.0E+02		< 3.67E-04		< 3.9E-01	< 3.67E-04		< 3.5E-01	< 4.65E-04
U-238	< 1.32E+00		< 3.88E+03	< 1.71E+00		< 5.11E+03		< 1.32E+00		< 3.75E+03		< 3.01E-02		< 3.19E+01	< 2.06E-02		< 2.0E+01	< 2.06E-02
Np-239	< 4.00E-02		< 1.18E+02	< 8.41E-02		< 2.52E+02		< 9.14E-02		< 2.60E+02		< 3.95E-04		< 4.18E-01	< 3.67E-04		< 3.5E-01	< 4.04E-04
Pu-239	< 1.42E+02		< 4.18E+05	< 3.16E+02		< 9.45E+05		< 3.35E+02		< 9.51E+05		< 1.23E+00		< 1.30E+03	< 1.45E+00		< 1.4E+03	< 1.51E+00
Am-241	< 7.06E-02		< 2.1E+02	< 1.50E-01		< 4.5E+02		< 1.57E-01		< 4.5E+02		< 6.40E-04		< 6.8E-01	< 5.81E-04		< 5.5E-01	< 7.42E-04
Am-243	< 2.34E-02		< 6.9E+01	< 4.71E-02		< 1.4E+02		< 5.42E-02		< 1.5E+02		< 2.54E-04		< 2.7E-01	< 2.06E-04		< 2.0E-01	< 2.89E-04
Cm-243	< 1.05E-01		< 3.1E+02	< 2.35E-01		< 7.0E+02		< 2.46E-01		< 7.0E+02		< 8.86E-04		< 9.4E-01	< 8.60E-04		< 8.2E-01	< 1.13E-03
Cm-245	< 3.24E-02		< 9.5E+01	< 6.97E-02		< 2.1E+02		< 7.61E-02		< 2.2E+02		< 3.90E-04		< 4.1E-01	< 2.92E-04		< 2.8E-01	< 3.41E-04

**Crucible Scale Vittrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

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**Appendix F. Continued, ICP-MS Data Peroxide Fusion Analysis– Hydrochloric Acid
Uptake**

	Half-life	Rad1	wt%		Rad2	wt%		Rad3	wt%		ARG1	wt%		ARG2	wt%	ReagBlnk
	(years)	(ug/L)		(uCi/g)	(ug/L)		(uCi/g)	(ug/L)		(uCi/g)	(ug/L)		(ug/L)		(ug/L)	(ug/L)
mass 230		< 1.70	< 5.0E-04		< 1.70	< 5.1E-04		< 1.70	< 4.8E-04		< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 231(Pa)	3.28E+04	< 1.70	< 5.0E-04	< 2.4E-01	< 1.70	< 5.1E-04	< 2.4E-01	< 1.70	< 4.8E-04	< 2.3E-01	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 232 (Th)	1.40E+10	145.16	4.3E-02	4.7E-05	139.79	4.2E-02	4.6E-05	138.10	3.9E-02	4.3E-05	2.30	2.4E-04	3.02	2.9E-04	0.89	
mass 233 (U)	1.59E+00	< 1.70	< 5.0E-04	< 5.0E-02	< 1.70	< 5.1E-04	< 5.0E-02	< 1.70	< 4.8E-04	< 4.7E-02	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 234 (U)	2.45E+00	< 1.70	< 5.0E-04	< 3.1E-02	< 1.70	< 5.1E-04	< 3.2E-02	< 1.70	< 4.8E-04	< 3.0E-02	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 235 (U)	7.04E+08	3.51	1.0E-03	2.2E-05	3.01	9.0E-04	1.9E-05	3.17	9.0E-04	2.0E-05	0.35	3.7E-05	0.27	2.6E-05	0.36	
mass 236 (U)	2.34E+07	< 1.70	< 5.0E-04	< 3.2E-04	< 1.70	< 5.1E-04	< 3.3E-04	< 1.70	< 4.8E-04	< 3.1E-04	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 237 (Np)	2.14E+06	< 1.70	< 5.0E-04	< 3.5E-03	1.73	5.2E-04	3.6E-03	1.83	5.2E-04	3.7E-03	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 238 (U)	4.47E+09	401.23	1.2E-01	4.0E-04	457.04	1.4E-01	4.6E-04	387.76	1.1E-01	3.7E-04	80.23	8.5E-03	51.51	4.9E-03	87.12	
mass 239 (Pu)	2.41E+04	8.40	2.5E-03	1.5E+00	7.73	2.3E-03	1.4E+00	8.84	2.5E-03	1.6E+00	0.21	2.2E-05	0.29	2.8E-05	0.19	
mass 240 (Pu)	6.56E+03	< 1.70	< 5.0E-04	< 1.1E+00	< 1.70	< 5.1E-04	< 1.2E+00	< 1.70	< 4.8E-04	< 1.1E+00	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 241 (Am)	4.33E+02	< 1.70	< 5.0E-04	< 1.7E+01	< 1.70	< 5.1E-04	< 1.7E+01	< 1.70	< 4.8E-04	< 1.7E+01	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 242 (Pu)	3.76E+05	< 1.70	< 5.0E-04	< 2.0E-02	< 1.70	< 5.1E-04	< 2.0E-02	< 1.70	< 4.8E-04	< 1.9E-02	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 243 (Am)	7.37E+03	< 1.70	< 5.0E-04	< 1.0E+00	< 1.70	< 5.1E-04	< 1.0E+00	< 1.70	< 4.8E-04	< 9.6E-01	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 244 (Cm)	1.81E+01	< 1.70	< 5.0E-04	< 4.1E+02	< 1.70	< 5.1E-04	< 4.1E+02	< 1.70	< 4.8E-04	< 3.9E+02	< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 245		< 1.70	< 5.0E-04		< 1.70	< 5.1E-04		< 1.70	< 4.8E-04		< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 246		< 1.70	< 5.0E-04		< 1.70	< 5.1E-04		< 1.70	< 4.8E-04		< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
mass 247		< 1.70	< 5.0E-04		< 1.70	< 5.1E-04		< 1.70	< 4.8E-04		< 0.17	< 1.8E-05	< 0.17	< 1.6E-05	< 0.17	
Mass Number		(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(ug/L)	wt%	(ug/L)	(ug/L)
81		< 370.03	< 1.1E-01		< 370.03	< 1.1E-01		< 370.03	< 1.1E-01		105.89	1.1E-02	139.62	1.3E-02	67.68	
82		< 12.96	< 3.8E-03		< 12.96	< 3.9E-03		< 12.96	< 3.7E-03		< 1.30	< 1.4E-04	< 1.30	< 1.2E-04	< 1.30	
83		< 10.10	< 3.0E-03		< 10.10	< 3.0E-03		< 10.10	< 2.9E-03		7.29	7.7E-04	18.91	1.8E-03	< 1.01	
84		89.59	2.6E-02		86.57	2.6E-02		88.53	2.5E-02		0.46	4.8E-05	1.10	1.0E-04	0.41	
85		2.35	6.9E-04		2.64	7.9E-04		2.75	7.8E-04		30.72	3.3E-03	34.01	3.2E-03	0.34	
86		1601.87	4.7E-01		1518.71	4.6E-01		1596.85	4.5E-01		7.77	8.2E-04	19.84	1.9E-03	6.49	
87		1155.19	3.4E-01		1079.95	3.2E-01		1112.18	3.2E-01		18.02	1.9E-03	27.86	2.6E-03	4.76	
88		13567.62	4.0E+00		12566.07	3.8E+00		13278.00	3.8E+00		66.87	7.1E-03	168.60	1.6E-02	55.31	
89		19.46	5.7E-03		19.18	5.8E-03		18.82	5.3E-03		6.87	7.3E-04	7.91	7.5E-04	0.18	
90		379.73	1.1E-01		358.43	1.1E-01		321.74	9.1E-02		414.51	4.4E-02	517.43	4.9E-02	9.46	
91		114.88	3.4E-02		114.05	3.4E-02		100.72	2.9E-02		89.29	9.5E-03	109.40	1.0E-02	2.07	
92		187.77	5.5E-02		180.58	5.4E-02		162.71	4.6E-02		157.13	1.7E-02	327.82	3.1E-02	6.75	
93		51.70	1.5E-02		49.64	1.5E-02		44.01	1.2E-02		26.53	2.8E-03	56.44	5.4E-03	7.93	
94		187.78	5.5E-02		185.35	5.6E-02		157.81	4.5E-02		156.45	1.7E-02	274.11	2.6E-02	5.51	
95		3.00	8.8E-04		2.39	7.2E-04		1.09	3.1E-04		1.26	1.3E-04	137.43	1.3E-02	3.19	
96		70.97	2.1E-02		70.02	2.1E-02		59.29	1.7E-02		27.59	2.9E-03	170.21	1.6E-02	3.79	
97		1.83	5.4E-04		1.45	4.4E-04		1.55	4.4E-04		0.76	8.0E-05	82.72	7.9E-03	1.92	
98		5.05	1.5E-03		4.63	1.4E-03		5.81	1.6E-03		10.26	1.1E-03	214.78	2.0E-02	16.97	
99 (Tc-99)	2.13E+05	< 0.95	< 2.8E-04	< 4.7E-02	< 0.95	< 2.9E-04	< 4.8E-02	< 0.95	< 2.7E-04	< 4.6E-02	0.18	1.9E-05	0.25	2.4E-05	0.21	
100		2.42	7.1E-04		2.64	7.9E-04		2.79	7.9E-04		4.13	4.4E-04	86.21	8.2E-03	6.76	
101		9.90	2.9E-03		9.39	2.8E-03		5.00	1.4E-03		0.28	3.0E-05	0.28	2.6E-05	0.34	
102		10.11	3.0E-03		7.97	2.4E-03		4.72	1.3E-03		0.57	6.1E-05	0.55	5.2E-05	0.99	
103		3.63	1.1E-03		3.85	1.2E-03		3.65	1.0E-03		0.05	4.8E-06	0.08	7.2E-06	0.05	
104		10.17	3.0E-03		8.62	2.6E-03		7.30	2.1E-03		0.31	3.2E-05	0.28	2.6E-05	0.33	
105		4.86	1.4E-03		4.34	1.3E-03		4.50	1.3E-03		0.29	3.1E-05	0.17	1.6E-05	0.08	
106		6.69	2.0E-03		6.37	1.9E-03		5.45	1.5E-03		6.22	6.6E-04	6.98	6.6E-04	0.27	
107		373.54	1.1E-01		373.03	1.1E-01		376.41	1.1E-01		2.83	3.0E-04	3.21	3.0E-04	< 1.96	
108		3.69	1.1E-03		3.69	1.1E-03		3.12	8.8E-04		3.30	3.5E-04	3.46	3.3E-04	0.53	
109		354.52	1.0E-01		352.70	1.1E-01		356.56	1.0E-01		1.42	1.5E-04	2.01	1.9E-04	< 1.37	
110		6.37	1.9E-03		5.69	1.7E-03		6.31	1.8E-03		2.77	2.9E-04	3.65	3.5E-04	0.32	
111		4.10	1.2E-03		3.76	1.1E-03		4.03	1.1E-03		0.45	4.7E-05	1.08	1.0E-04	0.26	
112		7.55	2.2E-03		6.72	2.0E-03		7.92	2.2E-03		1.76	1.9E-04	3.34	3.2E-04	0.41	
113		< 238.29	< 7.0E-02	< 238.29	< 7.2E-02	< 238.29	< 6.8E-02	< 238.29	< 6.8E-02	< 23.83	< 2.5E-03	< 23.83	< 2.3E-03	< 23.83		
114		6.72	2.0E-03		6.07	1.8E-03		7.73	2.2E-03		1.26	1.3E-04	2.86	2.7E-04	0.37	
115		< 5141.35	< 1.5E+00	< 5141.35	< 1.5E+00	< 5141.35	< 1.5E+00	< 5141.35	< 1.5E+00	< 514.13	< 5.4E-02	< 514.13	< 4.9E-02	< 514.13		
116		4.80	1.4E-03		5.40	1.6E-03		4.78	1.4E-03		9.57	1.0E-03	10.96	1.0E-03	0.54	
117		1.80	5.3E-04		1.66	5.0E-04		2.04	5.8E-04		8.31	8.8E-04	9.69	9.2E-04	0.50	
118		< 4.87	< 1.4E-03		4.91	1.5E-03		< 4.87	< 1.4E-03		26.69	2.8E-03	29.18	2.8E-03	1.22	
119		7.97	2.3E-03		9.29	2.8E-03		7.30	2.1E-03		10.48	1.1E-03	11.59	1.1E-03	1.57	
120		7.01	2.1E-03		5.86	1.8E-03		5.48	1.6E-03		36.10	3.8E-03	39.69	3.8E-03	1.51	
121		< 6.73	< 2.0E-03	< 6.73	< 2.0E-03	< 6.73	< 1.9E-03	< 6.73	< 1.9E-03		2.17	2.3E-04	1.82	1.7E-04	0.96	
122		< 3.40	< 1.0E-03	< 3.40	< 1.0E-03	< 3.40	< 9.7E-04	< 3.40	< 9.7E-04		7.16	7.6E-04	8.08	7.7E-04	0.52	
123		< 0.80	< 2.4E-04	< 0.80	< 2.4E-04	< 0.80	< 2.3E-04	< 0.80	< 2.3E-04		1.59	1.7E-04	1.28	1.2E-04	1.53	
124		< 2.53	< 7.4E-04	< 2.53	< 7.6E-04	< 2.53	< 7.2E-04	< 2.53	< 7.2E-04		6.44	6.8E-04	7.65	7.3E-04	0.34	
125		0.88	2.6E-04		0.78	< 2.3E-04		0.78	< 2.2E-04		< 0.08	< 8.3E-06	< 0.08	< 7.4E-06	< 0.08	
126		0.99	2.9E-04		1.34	4.0E-04		1.18	3.4E-04		< 0.08	< 8.6E-06	< 0.08	< 7.9E-06	< 0.08	
127		< 2.84	< 8.4E-04	< 2.84	< 8.5E-04	< 2.84	< 8.1E-04	< 2.84	< 8.1E-04		< 0.28	< 3.0E-05	< 0.28	< 2.7E-05	0.29	
128		4.42	1.3E-03		4.77	1.4E-03		5.31	1.5E-03		0.07	7.7E-06	< 0.04	< 3.9E-06	0.05	
129		< 11.20	< 3.3E-03	< 11.20	< 3.4E-03	< 11.20	< 3.2E-03	< 11.20	< 3.2E-03		< 1.12	< 1.2E-04	< 1.12	< 1.1E-04	< 1.12	
130		15.58	4.6E-03		15.46	4.6E-03		17.06	4.8E-03		0.83	8				

Crucible Scale Vittrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0

Appendix F. Continued, ICP-MS Data Peroxide Fusion Analysis - Hydrochloric Acid
Uptake

Mass Number	Half-life (years)	Rad1 (ug/L)	wt%	(uCi/g)	Rad2 (ug/L)	wt%	(uCi/g)	Rad3 (ug/L)	wt%	(uCi/g)	ARG1 (ug/L)	wt%	ARG2 (ug/L)	wt%	ReagBlk (ug/L)
136		< 4.02	< 1.2E-03		< 4.02	< 1.2E-03		< 4.02	< 1.1E-03		57.52	6.1E-03	66.58	6.3E-03	1.32
137		166.82	4.9E-02		158.39	4.8E-02		167.75	4.8E-02		82.78	8.8E-03	94.29	9.0E-03	1.91
138		102.12	3.0E-02		96.68	2.9E-02		105.64	3.0E-02		525.30	5.6E-02	610.11	5.8E-02	12.33
139		27.14	8.0E-03		23.87	7.2E-03		23.31	6.6E-03		6.99	7.4E-04	7.85	7.5E-04	1.19
140		47.75	1.4E-02		44.59	1.3E-02		46.54	1.3E-02		10.55	1.1E-03	12.70	1.2E-03	0.58
141		19.10	5.6E-03		14.18	4.3E-03		18.41	5.2E-03		1.15	1.2E-04	1.34	1.3E-04	< 0.15
142		44.34	1.3E-02		41.30	1.2E-02		43.10	1.2E-02		3.00	3.2E-04	4.48	4.3E-04	0.67
143		20.84	6.1E-03		19.43	5.8E-03		19.40	5.5E-03		0.77	8.1E-05	1.34	1.3E-04	0.33
144		24.24	7.1E-03		21.63	6.5E-03		22.51	6.4E-03		1.55	1.6E-04	2.57	2.4E-04	0.63
145		14.29	4.2E-03		13.42	4.0E-03		13.69	3.9E-03		0.58	6.2E-05	0.90	8.5E-05	0.29
146		12.17	3.6E-03		11.62	3.5E-03		11.57	3.3E-03		1.11	1.2E-04	1.88	1.8E-04	0.44
147		9.80	2.9E-03		9.53	2.9E-03		10.05	2.9E-03		0.22	2.3E-05	0.32	3.0E-05	0.10
148		6.96	2.0E-03		7.07	2.1E-03		6.31	1.8E-03		0.28	3.0E-05	0.75	7.1E-05	0.18
149		< 0.85	< 2.5E-04		< 0.85	< 2.5E-04		< 0.85	< 2.4E-04		0.17	1.8E-05	0.24	2.3E-05	< 0.08
150		8.25	2.4E-03		7.36	2.2E-03		7.37	2.1E-03		0.45	4.7E-05	0.80	7.6E-05	0.24
151		1.03	3.0E-04		1.08	3.2E-04		1.39	3.9E-04		0.16	1.7E-05	0.17	1.6E-05	< 0.08
152		2.51	7.4E-04		2.29	6.9E-04		2.28	6.5E-04		0.32	3.4E-05	0.40	3.8E-05	0.09
153		1.26	3.7E-04		1.17	3.5E-04		1.02	2.9E-04		0.17	1.8E-05	0.26	2.5E-05	< 0.08
154		0.94	2.8E-04		0.78	2.3E-04		0.76	2.2E-04		0.67	7.1E-05	0.80	7.6E-05	0.09
155		1.18	3.5E-04		1.42	4.3E-04		0.92	2.6E-04		0.84	8.9E-05	0.91	8.6E-05	0.09
156		1.95	5.8E-04		1.93	5.8E-04		1.73	4.9E-04		0.36	3.8E-05	0.43	4.1E-05	< 0.11
157		0.69	2.0E-04		0.77	2.3E-04		0.61	1.7E-04		0.21	2.2E-05	0.26	2.5E-05	0.04
158		2.07	6.1E-04		1.66	4.7E-04		1.62	4.6E-04		0.27	2.9E-05	0.34	3.2E-05	< 0.08
159		< 0.53	< 1.6E-04		< 0.53	< 1.6E-04		< 0.53	< 1.5E-04		0.17	1.8E-05	0.18	1.7E-05	< 0.05
160		0.69	2.0E-04		0.65	2.0E-04		0.68	1.9E-04		0.18	1.9E-05	0.30	2.8E-05	0.06
161		< 0.58	< 1.7E-04		< 0.58	< 1.7E-04		< 0.58	< 1.6E-04		0.24	2.5E-05	0.20	1.9E-05	< 0.06
162		< 0.38	< 1.1E-04		< 0.38	< 1.1E-04		0.47	1.3E-04		0.24	2.5E-05	0.33	3.1E-05	< 0.04
163		< 0.46	< 1.3E-04		< 0.46	< 1.4E-04		< 0.46	< 1.3E-04		0.26	2.8E-05	0.29	2.8E-05	< 0.05
164		< 0.80	< 2.4E-04		< 0.80	< 2.4E-04		< 0.80	< 2.3E-04		0.26	2.7E-05	0.23	2.2E-05	< 0.08
165		< 0.14	< 4.1E-05		< 0.14	< 4.1E-05		< 0.14	< 3.9E-05		0.21	2.2E-05	0.23	2.2E-05	< 0.01
166		< 0.32	< 9.5E-05		< 0.32	< 9.7E-05		< 0.32	< 9.2E-05		0.25	2.7E-05	0.29	2.8E-05	< 0.03
167		< 0.79	< 2.3E-04		< 0.79	< 2.4E-04		< 0.79	< 2.3E-04		0.19	2.1E-05	0.27	2.6E-05	< 0.08
168		< 1.07	< 3.2E-04		< 1.07	< 3.2E-04		< 1.07	< 3.0E-04		0.23	2.4E-05	0.24	2.3E-05	< 0.11
169		< 0.80	< 2.4E-04		< 0.80	< 2.4E-04		< 0.80	< 2.3E-04		0.16	1.7E-05	0.18	1.7E-05	< 0.08
170		< 0.43	< 1.3E-04		< 0.43	< 1.3E-04		< 0.43	< 1.2E-04		0.17	1.8E-05	0.19	1.8E-05	< 0.04
171		< 0.56	< 1.6E-04		< 0.56	< 1.7E-04		< 0.56	< 1.6E-04		0.17	1.8E-05	0.19	1.8E-05	< 0.06
172		< 0.16	< 4.7E-05		< 0.16	< 4.8E-05		< 0.16	< 4.5E-05		0.22	2.3E-05	0.26	2.5E-05	0.02
173		< 0.42	< 1.3E-04		< 0.42	< 1.3E-04		< 0.42	< 1.2E-04		0.18	1.9E-05	0.22	2.1E-05	< 0.04
174		< 0.26	< 7.7E-05		< 0.26	< 7.8E-05		< 0.26	< 7.4E-05		0.26	2.8E-05	0.36	3.4E-05	< 0.03
175		< 1.03	< 3.0E-04		< 1.03	< 3.1E-04		< 1.03	< 2.9E-04		0.20	2.1E-05	0.24	2.3E-05	< 0.10
176		< 0.68	< 2.0E-04		< 0.68	< 2.0E-04		< 0.68	< 1.9E-04		1.10	1.2E-04	1.55	1.5E-04	< 0.07
177		< 0.56	< 1.6E-04		< 0.56	< 1.7E-04		< 0.56	< 1.6E-04		5.15	5.5E-04	6.38	6.1E-04	0.09
178		< 0.87	< 2.6E-04		< 0.87	< 2.6E-04		< 0.87	< 2.5E-04		7.66	8.1E-04	9.56	9.1E-04	0.11
179		< 0.97	< 2.9E-04		< 0.97	< 2.9E-04		< 0.97	< 2.8E-04		3.73	4.0E-04	4.85	4.6E-04	< 0.10
180		1.97	5.8E-04		1.37	4.1E-04		< 1.01	< 2.9E-04		9.57	1.0E-03	12.29	1.2E-03	0.11
181		7.54	2.2E-03		5.54	1.7E-03		3.96	1.1E-03		13.62	1.4E-03	20.76	2.0E-03	1.69
182		150.40	4.4E-02		136.81	4.1E-02		139.39	4.0E-02		0.35	3.7E-05	2.49	2.4E-04	0.58
183		78.61	2.3E-02		72.42	2.2E-02		75.28	2.1E-02		0.16	1.7E-05	1.31	1.2E-04	0.35
184		172.02	5.1E-02		159.27	4.8E-02		154.73	4.4E-02		0.30	3.1E-05	2.77	2.6E-04	0.71
185		< 1.05	< 3.1E-04		< 1.05	< 3.1E-04		< 1.05	< 3.0E-04		0.83	8.8E-05	0.80	7.7E-05	1.07
186		159.27	4.7E-02		145.08	4.4E-02		141.63	4.0E-02		0.28	2.9E-05	2.56	2.4E-04	0.60
187		0.53	1.6E-04		0.64	1.9E-04		0.86	2.4E-04		1.23	1.3E-04	1.38	1.3E-04	1.86
188		< 0.17	< 4.9E-05		< 0.17	< 5.0E-05		< 0.17	< 4.7E-05		< 0.02	< 1.8E-06	< 0.02	< 1.6E-06	< 0.02
189		< 0.10	< 3.0E-05		< 0.10	< 3.1E-05		< 0.10	< 2.9E-05		< 0.01	< 1.1E-06	0.01	1.2E-06	< 0.01
190		< 0.26	< 7.6E-05		< 0.26	< 7.7E-05		< 0.26	< 7.3E-05		< 0.03	< 2.7E-06	< 0.03	< 2.4E-06	< 0.03
191		< 0.19	< 3.5E-04		< 0.19	< 3.6E-04		< 0.19	< 3.4E-04		< 0.12	< 1.3E-05	< 0.12	< 1.1E-05	< 0.12
192		< 0.63	< 1.8E-04		< 0.63	< 1.9E-04		< 0.63	< 1.8E-04		< 0.06	< 6.6E-06	< 0.06	< 5.9E-06	< 0.06
193		< 0.76	< 2.2E-04		< 0.76	< 2.3E-04		< 0.76	< 2.2E-04		0.08	8.9E-06	0.13	1.2E-05	< 0.08
194		1.94	5.7E-04		2.08	6.2E-04		2.25	6.4E-04		0.14	1.5E-05	0.14	1.3E-05	< 0.10
195		2.10	6.2E-04		2.01	6.0E-04		2.18	6.2E-04		0.08	8.5E-06	0.12	1.1E-05	0.07
196		1.84	5.4E-04		1.68	5.0E-04		1.53	4.4E-04		0.18	1.9E-05	0.19	1.8E-05	
197		< 2.35	< 6.9E-04		< 2.35	< 7.1E-04		< 2.35	< 6.7E-04		< 0.24	< 2.5E-05	< 0.24	< 2.2E-05	< 0.24
198		3.45	1.0E-03		< 2.88	< 8.6E-04		< 2.88	< 8.2E-04		< 0.29	< 3.0E-05	< 0.29	< 2.7E-05	< 0.29
199		4.19	1.2E-03		2.60	7.8E-04		< 2.10	< 6.0E-04		0.23	2.4E-05	< 0.21	< 2.0E-05	< 0.21
200		6.10	1.8E-03		< 4.69	< 1.4E-03		< 4.69	< 1.3E-03		< 0.47	< 5.0E-05	< 0.47	< 4.5E-05	< 0.47
201		2.76	8.1E-04		< 1.95	< 5.9E-04		< 1.95	< 5.5E-04		0.27	2.8E-05	0.20	1.9E-05	< 0.20
202		7.49	2.2E-03		< 4.53	< 1.4E-03		< 4.53	< 1.3E-03		0.47	5.0E-05	< 0.45	< 4.3E-05	< 0.45
203		< 764.43	< 2.2E-01		< 764.43	< 2.3E-01		< 764.43	< 2.2E-01		< 76.44	< 8.1E-03	< 76.44	< 7.3E-03	< 76.44
204		13.69	4.0E-03		12.23	3.7E-03		12.56	3.6E-03		0.43	4.6E-05	0.59	5.6E-05	< 0.39
205		< 1871.32	< 5.5E-01		< 1871.32	< 5.6E-01		< 1871.32	< 5.3E-01		< 187.13	< 2.0E-02	< 187.13	< 1.8E-02	< 187.13
206		217.51	6.4E-02		206.07	6.2E-02		209.41	5.9E-02		7.10	7.5E-04	10.05	9.6E-04	6.31
207		209.32	6.2E-02		190.56	5.7E-02		200.93	5.7E-02		6.03	6.4E-04	9.01	8.6E-04	5.58
208		470.44	1.4E-01		443.23	1.3E-01		458.31	1.3E-01		14.23	1.5E-03	20.69	2.0E-03	13.12

Appendix G. Glass Analysis by Acid Dissolution

	A&B		A&B		A&B		Best Anal.
Sample	DG-AD1		DG-AD2		DG-AD3		RPP-DG
Glass	0.238		0.237		0.250		AD1,2
Vol.ml	250		250		250		
Dilution(1)	952.00		948.00		1000.00		
							Average
Dilution(2)	2.918	[10X]	2.932	[10X]	2.812	[10X]	
Elements	mg/L	Ele%	mg/L	Ele%	mg/L	Ele %	Ele %
ICP-ES							
Ag	0.056	0.017	0.070	0.022	< 0.050	< 0.014	0.019
Al	12.953	3.970	11.920	3.687	7.734	2.175	3.828
B	321.842		319.493		261.863		N.D.
Ba	0.183	0.056	0.177	0.055	0.341	0.096	0.055
Ca	1.503	0.461	1.368	0.423	2.276	0.640	0.442
Cd	0.039	0.012	0.045	0.014	< 0.020	< 0.006	0.013
Co	0.160	0.049	0.175	0.054	< 0.030	< 0.008	0.052
Cr	0.679	0.208	0.615	0.190	0.330	0.093	0.199
Cu	0.688	0.211	0.631	0.195	0.294	0.083	0.203
Fe	30.349	9.302	27.168	8.403	23.639	6.647	8.852
La	< 0.100	< 0.031	< 0.100	< 0.031	< 0.100	< 0.028	N.A.
Li	6.710	2.057	6.035	1.867	5.723	1.609	1.962
Mg	0.302	0.093	0.278	0.086	0.218	0.061	0.089
Mn	8.853	2.714	7.996	2.473	6.420	1.805	2.593
Mo	< 0.030	< 0.009	< 0.030	< 0.009	1.075	0.302	N.A.
Na	31.965	9.798	28.988	8.965	23.197	6.523	9.382
Ni	0.786	0.241	0.716	0.221	0.353	0.099	0.231
P	1.057	0.324	0.848	0.262	10.285	2.892	0.293
Pb	1.023	0.314	0.977	0.302	0.772	0.217	0.308
Si	84.233	25.818	77.871	24.084	67.252	18.911	* 24.951
Sn	0.168	0.051	< 0.160	< 0.049	7.737	2.176	0.050
Sr	18.706	5.734	17.148	5.304	15.853	4.458	5.519
Ti	0.309	0.095	0.294	0.091	1.257	0.353	0.093
V	0.049	0.015	0.062	0.019	< 0.030	< 0.008	0.017
Zn	0.117	0.036	0.110	0.034	0.627	0.176	0.035
Zr	1.393	0.427	1.264	0.391	1.578	0.444	0.409
AA	mg/L		mg/L		mg/L		
K	0.801	0.245	0.726	0.224	0.736	0.207	0.235
Na	25.826	7.916	23.448	7.252	26.819	7.542	7.584
Total		62.247		57.366		49.968	59.807
Isotopes	dpm/g		dpm/g		dpm/g		Avg. 1,2,3
	2.918X	uCi/g	2.932X	uCi/g	2.812X	uCi/g	uCi/g
Am-241	4.08E+06	1.84E+00	3.37E+06	1.52E+00	3.53E+06	1.59E+00	1.65E+00
Cm-244	1.33E+06	6.00E-01	1.01E+06	4.55E-01	9.24E+05	4.16E-01	4.90E-01
Pu-238	9.82E+05	4.43E-01	9.23E+05	4.16E-01	1.93E+06	8.71E-01	5.76E-01
Pu-239-40	4.39E+06	1.98E+00	3.19E+06	1.44E+00	3.53E+06	1.59E+00	1.67E+00
Pu-241	8.06E+06	3.63E+00	6.83E+06	3.08E+00	7.29E+06	3.28E+00	3.33E+00
Sr-90	1.03E+09	4.63E+02	9.38E+08	4.22E+02	8.32E+08	3.75E+02	4.20E+02
Alpha	2.39E+08	1.08E+02	2.58E+08	1.16E+02	2.79E+08	1.26E+02	1.17E+02
Beta	1.16E+11	5.23E+04	9.76E+10	4.40E+04	1.01E+11	4.56E+04	4.73E+04
Cs-137		4.17E+04		3.87E+04		3.70E+04	3.91E+04
Ref. DGlassAD.xls							
							* Need to bias correct -1.96 Ele. %

**Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes**

**WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0**

Appendix G. Glass Analysis by Acid Dissolution, Continued

A&B		A&B		ARG-1		ARG		B						
DG-STAD1		DG-STAD2		Stand	Target			Blank						
0.239		0.252						0.000						
250.000		250.000						250.000						
956.00		1008.00												
1.000	[10X]	1.000	[10X]	Average		Difference		1000.0	Dilution					
mg/L	Ele %	mg/L	Ele %	Ele %	Ele %	Ele %	Ele %	mg/L	Elements					
<	<	<	<				<	0.050	Ag					
22.721	2.377	23.442	2.326	2.351	2.50	0.15		0.336	Al					
954.485		942.931		N.A.	2.69			933.681	B					
0.878	0.092	0.908	0.090	0.091	0.08	-0.01		0.143	Ba					
9.777	1.023	10.196	1.012	1.017	1.09	0.07	<	0.010	Ca					
0.041	0.004	0.025	0.002	0.003	0.06	0.06	<	0.020	Cd					
0.109	0.011	0.093	0.009	0.010				0.047	Co					
0.717	0.075	0.697	0.069	0.072			<	0.090	Cr					
0.056	0.006	0.041	0.004	0.005	0.00	0.00		0.034	Cu					
95.154	9.953	98.409	9.763	9.858	9.79	-0.07		0.203	Fe					
<	<	<	<				<	0.100	La					
14.447	1.511	15.211	1.509	1.510	1.49	-0.02	<	0.030	Li					
5.068	0.530	5.200	0.516	0.523	0.52	0.00		0.010	Mg					
14.122	1.477	14.533	1.442	1.459	1.46	0.00	<	0.010	Mn					
0.031	0.003	<	<				<	0.030	Mo					
82.032	8.581	85.959	8.528	8.554	8.52	-0.03		0.600	Na					
8.134	0.851	8.307	0.824	0.837	0.83	-0.01	<	0.090	Ni					
1.459	0.153	1.574	0.156	0.154	0.11	-0.04	<	0.350	P					
<	<	<	<				<	0.460	Pb					
239.513	25.054	238.642	23.675	24.364	22.40	-1.96		45.580	Si					
0.255	0.027	0.276	0.027	0.027			<	0.160	Sn					
0.042	0.004	0.044	0.004	0.004	0.00	0.00	<	0.010	Sr					
6.693	0.700	6.946	0.689	0.695	0.69	0.00		0.048	Ti					
0.162	0.017	0.137	0.014	0.015				0.054	V					
0.198	0.021	0.199	0.020	0.020	0.02	0.00	<	<.03	Zn					
1.444	0.151	1.279	0.127	0.139	0.10	-0.04		0.030	Zr					
mg/L		mg/L												
20.448	2.139	21.660	2.149	2.144			<	0.135	K					
74.979	7.843	79.103	7.848	7.845				0.217	Na					
	54.823		53.018	53.855	52.35									
dpm/g		dpm/g		Average						Isotopes				
X1.0	uCi/g	X1.0	uCi/g	uCi/g				dpm/ml	uCi/ml					
4.28E+04	1.93E-02	5.69E+03	2.56E-03	1.09E-02				2.50E+00	1.126E-06	Am-241				
4.78E+04	2.15E-02	4.21E+04	1.89E-02	2.02E-02				2.58E+01	1.162E-05	Cm-244				
3.61E+04	1.63E-02	3.77E+04	1.70E-02	1.66E-02				3.11E+02	1.40E-04	Pu-238				
2.05E+04	9.24E-03	5.62E+04	2.53E-02	1.73E-02				1.17E+03	5.27E-04	Pu-239-40				
6.07E+04	2.73E-02	1.33E+05	5.98E-02	4.36E-02				LLD	<	3.58E+02	<	1.61E-04	Pu-241	
1.46E+07	6.60E+00	<	1.36E+07	<	6.13E+00	<	6.36E+00	MDA	<	1.30E+04	<	5.86E-03	Sr-90	
<	1.49E+07	<	6.71E+00	<	1.49E+07	<	6.71E+00	LLD	<	1.44E+04	<	6.49E-03	Alpha	
3.18E+08	1.43E+02	<	5.22E+07	<	2.35E+01	<	8.34E+01			2.15E+05		9.68E-02	Beta	
	8.27E+00		1.61E+01	1.22E+01						1.35E-02		6.08E-09	Cs-137	

Appendix G, Glass Analysis by Acid Dissolution, Continued
Gamma Spectroscopy Data

Gamma Spectroscopy	Rad1 (uCi/mL)	(uCi/g)	Rad2 (uCi/mL)	(uCi/g)	Rad3 (uCi/mL)	(uCi/g)	ARG1 (uCi/mL)	(uCi/g)	ARG2 (uCi/mL)	(uCi/g)	ReagBlink (uCi/mL)
K-40	< 1.82E-01	< 5.58E+02	< 1.31E-01	< 4.05E+02	< 2.02E-01	< 5.68E+02	< 1.92E-01	< 2.01E+02	< 1.59E-01	< 1.58E+02	< 1.13E-01
Co-60	< 9.97E-03	< 3.06E+01	< 9.97E-03	< 3.08E+01	< 9.97E-03	< 2.80E+01	< 1.11E-02	< 1.16E+01	< 2.91E-03	< 2.89E+00	< 7.90E-03
Zn-65	< 1.98E-02	< 5.76E+01	< 1.49E-02	< 4.61E+01	< 2.18E-02	< 6.13E+01	< 1.88E-02	< 1.97E+01	< 1.49E-02	< 1.48E+01	< 2.43E-02
Rb-84	< 1.43E-02	< 4.38E+01	< 8.79E-03	< 2.72E+01	< 1.11E-02	< 3.12E+01	< 1.29E-02	< 1.35E+01	< 1.79E-02	< 1.78E+01	< 1.29E-02
Si-85	< 5.10E-02	< 1.56E+02	< 4.78E-02	< 1.48E+02	< 5.07E-02	< 1.43E+02	< 1.12E-02	< 1.17E+01	< 1.23E-02	< 1.22E+01	< 1.03E-02
Nb-94	< 9.80E-03	< 3.00E+01	< 1.22E-02	< 3.77E+01	< 1.29E-02	< 3.63E+01	< 2.21E-03	< 2.31E+00	< 9.80E-03	< 9.72E+00	< 9.80E-03
Zr-95	< 9.67E-03	< 2.96E+01	< 1.41E-02	< 4.36E+01	< 1.41E-02	< 3.96E+01	< 1.85E-02	< 1.94E+01	< 1.72E-02	< 1.71E+01	< 1.97E-02
Tc-99m	< 2.58E-02	< 7.91E+01	< 2.48E-02	< 7.67E+01	< 2.57E-02	< 7.23E+01	< 4.78E-03	< 5.00E+00	< 4.64E-03	< 4.60E+00	< 3.66E-03
Ru-106	< 3.83E-01	< 1.17E+03	< 3.29E-01	< 1.02E+03	< 3.69E-01	< 1.04E+03	< 6.59E-02	< 6.89E+01	< 1.07E-01	< 1.06E+02	< 4.51E-02
Sb-125	< 1.87E-01	< 5.73E+01	< 1.85E-01	< 5.72E+02	< 1.91E-01	< 5.37E+02	< 1.37E-02	< 1.43E+01	< 2.36E-02	< 2.33E+01	< 1.78E-02
Sb-126	< 7.50E-03	< 2.30E+01	< 1.21E-02	< 3.74E+01	< 5.95E-03	< 1.67E+01	< 5.30E-03	< 5.54E+00	< 5.91E-03	< 5.86E+00	< 8.93E-03
Sn-126	< 6.96E-02	< 2.13E+02	< 6.84E-02	< 2.12E+02	< 6.91E-02	< 1.94E+02	< 1.29E-02	< 1.35E+01	< 1.51E-02	< 1.50E+01	< 1.48E-02
Cs-134	< 1.39E-02	< 4.26E+01	< 1.24E-02	< 3.84E+01	< 1.46E-02	< 4.11E+01	< 5.57E-03	< 5.83E+00	< 7.87E-03	< 7.81E+00	< 7.87E-03
Cs-137	< 1.36E+01	< 4.17E+04	< 1.25E+01	< 3.87E+04	< 1.31E+01	< 3.68E+04	< 7.91E-03	< 8.27E+00	< 1.75E-02	< 1.74E+01	< 1.35E-02
Ce-144	< 2.08E-01	< 6.38E+02	< 1.98E-01	< 6.12E+02	< 2.04E-01	< 5.74E+02	< 2.86E-02	< 2.99E+01	< 3.27E-02	< 3.24E+01	< 3.39E-02
Eu-152	< 5.07E-02	< 1.55E+02	< 5.54E-02	< 1.71E+02	< 7.16E-02	< 2.01E+02	< 5.25E-02	< 5.49E+01	< 5.78E-02	< 5.73E+01	< 5.06E-02
Eu-154	< 2.47E-02	< 7.57E+01	< 9.08E-03	< 2.81E+01	< 2.53E-02	< 7.11E+01	< 9.90E-03	< 1.04E+01	< 9.90E-03	< 9.82E+00	< 8.90E-03
Eu-155	< 8.05E-02	< 2.47E+02	< 7.79E-02	< 2.41E+02	< 8.08E-02	< 2.27E+02	< 1.57E-02	< 1.64E+01	< 1.42E-02	< 1.41E+01	< 1.50E-02
Tl-208	< 1.74E-02	< 5.33E+01	< 1.50E-02	< 4.64E+01	< 1.74E-02	< 4.89E+01	< 1.12E-02	< 1.17E+01	< 9.47E-03	< 9.39E+00	< 1.17E-02
Pb-212	< 8.64E-02	< 2.65E+02	< 8.11E-02	< 2.51E+02	< 8.45E-02	< 2.38E+02	< 1.30E-02	< 1.36E+01	< 1.60E-02	< 1.59E+01	< 1.45E-02
Bi-214	< 9.34E-02	< 2.86E+02	< 9.87E-02	< 3.05E+02	< 1.07E-01	< 3.01E+02	< 2.45E-02	< 2.56E+01	< 3.15E-02	< 3.13E+01	< 2.27E-02
Pb-214	< 1.49E-01	< 4.57E+02	< 1.48E-01	< 4.58E+02	< 1.52E-01	< 4.27E+02	< 2.63E-02	< 2.75E+01	< 2.78E-02	< 2.76E+01	< 2.21E-02
Ra-224	< 9.24E-01	< 2.83E+03	< 8.64E-01	< 2.67E+03	< 9.09E-01	< 2.56E+03	< 1.47E-01	< 1.54E+02	< 1.73E-01	< 1.72E+02	< 1.47E-01
Ra-226	< 8.37E-01	< 2.57E+03	< 9.82E-01	< 3.04E+03	< 8.06E-01	< 2.27E+03	< 1.19E-01	< 1.24E+02	< 1.61E-01	< 1.60E+02	< 1.28E-01
Ac-228	< 3.90E-02	< 1.20E+02	< 3.90E-02	< 1.21E+02	< 3.90E-02	< 1.10E+02	< 3.49E-02	< 3.65E+01	< 4.87E-02	< 4.83E+01	< 3.01E-02
Pa-233	< 1.11E-01	< 3.40E+02	< 1.05E-01	< 3.25E+02	< 1.09E-01	< 3.07E+02	< 1.62E-02	< 1.69E+01	< 1.22E-02	< 1.21E+01	< 1.48E-02
Pa-234	< 7.32E-02	< 2.24E+02	< 6.01E-02	< 1.86E+02	< 5.85E-02	< 1.65E+02	< 1.61E-02	< 1.68E+01	< 1.69E-02	< 1.68E+01	< 2.03E-02
Th-234	< 9.58E-01	< 2.94E+03	< 8.86E-01	< 2.74E+03	< 9.22E-01	< 2.59E+03	< 2.07E-01	< 2.17E+02	< 2.24E-01	< 2.22E+02	< 2.25E-01
U-234	< 7.30E+01	< 2.24E+05	< 6.64E+01	< 2.05E+05	< 7.08E+01	< 1.99E+05	< 1.43E+01	< 1.50E+04	< 1.48E+01	< 1.47E+04	< 1.37E+01
U-235	< 4.94E-02	< 1.51E+02	< 5.98E-02	< 1.85E+02	< 4.92E-02	< 1.38E+02	< 7.22E-03	< 7.55E+00	< 1.02E-02	< 1.01E+01	< 7.76E-03
Np-237	< 1.11E-01	< 3.40E+02	< 1.05E-01	< 3.25E+02	< 1.09E-01	< 3.07E+02	< 1.62E-02	< 1.69E+01	< 1.22E-02	< 1.21E+01	< 1.48E-02
U-238	< 2.48E+00	< 7.60E+03	< 1.71E+00	< 5.29E+03	< 1.71E+00	< 4.81E+03	< 1.87E+00	< 1.96E+03	< 1.32E+00	< 1.31E+03	< 1.71E+00
Np-239	< 1.00E-01	< 3.07E+02	< 9.13E-02	< 2.82E+02	< 9.70E-02	< 2.73E+02	< 1.55E-02	< 1.62E+01	< 1.42E-02	< 1.41E+01	< 1.42E-02
Pu-239	< 3.69E+02	< 1.13E+06	< 3.41E+02	< 1.05E+06	< 3.68E+02	< 1.03E+06	< 5.60E+01	< 5.86E+04	< 6.05E+01	< 6.00E+04	< 7.03E+1
Am-241	< 1.53E-01	< 4.69E+02	< 1.41E-01	< 4.36E+02	< 1.43E-01	< 4.02E+02	< 2.62E-02	< 2.74E+01	< 2.74E-02	< 2.72E+01	< 2.50E-02
Am-243	< 5.62E-02	< 1.72E+02	< 5.25E-02	< 1.62E+02	< 5.37E-02	< 1.51E+02	< 1.05E-02	< 1.10E+01	< 1.24E-02	< 1.23E+01	< 1.32E-02
Cm-243	< 2.76E-01	< 8.46E+02	< 2.55E-01	< 7.89E+02	< 2.64E-01	< 7.42E+02	< 3.63E-02	< 3.80E+01	< 4.32E-02	< 4.29E+01	< 3.72E-02
Cm-245	< 8.17E-02	< 2.50E+02	< 7.61E-02	< 2.35E+02	< 7.78E-02	< 2.19E+02	< 1.23E-02	< 1.29E+01	< 1.34E-02	< 1.33E+01	< 1.17E-02

Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0

Appendix G. Glass Analysis by Acid Dissolution, Continued

ICP-MS Data Acid Dissolution Analysis

	Half-life (years)	Rad1 (ug/L)	wt%	(uCi/g)	Rad2 (ug/L)	wt%	(uCi/g)	Rad3 (ug/L)	wt%	(uCi/g)	ARG1 (ug/L)	wt%	ARG2 (ug/L)	wt%	ReagBlnk (ug/L)
mass 230	< 0.09	< 2.8E-05			< 0.09	< 2.8E-05		< 0.09	< 2.5E-05		< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 231(Pa)	3.28E+04	< 0.09	< 2.8E-05	< 1.3E-02	< 0.09	< 2.8E-05	< 1.3E-02	< 0.09	< 2.5E-05	< 1.3E-02	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 232 (Th)	1.40E+10	22.13	6.8E-03	7.5E-06	3.79	1.2E-03	1.3E-06	32.88	9.2E-03	1.1E-05	5.53	5.8E-04	3.68	3.6E-04	7.87
mass 233 (U)	1.59E+00	< 0.09	< 2.8E-05	< 2.7E-03	< 0.09	< 2.8E-05	< 2.7E-03	< 0.09	< 2.5E-05	< 2.6E-03	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 234 (U)	2.45E+00	< 0.09	< 2.8E-05	< 1.7E-03	< 0.09	< 2.8E-05	< 1.7E-03	< 0.09	< 2.5E-05	< 1.7E-03	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 235 (U)	7.04E+08	2.56	7.9E-04	1.7E-05	2.18	6.8E-04	1.5E-05	2.09	5.9E-04	1.3E-05	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 236 (U)	2.34E+07	0.13	4.0E-05	2.6E-05	< 0.09	< 2.8E-05	< 1.8E-05	< 0.09	< 2.5E-05	< 1.7E-05	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 237 (Np)	2.14E+06	1.38	4.2E-04	3.0E-03	1.00	3.1E-04	2.2E-03	0.77	2.2E-04	1.6E-03	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 238 (U)	4.47E+09	398.76	1.2E-01	4.1E-04	354.94	1.1E-01	3.7E-04	352.66	9.9E-02	3.5E-04	7.34	7.7E-04	12.34	1.2E-03	4.36
mass 239 (Pu)	2.41E+04	9.39	2.9E-03	1.8E+00	7.55	2.3E-03	1.4E+00	5.98	1.7E-03	1.1E+00	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 240 (Pu)	6.56E+03	0.51	1.6E-04	3.5E-01	0.52	1.6E-04	3.6E-01	0.48	1.3E-04	3.2E-01	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 241 (Am)	4.33E+02	0.23	7.1E-05	2.5E+00	0.22	6.9E-05	2.4E+00	0.16	4.6E-05	1.7E+00	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 242 (Pu)	3.76E+05	< 0.09	< 2.8E-05	< 1.1E-03	< 0.09	< 2.8E-05	< 1.1E-03	< 0.09	< 2.5E-05	< 1.0E-03	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 243 (Am)	7.37E+03	< 0.09	< 2.8E-05	< 5.5E-02	< 0.09	< 2.8E-05	< 5.5E-02	< 0.09	< 2.5E-05	< 5.3E-02	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 244 (Cm)	1.81E+01	< 0.09	< 2.8E-05	< 2.2E+01	< 0.09	< 2.8E-05	< 2.2E+01	< 0.09	< 2.5E-05	< 2.2E+01	< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 245	< 0.09	< 2.8E-05			< 0.09	< 2.8E-05		< 0.09	< 2.5E-05		< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 246	< 0.09	< 2.8E-05			< 0.09	< 2.8E-05		< 0.09	< 2.5E-05		< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
mass 247	< 0.09	< 2.8E-05			< 0.09	< 2.8E-05		< 0.09	< 2.5E-05		< 0.90	< 9.4E-05	< 0.90	< 8.9E-05	< 0.90
Mass Number		(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(uCi/g)	(ug/L)	wt%	(ug/L)	wt%	(ug/L)
81		10.71	3.3E-03		23.44	7.2E-03		7.37	< 2.1E-03		73.66	< 7.7E-03	73.66	< 7.3E-03	< 73.66
82		< 0.70	< 2.2E-04		< 0.70	< 2.2E-04		< 0.70	< 2.0E-04		7.05	< 7.4E-04	7.05	< 7.0E-04	< 7.05
83		1.05	3.2E-04		0.90	2.8E-04		0.94	2.7E-04		8.52	< 8.9E-04	8.52	< 8.4E-04	< 8.52
84		88.00	2.7E-02		78.18	2.4E-02		83.77	2.4E-02		7.22	< 7.5E-04	7.22	< 7.2E-04	< 7.22
85		2.94	9.0E-04		3.20	9.9E-04		2.68	7.5E-04		34.62	< 3.6E-03	37.48	< 3.7E-03	< 0.68
86		1633.48	5.0E-01		1461.98	4.5E-01		1494.21	4.2E-01		3.92	< 4.1E-04	3.48	< 3.5E-04	< 3.48
87		1047.77	3.2E-01		994.84	3.1E-01		1012.32	2.8E-01		16.91	< 1.8E-03	15.69	< 1.6E-03	< 5.58
88		14363.08	4.4E+00		13011.01	4.0E+00		12600.86	3.5E+00		36.18	< 3.8E-03	34.10	< 3.4E-03	< 3.42
89		20.99	6.4E-03		18.81	5.8E-03		13.90	3.9E-03		7.91	< 8.3E-04	8.03	< 8.0E-04	< 1.63
90		457.43	1.4E-01		431.70	1.3E-01		828.30	2.3E-01		503.89	< 5.3E-02	485.00	< 4.8E-02	< 11.80
91		146.88	4.5E-02		127.15	3.9E-02		243.33	6.8E-02		112.11	< 1.2E-02	121.45	< 1.2E-02	< 2.76
92		217.18	6.7E-02		202.52	6.3E-02		486.42	1.4E-01		187.76	< 2.0E-02	182.63	< 1.8E-02	< 5.70
93		70.97	2.2E-02		61.29	1.9E-02		61.31	1.7E-02		54.19	< 5.7E-03	56.02	< 5.6E-03	< 0.86
94		216.45	6.6E-02		194.38	6.0E-02		448.18	1.3E-01		181.97	< 1.9E-02	181.77	< 1.8E-02	< 5.76
95		1.76	5.4E-04		1.70	5.3E-04		127.72	3.6E-02		< 1.56	< 1.6E-04	< 1.56	< 1.5E-04	< 1.56
96		80.04	2.5E-02		69.62	2.2E-02		249.67	7.0E-02		28.06	< 2.9E-03	30.97	< 3.1E-03	< 2.81
97		0.77	2.4E-04		0.76	2.4E-04		78.99	2.2E-02		< 2.53	< 2.7E-04	< 2.53	< 2.5E-04	< 2.53
98		1.40	4.3E-04		1.35	4.2E-04		208.85	5.9E-02		< 1.13	< 1.2E-04	< 1.13	< 1.1E-04	< 1.13
99 (Tc-99)	2.13E+05	0.18	5.5E-05	9.4E-03	0.16	4.9E-05	8.2E-03	0.18	5.1E-05	9.2E-03	0.03	< 3.1E-06	0.12	< 1.2E-05	< 0.17
100		0.84	2.6E-04		0.92	2.8E-04		78.10	2.2E-02		< 2.14	< 2.2E-04	< 2.14	< 2.1E-04	< 2.14
101		11.83	3.6E-03		10.59	3.3E-03		11.41	3.2E-03		< 0.34	< 3.5E-05	< 0.34	< 3.3E-05	< 0.34
102		12.61	3.9E-03		11.62	3.6E-03		11.86	3.3E-03		< 1.37	< 1.4E-04	< 1.37	< 1.4E-04	< 1.37
103		4.55	1.4E-03		4.02	1.2E-03		5.09	1.4E-03		< 1.05	< 1.1E-04	< 1.05	< 1.0E-04	< 1.05
104		15.62	4.8E-03		15.00	4.6E-03		19.59	5.5E-03		< 0.54	< 5.6E-05	< 0.54	< 5.3E-05	< 0.54
105		6.46	2.0E-03		6.48	2.0E-03		7.98	2.2E-03		< 1.24	< 1.3E-04	< 1.24	< 1.2E-04	< 1.24
106		11.28	3.5E-03		10.61	3.3E-03		27.62	7.8E-03		14.77	< 1.5E-03	12.85	< 1.3E-03	< 1.55
107		94.18	2.9E-02		98.27	3.0E-02		145.58	4.1E-02		4.14	< 4.3E-04	2.35	< 2.3E-04	< 0.62
108		4.87	1.5E-03		4.78	1.5E-03		10.43	2.9E-03		4.69	< 4.9E-04	3.88	< 3.8E-04	< 0.35
109		86.06	2.6E-02		89.14	2.8E-02		135.62	3.8E-02		< 1.32	< 1.4E-04	< 1.32	< 1.3E-04	< 1.32
110		8.99	2.8E-03		9.00	2.8E-03		14.77	4.2E-03		3.66	< 3.8E-04	4.39	< 4.4E-04	< 1.96
111		3.74	1.1E-03		3.59	1.1E-03		3.66	1.0E-03		< 0.93	< 9.7E-05	< 0.93	< 9.2E-05	< 0.96
112		9.10	2.8E-03		8.38	2.6E-03		8.52	2.4E-03		1.44	< 1.5E-04	1.80	< 1.8E-04	< 2.04
113		< 12.16	< 3.7E-03		< 12.16	< 3.8E-03		< 12.16	< 3.4E-03		< 121.62	< 1.3E-02	< 121.62	< 1.2E-02	< 121.62
114		7.58	2.3E-03		7.54	2.3E-03		7.05	2.0E-03		< 2.61	< 2.7E-04	< 2.61	< 2.6E-04	< 2.61
115		< 514.13	1.6E-01		< 514.13	1.6E-01		< 514.13	1.6E-01		< 5141.35	< 5.4E-01	< 5141.35	< 5.4E-01	< 5141.35
116		4.13	1.3E-03		3.25	1.0E-03		4.06	1.1E-03		8.36	< 8.7E-04	9.26	< 9.2E-04	< 1.47
117		2.75	8.4E-04		2.03	6.3E-04		1.93	5.4E-04		9.66	< 1.0E-03	10.35	< 1.0E-03	< 1.33
118		7.47	2.3E-03		5.87	1.8E-03		6.22	1.7E-03		26.57	< 2.8E-03	28.67	< 2.8E-03	< 2.13
119		9.44	2.9E-03		8.49	2.6E-03		9.79	2.8E-03		9.71	< 1.0E-03	11.00	< 1.1E-03	< 0.82
120		9.95	3.0E-03		8.48	2.6E-03		8.42	2.4E-03		35.69	< 3.7E-03	39.06	< 3.9E-03	< 2.63
121		0.92	2.8E-04		< 0.66	< 2.0E-04		1.06	3.0E-04		< 6.59	< 6.9E-04	< 6.59	< 6.5E-04	< 6.59
122		2.37	7.3E-04		1.70	5.3E-04		1.95	5.5E-04		6.28	< 6.6E-04	6.98	< 6.9E-04	< 1.71
123		0.86	2.6E-04		0.63	2.0E-04		0.81	2.3E-04		< 2.65	< 2.8E-04	< 2.65	< 2.6E-04	< 2.65
124		1.80	5.5E-04		1.52	4.7E-04		1.70	4.8E-04		5.79	< 6.1E-04	6.46	< 6.4E-04	< 1.79
125		0.59	1.8E-04		0.40	1.3E-04		0.77	2.2E-04		0.41	< 4.3E-05	0.46	< 4.6E-05	< 0.37
126		0.93	2.9E-04		0.93	2.9E-04		0.94	2.6E-04		< 1.79	< 1.9E-04	< 1.79	< 1.8E-04	< 1.79
127		< 0.10	< 3.2E-05		< 0.10	< 3.2E-05		0.23	6.6E-05		< 1.04	< 1.1E-04	< 1.04	< 1.0E-04	< 1.18
128		4.99	1.5E-03		4.44	1.4E-03		7.87	2.2E-03		< 7.21	< 7.5E-04	< 7.21	< 7.2E-04	< 7.21
129		< 0.31	< 9.4E-05		< 0.31	< 9.4E-05		< 0.31	< 8.6E-05		3.26	< 3.4E-04	3.05	< 3.0E-04	< 3.05
130		13.76	4.2E-03		11.65	3.6E-03		20.10	5.7E-03		1.24	< 1.3E-04	0.72	< 7.1E-05	< 0.72
131		< 1.17	< 3.6E-04		< 1.17	< 3.6E-04		< 1.17	< 3.3E-04		< 11.67	< 1.2E-03	< 11.67	< 1.2E-03	< 11.67
132		< 0.29	< 9.0E-05		< 0.29	< 9.1E-05		0.34	9.5E-05		< 2.93	< 3.1E-04	< 2.93	< 2.9E-04	< 2.93
133		2													

Crucible Scale Vittrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

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Appendix G. Glass Analysis by Acid Dissolution, Continued,

ICP-MS Data Acid Dissolution Analysis

Mass Number	Rad1 (ug/L)	wt%	Rad2 (ug/L)	wt%	Rad3 (ug/L)	wt%	ARG1 (ug/L)	wt%	ARG2 (ug/L)	wt%	ReagBlink (ug/L)
135	93.23	2.9E-02	80.02	2.5E-02	102.28	2.9E-02	61.22	6.4E-03	63.31	6.3E-03	8.28
136	7.59	2.3E-03	7.67	2.4E-03	35.97	1.0E-02	71.79	7.5E-03	71.90	7.1E-03	10.83
137	183.59	5.6E-02	158.30	4.9E-02	188.80	5.3E-02	102.86	1.1E-02	108.91	1.1E-02	15.23
138	133.05	4.1E-02	130.75	4.0E-02	391.08	1.1E-01	620.52	6.5E-02	649.24	6.4E-02	102.39
139	23.85	7.3E-03	22.13	6.8E-03	16.25	4.6E-03	5.68	5.9E-04	6.73	6.7E-04	< 1.68
140	40.56	1.2E-02	33.56	1.0E-02	531.31	1.5E-01	8.14	8.5E-04	9.56	9.5E-04	< 1.26
141	21.68	6.6E-03	19.38	6.0E-03	13.23	3.7E-03	< 1.72	< 1.8E-04	1.72	1.7E-04	< 1.72
142	39.24	1.2E-02	34.01	1.1E-02	851.31	2.4E-01	2.29	2.4E-04	2.64	2.6E-04	< 1.19
143	20.30	6.2E-03	17.71	5.5E-03	385.32	1.1E-01	< 2.13	< 2.2E-04	2.13	< 2.1E-04	< 2.13
144	22.85	7.0E-03	21.40	6.6E-03	701.02	2.0E-01	1.13	1.2E-04	1.53	1.5E-04	< 0.08
145	14.16	4.3E-03	12.47	3.9E-03	290.53	8.2E-02	< 0.69	< 7.2E-05	0.69	< 6.8E-05	< 0.69
146	12.54	3.8E-03	11.46	3.5E-03	557.23	1.6E-01	< 1.34	< 1.4E-04	1.34	< 1.3E-04	< 1.34
147	11.11	3.4E-03	9.55	3.0E-03	7.04	2.0E-03	< 0.42	< 4.4E-05	0.42	< 4.2E-05	< 0.42
148	7.28	2.2E-03	6.92	2.1E-03	213.25	6.0E-02	< 0.69	< 7.2E-05	0.69	< 6.8E-05	< 0.69
149	0.58	1.8E-04	0.52	1.6E-04	0.38	1.1E-04	< 0.46	< 4.8E-05	0.46	< 4.6E-05	< 0.46
150	9.20	2.8E-03	7.57	2.3E-03	215.90	6.1E-02	< 0.69	< 7.2E-05	0.69	< 6.8E-05	< 0.69
151	0.91	2.8E-04	0.93	2.9E-04	0.63	1.8E-04	< 0.34	< 3.6E-05	0.34	< 3.4E-05	< 0.34
152	2.41	7.4E-04	2.04	6.3E-04	1.58	4.4E-04	< 0.76	< 8.0E-05	0.76	< 7.6E-05	< 0.76
153	1.19	3.6E-04	1.02	3.2E-04	0.82	2.3E-04	< 1.08	< 1.1E-04	1.08	< 1.1E-04	< 1.08
154	0.83	2.6E-04	0.84	2.6E-04	1.33	3.7E-04	1.29	1.4E-04	1.31	1.3E-04	< 0.99
155	1.73	5.3E-04	1.65	5.1E-04	1.81	5.1E-04	< 1.24	< 1.3E-04	1.24	< 1.2E-04	< 1.24
156	2.91	8.9E-04	2.93	9.1E-04	36.95	1.0E-02	1.33	1.4E-04	0.01	1.5E-06	0.14
157	0.86	2.6E-04	0.72	2.2E-04	1.26	3.5E-04	< 0.85	< 8.9E-05	0.85	< 8.5E-05	< 0.85
158	2.19	6.7E-04	2.25	7.0E-04	28.50	8.0E-03	< 0.54	< 5.6E-05	0.54	< 5.3E-05	< 0.54
159	0.53	1.6E-04	0.51	1.6E-04	11.11	3.1E-03	< 1.51	< 1.6E-04	1.51	< 1.5E-04	< 1.51
160	0.56	1.7E-04	0.50	1.6E-04	21.19	6.0E-03	< 1.27	< 1.3E-04	1.27	< 1.3E-04	< 1.27
161	0.37	1.1E-04	0.33	1.0E-04	7.37	2.1E-03	0.25	2.6E-05	0.29	2.9E-05	0.11
162	0.31	9.5E-05	0.32	9.8E-05	14.75	4.1E-03	0.52	5.4E-05	0.50	5.0E-05	0.09
163	< 0.11	< 3.4E-05	< 0.11	< 3.4E-05	0.50	1.4E-04	< 1.11	< 1.2E-04	1.11	< 1.1E-04	< 1.11
164	0.21	6.5E-05	0.17	5.4E-05	4.93	1.4E-03	< 1.09	< 1.1E-04	1.09	< 1.1E-04	< 1.09
165	< 0.08	< 2.6E-05	< 0.08	< 2.6E-05	0.14	4.0E-05	< 0.84	< 8.8E-05	0.84	< 8.4E-05	< 0.84
166	0.10	3.1E-05	0.11	3.4E-05	4.50	1.3E-03	< 0.69	< 7.2E-05	0.69	< 6.8E-05	< 0.69
167	< 0.14	< 4.2E-05	< 0.14	< 4.3E-05	0.20	5.6E-05	< 1.37	< 1.4E-04	1.37	< 1.4E-04	< 1.37
168	< 0.10	< 2.9E-05	< 0.10	< 3.0E-05	< 0.10	< 2.7E-05	< 0.95	< 1.0E-04	0.95	< 9.5E-05	< 0.95
169	< 0.02	< 6.9E-06	< 0.02	< 7.0E-06	0.16	4.5E-05	< 0.23	< 2.4E-05	0.33	3.3E-05	< 0.23
170	< 0.04	< 1.4E-05	< 0.04	< 1.4E-05	< 0.04	< 1.3E-05	< 0.45	< 4.7E-05	0.45	< 4.4E-05	< 0.45
171	< 0.08	< 2.4E-05	< 0.08	< 2.4E-05	< 0.08	< 2.2E-05	< 0.77	< 8.1E-05	0.77	< 7.7E-05	< 0.77
172	< 0.00	< 1.2E-06	0.02	7.4E-06	0.04	1.1E-05	0.17	1.8E-05	0.29	2.9E-05	< 0.04
173	< 0.07	< 2.0E-05	< 0.07	< 2.0E-05	< 0.07	< 1.9E-05	< 0.66	< 6.9E-05	0.66	< 6.5E-05	< 0.66
174	< 0.07	< 2.0E-05	< 0.07	< 2.0E-05	< 0.07	< 1.8E-05	< 0.65	< 6.8E-05	0.65	< 6.5E-05	< 0.65
175	< 0.12	< 3.7E-05	< 0.12	< 3.7E-05	< 0.12	< 3.4E-05	< 1.21	< 1.3E-04	1.21	< 1.2E-04	< 1.21
176	0.05	1.5E-05	0.08	2.5E-05	0.94	2.7E-04	1.52	1.6E-04	1.17	1.2E-04	< 0.19
177	0.17	5.1E-05	0.18	5.7E-05	3.92	1.1E-03	4.99	5.2E-04	4.44	4.4E-04	0.10
178	0.21	6.6E-05	0.30	9.4E-05	5.32	1.5E-03	6.47	6.8E-04	6.28	6.2E-04	< 0.30
179	0.14	4.2E-05	0.13	4.1E-05	2.80	7.9E-04	3.08	3.2E-04	3.66	3.6E-04	< 0.24
180	0.98	3.0E-04	1.13	3.5E-04	7.47	2.1E-03	8.82	9.2E-04	8.88	8.8E-04	< 0.75
181	7.49	2.3E-03	6.27	1.9E-03	5.67	1.6E-03	12.21	1.3E-03	13.70	1.4E-03	0.30
182	158.82	4.9E-02	139.63	4.3E-02	122.24	3.4E-02	1.39	1.5E-04	1.47	1.5E-04	< 0.64
183	85.77	2.6E-02	74.61	2.3E-02	64.20	1.8E-02	< 0.91	< 9.5E-05	0.91	< 9.0E-05	< 0.91
184	187.93	5.8E-02	164.62	5.1E-02	147.99	4.2E-02	< 1.75	< 1.8E-04	1.75	< 1.7E-04	< 1.75
185	0.02	5.2E-06	0.02	5.2E-06	0.03	7.5E-06	0.04	4.2E-06	0.01	< 1.5E-06	0.17
186	175.79	5.4E-02	149.29	4.6E-02	136.72	3.8E-02	1.24	1.3E-04	1.28	1.3E-04	0.48
187	< 0.18	< 5.4E-05	< 0.18	< 5.5E-05	< 0.18	< 5.0E-05	< 1.78	< 1.9E-04	1.78	< 1.8E-04	< 1.78
188	< 0.06	< 1.8E-05	< 0.06	< 1.8E-05	< 0.06	< 1.6E-05	< 0.58	< 6.1E-05	0.58	< 5.8E-05	< 0.58
189	< 0.02	< 7.3E-06	< 0.02	< 7.4E-06	< 0.02	< 6.7E-06	< 0.24	< 2.5E-05	0.24	< 2.4E-05	< 0.24
190	< 0.05	< 1.6E-05	< 0.05	< 1.7E-05	< 0.05	< 1.5E-05	< 0.54	< 5.6E-05	0.54	< 5.3E-05	< 0.54
191	< 0.05	< 1.5E-05	< 0.05	< 1.5E-05	< 0.05	< 1.4E-05	< 0.49	< 5.2E-05	0.49	< 4.9E-05	< 0.49
192	< 0.08	< 2.3E-05	< 0.08	< 2.4E-05	< 0.08	< 2.1E-05	< 0.76	< 8.0E-05	0.76	< 7.6E-05	< 0.76
193	< 0.07	< 2.2E-05	< 0.07	< 2.2E-05	< 0.07	< 2.0E-05	< 0.72	< 7.5E-05	0.72	< 7.1E-05	< 0.72
194	0.92	2.8E-04	0.72	2.2E-04	0.57	1.6E-04	< 0.04	< 4.7E-06	0.04	< 4.5E-06	< 0.04
195	0.89	2.7E-04	0.77	2.4E-04	0.58	1.6E-04	< 0.24	< 2.5E-05	0.24	< 2.4E-05	< 0.24
196	0.61	1.9E-04	0.58	1.8E-04	0.49	1.4E-04	0.33	3.4E-05	0.12	1.2E-05	0.06
197	0.64	2.0E-04	0.42	1.3E-04	0.08	2.3E-05	0.29	3.0E-05	0.39	3.8E-05	0.14
198	1.35	4.1E-04	1.08	3.4E-04	0.81	2.3E-04	< 0.09	< 9.3E-06	0.18	1.7E-05	0.15
199	0.72	2.2E-04	0.51	1.6E-04	0.35	9.8E-05	< 1.49	< 1.6E-04	1.49	< 1.5E-04	< 1.49
200	1.36	4.2E-04	0.91	2.8E-04	0.76	2.1E-04	< 2.83	< 3.0E-04	2.83	< 2.8E-04	< 2.83
201	0.18	5.6E-05	0.14	4.3E-05	< 0.06	< 1.7E-05	< 0.60	< 6.3E-05	0.60	< 5.9E-05	< 0.60
202	1.37	4.2E-04	0.88	2.7E-04	0.62	1.8E-04	< 1.55	< 1.6E-04	1.55	< 1.5E-04	< 1.55
203	0.21	6.6E-05	< 0.18	< 5.5E-05	< 0.18	< 5.0E-05	< 1.79	< 1.9E-04	1.79	< 1.8E-04	< 1.79
204	12.98	4.0E-03	11.80	3.7E-03	10.14	2.9E-03	< 1.77	< 1.8E-04	1.77	< 1.8E-04	< 1.77
205	< 0.39	< 1.2E-04	< 0.39	< 1.2E-04	< 0.39	< 1.1E-04	< 3.92	< 4.1E-04	3.92	< 3.9E-04	< 3.92
206	211.62	6.5E-02	188.65	5.8E-02	161.30	4.5E-02	< 2.61	< 2.7E-04	2.61	< 2.6E-04	< 2.61
207	200.36	6.1E-02	185.09	5.7E-02	146.39	4.1E-02	< 6.24	< 6.5E-04	6.24	< 6.2E-04	< 6.24
208	480.71	1.5E-01	428.00	1.3E-01	366.78	1.0E-01	< 3.89	< 4.1E-04	3.89	< 3.9E-04	< 3.89

Appendix H. Product Consistency Test

EXPERIMENTAL

Product Consistency Test

The durability of the HLW Envelope D glass was measured using the ASTM C-1285 standard nuclear waste glass durability test commonly referred to as the Product Consistency Test (PCT) [1]. This is a crushed glass leach test at 90 °C for 7 days using deionized water as leachate. The ground glass samples used for the PCT were prepared by grinding in a rotary blade grinder. This grinder contains a tungsten carbide blade and a stainless steel chamber. Tests were performed in sealed stainless steel vessels. Quadruplicate samples of the envelope D glass were tested at 90°C +/- 2°C following the ASTM-1285-97 procedure as required by the Hanford RPP/WTP contract [2]. Final leachate pH's were measured and final elemental concentrations of the filtered, acidified leachates were measured by ICP-ES. Purified ASTM Type I water obtained from a MilliQ water purification system was used as leachate in all tests. Ultrapure nitric acid was used to acidify the leachates prior to analysis.

RESULTS AND DISCUSSION

Results of 'Association of Standards and Test Methods' (ASTM) Test C 1285 – 97 Leach Test 'Product Consistency Test' (PCT) on C-106 HLW Glass

The two tables in this section show the results of the standard ASTM C 1285 –97 test on the radioactive C-106 glass. This standard test is commonly called the Product Consistency Test (PCT) and is performed at 90°C [1]. The procedure for PCT-A of the ASTM C 1285-97 was strictly followed for this test. Quadruplicate samples of the C-106 glass were used and, as prescribed by the procedure, triplicate blanks along with the standard Analytical Reference Material (ARM) glass [3]. The Environmental Assessment (EA) glass was also leached to compare its response to that for the C-106 glass [4].

In the contract, SRTC was required to subject the C-106 glass to the PCT and report the results for B, Si, and Na. The standard (ARM) glass [3] was also tested with the with the C-106 and EA glass to confirm that the test conditions for the PCT were properly controlled. It should be noted that the ARM reference glass used in the PCT is different from the previously mentioned analytical reference glass ARG-1 that was used as an analytical reference glass for glass dissolution and compositional analysis. The ARM glass is typically used in the PCT to confirm that test conditions for the PCT were properly controlled. This is accomplished by comparison of the ARM glass leach test data to historical databases from previous PCT tasks. Table 1 gives the average concentrations in ppm of B, Si, and Na, in the final leachates after the tests. The averages of the final pH values of the leachates are also presented. The concentrations have been corrected for the acidification dilutions of the leachates as required by the ASTM procedure. Note that the raw data that is the bases of these averages are shown in Tables 3, 4 and 5 for the radioactive C-106 glass, the EA glass and the ARM glass, respectively, that are located at the end of this Appendix H.

Appendix H, Table 1. Average Concentrations (ppm) of B, Si, and Na, and the Final pH from the 90°C PCT.

Sample ID	B	Si	Na	pH (b)
Blanks(a)	0.04	<0.15	<0.15	6.8
ARM(a)	18.9	70.1	40.8	9.8
EA Glass(c)	565.	908.	1598.	11.5
C-106 Glass(c)	13.3	90.8	84.4	10.7

(a) Based on triplicate samples.

(b) Initial pH of the leach water was 6.57

(c) Based on quadruplicate samples.

The results for the blanks indicate that contamination of the leachates from possible impurities in the water or impurities on the stainless steel vessels was negligible. The results for the standard ARM-1 glass were compared to a control chart based on results for previous Product Consistency Tests on this standard glass [4]. This comparison is part of the ASTM procedure. The results for the ARM glass were between the lower and upper control limits (See Table 5 near the end of this Appendix H for PCT data sheet on ARM glass) indicating that the test conditions were properly controlled. Standard solutions containing B, Si, and Na were submitted for analysis with the leachates. The measured results agreed within 10% of the known values shown in Table 5, indicating that the analyses were sufficiently accurate. Thus the results of the PCT are acceptable.

The final pH is an approximate indication of the durability of the glass in a PCT. The higher the final pH, the lower the durability. The measured concentrations are a much more accurate indication. Based on the results in Table 1 the C-106 glass appears slightly more durable than the EA glass. Normalized mass losses are the best indication of the durability of a glass in a PCT. Normalization accounts for the concentration of an element in the glass. The normalized release is a measure of the total mass of glass leached in a PCT based on a specific element in the glass. In the PCT, the glass is carefully sieved through standard mesh size sieves so that the surface area of the glass is reproducible from test to test. The exposed surface area of the glass in a PCT has been estimated by assuming that the particles are spherical and that the distribution of particle sizes is Gaussian [5]. The size of the holes in the 100 and 200 mesh sieves are 0.149 mm and 0.074 mm, respectively. Thus the diameter of the spheres range between these two

values with an average value of 1.12×10^{-4} m. Based on these assumptions the exposed surface area has been calculated to be 0.02 m^2 per gram of sieved glass.

The normalized mass loss in terms of grams of glass leached is calculated using the following equation,

$$NR_i = (C_i/C_{ig})/0.02 \times 10^3$$

Where NR_i is normalized release based on element i, in grams of glass leached per square meter of glass exposed in the PCT. C_i is the concentration of element i in ppm in the leachate and C_{ig} is the weight percent of element i in the glass. The PCT procedure prescribes that for every gram of glass, there is exactly 10 mL of leachate; thus there is 0.02 m^2 of glass surface area per 10 mL of leachate. The factor of 1000 in the denominator results from C_i being in ppm, C_{ig} in weight percent, and the test condition of 10 mL per 0.02 m^2 of glass.

Table 2 presents the normalized releases calculated from the PCT data and from the measured composition of the C-106 glass (see Table 8 in text). Results for the EA glass are also included. Table 2 presents the averages and standard deviations based on quadruplicate samples. The last column of Table 2 presents the results for the EA glass minus two standard deviations calculated from the data for the EA glass. According to the Waste Acceptance Product Specifications for Vitrified High Level Waste Forms (WAPS) cited in the RPP-WTP Contract Specification 1, to be acceptable the release from the C-106 glass should be less than that this value. As shown in Table 2 this is clearly the case.

**Appendix H, Table 2. Normalized Mass Losses (g glass/m^2) Based on B, Si, and Na,
For C-106 and EA Glass in a 90°C PCT**

	C-106 Glass	EA Glass	EA-2STD. DEV.
Element	Normalized Release^a	Normalized Release^a	Normalized Release
B	0.48±0.02	8.14±0.50	7.14
Si	0.22±0.00	1.99±0.05	1.89
Na	0.45±0.01	6.38±0.34	5.70

(a) Based on quadruplicate Product Consistency Tests.

CONCLUSIONS

The ASTM standard Product Consistency Test (PCT) was performed at 90°C on the C-106 radioactive glass and the Environmental Assessment (EA) glass. The PCT results indicate that normalized released for B, Si, and Na were well below those for the EA glass even after two standard deviations are subtracted from the average for the EA glass.

REFERENCES FOR PCT, Appendix H

1. Annual Book of ASTM Standards, Vol. 12.01. Association of Standards and Test Methods Committee C26, "Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)," **ASTM C1285-97**, (1998), pp. 774-791.
2. Note: This work was initiated under the Tank Waste Remediation System Privatization Contract with DOE:
TWRS Privatization, **Contract No. DE-AC27-96RL13308**, Mod. No. M014, Section C: Statement of Work, November 2000. (See: <http://www.hanford.gov/doe/contracts/de-ac06-96rl13308/conformed/index.html>)

The TWRS Contract was replaced by the Waste Treatment Plant DOE Contract in early 2001 with DOE:

WTP Contract, **Contract No. DE-AC27-01RV14136**, Mod. No. M008, Section C: Statement of Work, September 2001. (See: <http://www.hanford.gov/orp/contracts/de-ac27-01rv14136/toc.html>)

3. G. B. Mellinger and J. L. Daniel, "Approved Reference and Testing Materials for Use in Nuclear Waste Management Research and Development Programs", **PNL-4955-1**, Pacific Northwest Laboratory, December, 1983.
 4. "Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material (U)", C. M. Jantzen, N. E. Bibler, D. C. Beam, C. L. Crawford and M. A. Pickett, **WSRC-TR-92-346, Rev. 1**, June 1, 1993.
- C. M. Jantzen, M. A. Pickett, K. G. Brown, T. B. Edwards, and D. C. Beam, "Process/Product Models for the DWPF: Part 1. Predicting the glass Durability from Composition Using a Thermodynamic Energy Hydration Model (THERMO) (U)", Volume 2: Appendix G, Pages G9 – G11, **WSRC-TR-93-672, Rev. 1** (1995).

Crucible Scale Vittrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0

Appendix H, Table 3. Data for PCT @ 90° for Env. D, C-106 Glass

PROCEDURE ASTM-1285-97

DATA AND RESULTS FOR 7 DAY PCT TEST

TEST NAME

90C PCT WITH RPP ENVELOPE D GLASS

GLASS RPP ENVELOPE D HLW GLASS C-106 GLASS

LEACHATE DILUTION FACTORS:

PCT A, SA/V = 2000M-1

SPREADSHEET REDUCED TO 61% FOR PRINT OUT

DATE IN OVEN

DATE OUT OF OVEN

TIME IN OVEN

TIME OUT OF OVEN

INITIAL pH = 6.80

1135

1235

(OST STARTED 4/1/01)

RAW EXPERIMENTAL DATA:

SAMPLE	WEIGHTS	GLASS	WEIGHT	INITIAL	FINAL	W/H2O	INIT.	WATER	FINAL
NAME	EMPTY	W/GLASS	IN PCT	IN PCT	IN PCT	VOL.(ML)	LOSS	LOSS	PH
PA08 Blank 1	NA	N.A.	337.873	337.822	337.799	17.000	0.023	6.96	6.96
PA40 Blank 2	NA	N.A.	340.788	340.587	340.571	17.000	0.011	7.19	7.19
PA422 Blank 3	NA	N.A.	341.006	340.965	340.939	17.000	0.026	6.68	6.68

BLANK AVERAGE

339.788	339.786	339.756	15.374	0.030	10.60
339.337	339.296	339.274	15.361	0.022	10.75
341.256	341.061	341.039	15.310	0.022	10.78
338.561	338.551	338.522	15.683	0.029	10.67

SAMPLES

PA1410-RPP-D1	190.624	192.161	1.537	339.788	339.786	339.756	15.374	0.030	10.60	161752	7.557	53.455	48.841	9.858	6.425
PA1411-RPP-D2	191.813	193.349	1.536	339.337	339.296	339.274	15.361	0.022	10.75	161757	8.194	55.628	51.074	10.226	6.510
PA1412-RPP-D3	190.795	192.326	1.531	341.256	341.061	341.039	15.310	0.022	10.78	161762	8.081	53.780	51.074	10.152	6.391
PA1413-RPP-D4	190.616	192.184	1.568	338.561	338.551	338.522	15.683	0.029	10.67	161766	7.747	53.025	49.642	9.869	6.395

CALCULATED RESULTS: PH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR

SAMPLE	GLASS	INIT.	FINAL	%	PH VALUES
NAME	WEIGHT	VOL.(ML)	VOL.(ML)	LOSS	INITIAL
PA1410-RPP-D1	1.537	15.374	15.344	0.1951	6.80
PA1411-RPP-D2	1.536	15.361	15.339	0.1432	6.80
PA1412-RPP-D3	1.531	15.310	15.288	0.1437	6.80
PA1413-RPP-D4	1.568	15.683	15.654	0.1849	6.80

AVERAGE

STANDARD DEVIATION

REL. STD. DEVIATION (%)

13.29	90.83	84.42	16.87	10.82
0.50	1.93	1.86	0.32	0.09
3.73	2.12	2.21	1.90	0.86
13.86	21.103	9.332	1.924	0.019
0.96	0.43	0.90	0.88	
0.48	0.22	0.45	0.44	
0.92	0.05	0.01	0.01	
0.84	0.01	0.02	0.02	

QUALITY ASSURANCE INFORMATION:

LABS=CELL 14 AND B-111

PH METER ACCUMETAB 15

BALANCE SER.#=GT1098

OVEN SER.CELL 14

TEMPERATURE READOUT: GT-1188 FLUKE RTD

FILTER SIZE:45 MICRON

STANDARD COMPOSITION (PPM)

S-1	161750	20.03	52.77	84.60	10.12	4.34
S-2	161755	19.95	52.43	83.86	10.02	4.28
S-3	161760	19.82	52.06	83.31	9.89	4.11
S-4	161765	19.96	52.37	83.91	10.01	4.23
S-5	161768	19.98	52.39	83.71	9.96	4.32
S-6	161769	19.99	52.68	84.40	10.03	4.16

STANDARD COMPOSITION (PPM)

S-1	161750	20.03	52.77	84.60	10.12	4.34
S-2	161755	19.95	52.43	83.86	10.02	4.28
S-3	161760	19.82	52.06	83.31	9.89	4.11
S-4	161765	19.96	52.37	83.91	10.01	4.23
S-5	161768	19.98	52.39	83.71	9.96	4.32
S-6	161769	19.99	52.68	84.40	10.03	4.16

STANDARD COMPOSITION (PPM)

S-1	161750	20.03	52.77	84.60	10.12	4.34
S-2	161755	19.95	52.43	83.86	10.02	4.28
S-3	161760	19.82	52.06	83.31	9.89	4.11
S-4	161765	19.96	52.37	83.91	10.01	4.23
S-5	161768	19.98	52.39	83.71	9.96	4.32
S-6	161769	19.99	52.68	84.40	10.03	4.16

STANDARD COMPOSITION (PPM)

S-1	161750	20.03	52.77	84.60	10.12	4.34
S-2	161755	19.95	52.43	83.86	10.02	4.28
S-3	161760	19.82	52.06	83.31	9.89	4.11
S-4	161765	19.96	52.37	83.91	10.01	4.23
S-5	161768	19.98	52.39	83.71	9.96	4.32
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Crucible Scale Vitrification of
Pretreated C-106 Sludge Mixed with
Secondary Wastes

WSRC-TR-2001-00252
SRT-RPP-2001-00068, Rev.0

PROCEDURE ASTM-1285-97	
DATA AND RESULTS FOR 7 DAY PCT TEST	
PCT A. SAV/V = 2000M-1	TEST NAME 90C PCT WITH RPP ENVELOPE D GLASS
SPREADSHEET REDUCED TO 61% FOR PRINT OUT	GLASS ENVIRONMENTAL ASSESSMENT (EA) GLASS
	LEACHATE DILUTION FACTORS: BLANKS: 5 ML SPL AND 0.05 ML CONC HNO3 DF = 10.1/1.0 = 1.01 RPP ARM. STDS 1, 2, 4, & 5: 3 ML SPL/0.05 ML CONC HNO3, 2 ML ASTM H2O DF = 5.05/3 = 1.683 EA: 5 ML SPL/9.5 ML ASTM H2O/0.1 ML CONC HNO3 STDS: 3 & 6: 6 ML STANDARD DF = 10.1/5 = 2.02
DATE IN OVEN 3/27/01	1135
DATE OUT OF OVEN 4/3/01	1235
TIME IN OVEN TIME OUT OF OVEN (OST STARTED 4/17/01)	
INITIAL pH = 6.80	

RAW EXPERIMENTAL DATA:				SRTC				RESULTS (ppm) FOR ACIDIFIED LEACHATES.			
SAMPLE	EMPTY	W/GLASS	WEIGHT	INITIAL	FINAL	WATER	FINAL	ADS	RESULTS		
NAME			W/H2O	IN PCT	IN PCT	LOSS	PH	NUMBER	B	SI	LI
P406 Blank 1	NA	N.A.	337.873	337.822	337.799	17.000	0.023	6.86	161751	<0.030	<0.150
P420 Blank 2	NA	N.A.	340.788	340.582	340.555	17.000	0.027	7.19	161756	<0.030	<0.150
P422 Blank 3	NA	N.A.	341.006	340.965	340.939	17.000	0.026	6.68	161761	0.056	<0.150
BLANK AVERAGE											
SAMPLES											
P414-EA-D1	191.269	192.844	1.575	339.180	339.059	15.750	11.49	161754	25.921	43.618	74.010
P417-EA-D2	191.346	192.866	1.640	340.952	340.796	16.400	0.026	11.51	161759	29.907	46.468
P421-EA-D3	191.003	192.868	1.585	338.987	338.958	15.850	0.027	11.45	161764	27.619	44.456
P423-EA-D4	190.645	192.258	1.613	339.898	339.713	16.130	0.021	11.45	161767	28.502	45.164
CALCULATED RESULTS: pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED											
FOR BLANKS											
PH VALUES				% LOSS				CONCENTRATIONS (PPM)			
SAMPLE	GLASS	INITIAL	FINAL	VOL.(ML)	LOSS	PH	INITIAL	B	SI	Na	LI
NAME	WEIGHT	1.575	15.750	15.723	0.17	6.80	11.49	521.58	881.08	1495.00	172.41
P414-EA-D1	1.575	16.400	16.374	0.16	6.80	11.51	11.51	604.12	938.65	1701.22	192.85
P417-EA-D2	1.640	15.850	15.823	0.17	6.80	11.45	11.45	557.90	898.01	1577.54	183.19
P421-EA-D3	1.585	16.130	16.109	0.13	6.80	11.45	11.45	575.74	912.31	1618.46	187.88
P423-EA-D4	1.613	AVERAGE		15.98.06		24.37		564.84	907.52	1598.06	184.08
AVERAGE	STANDARD DEVIATION		REL. STD. DEVIATION (%)		34.55		85.83		24.37	85.83	8.73
REL. STD. DEVIATION (%)	NORMALIZED CALCULATIONS:		ELEMENTAL WEIGHT PERCENT IN GLASS		16.28		3.98		6.12	2.69	5.37
				NORMALIZED MASS LOSS (GRAMS GLASS/LITER)		16.28		3.98		12.76	9.39
				NORMALIZED MASS LOSS (GRAMS GLASS/METER 2)		8.14		1.99		6.38	4.70
				NORMALIZED MASS LOSS STANDARD DEVIATION		0.50		0.05		0.34	0.22
				NORMALIZED MASS LOSS PLUS 2 SIGMA		1.00		0.11		0.69	0.45
QUALITY ASSURANCE INFORMATION:											
LABS=CELL 14 AND B-111				STANDARD RESULTS:				LIMS NO.			
PH METER ACCUMET AB 15				EXPIRATION DATE 2/28/02				ICP RESULTS (PPM)			
BALANCE SER.#=GT1098				LOT NO. 102202				B			
OVEN SER.CELL 14				S-1				SI			
TEMPERATURE READOUT: GT-1188 FLUKE RTD				S-2				Na			
FILTER SIZE: 45 MICRON				S-3				LI			
RESEARCHER: NED BIBLER				S-4				RESULTS CORRECTED FOR DILUTION FACTOR OF 1.68			
				S-5				161750			
				S-6				20.0+/-0.1			
								50.0+/-0.3			
								81.0+/-0.4			
								10.00+/-0.05			
								4.00+/-0.02			

Appendix H, Table 4. Data for PCT @ 90°C for Environmental Assessment (EA) Glass

Appendix I. Toxicity Characteristic Leaching Procedure

1.0 Executive Summary

Analysis of extract from Toxicity Characteristic Leaching Procedure (TCLP) tests of the Tank C-106 glass waste form showed the concentration of Resource Conservation and Recovery Act (RCRA) metals were less than the Universal Treatment Standard (UTS) limits.^{3,4} All target analytes (including RCRA metals) were determined to the maximum allowable lower limit of detection (MALLD) given in the task specification¹ to this study.

2.0 Introduction and Background

Because the Tank C-106 radioactive waste is a “listed” Mixed Waste, the resulting immobilized High-Level Waste (IHLW) must be considered to be listed Mixed Waste. Future efforts to dispose of the IHLW will undoubtedly require that the waste form be shown to retain the hazardous components and not release them to the environment. Therefore, in addition to analyzing the glass waste form directly, the waste form will be characterized by a TCLP.

3.0 Experimental

After crushing the glass waste form, five-gram samples were collected. The first sample (Sample 1) was used to determine which extraction fluid should be used. As a result, Extraction Fluid # 1 was used (pH 4.93 acetic acid buffer). The extraction was performed on two of the samples, five grams of a TCLP-certified soil standard, and a blank. Because of the high dose rates associated with the waste form, activities up to filtration of the extracts were performed remotely in a shielded cell facility. A summary of the resulting subsamples has been given in Table III.1.

Appendix I, Table III.1. Preparation and Analytical Methods Used to Determine Analytes in TCLP Extracts

Sample	Description	Acid-Digestion?	Analytical Methods
1	Determine Appropriate Extraction Fluid	-	-
2a	Sample	Yes	ICP-ES
		No	AA, IC, ISE
Spike	Matrix Spike (Made from Sample 2a)	Yes	ICP-ES
		No	AA, IC, ISE
3a	Sample	Yes	ICP-ES
		No	AA, IC, ISE
3b	Sample	No	ICP-ES, AA, IC, ISE
TCLP a	TCLP Blank	Yes	ICP-ES
		No	AA, IC, ISE
TCLP b	TCLP Blank	No	ICP-ES, AA, IC, ISE
Standard a	Standard	Yes	ICP-ES
		No	AA
Standard b	Standard	No	ICP-ES, AA
Blank	Digestion Blank	Yes	ICP-ES

The TCLP was performed according to the standard EPA SW-846 procedure (Method 1311)² with three exceptions. First, because the sample was limited, 5 grams of sample and twenty milliliters of extraction fluid were used. Second, the temperature could not be controlled as specified in the procedure (23 ± 2 °C). As a result the extraction started at 21 °C but slowly dropped to 18 °C at the end of the TCLP. Results from the standard have been used to show the extent to which the temperature might have shifted the extract concentrations.

The third deviation from the standard method was in the acid digestion. Method 1311 states “TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes.” Sample 3 was split into two aliquots (3a and 3b) prior to the acid digestion. Acid digestion was not performed on Sample 3b. To avoid potential mercury losses, an acid digestion was not performed on any sample prior to the AA analyses.

4.0 Results

The purpose of this section is to present the results from TCLP extractions performed on the glass waste form samples. Discussions given in this section have been limited to explanations and qualifications of the results. The implications of these results have been discussed in Section 5.0.

A. Inductively-Coupled Plasma-Atomic Emission Spectroscopy

ICP-AES analyses were performed on extracts that were acid digested and on extracts directly (without an acid digestion). In addition, a matrix spike was prepared for most of the analytes determined by this method. Since recoveries were greater than 50 % and no result was within 20 % of the regulatory limits, TCLP protocol did not require an internal calibration quantitation.

1. TCLP Standard and Spike

Table IV.1 includes TCLP results from the standard soil extract and results from analysis of the spiked extract. Results from the standard soil extract were within the performance assessment (PA) limits except the chromium and antimony results. Recoveries have also been included in Table IV.1. For spiked analytes not detected in the unspiked sample, a range has been given, the lower value calculated from the unspiked sample detection limit and the higher value (thought to be more accurate in most cases) calculated assuming the unspiked concentration to be zero.

Appendix I, Table IV.1. Results from ICP-AES Analysis of the Standard Soil and the Spike TCLP Extracts

Analyte	Standard (mg/L)			Spike (mg/L)			Recovery (%)
	Digested	Direct	PA Limits	Spiked	Unspiked	Added	
Ag	0.703	0.759	0.51-1.1	0.439	0.033	0.415	98
Al	0.456	0.558	-	0.597	0.336	0.415	63
B	1.808	1.805	-	0.892	0.567	0.415	78
Ba	13.0	13.2	8.5-15	0.792	0.453	0.415	82
Be	0.531	0.528	0.37-1.2	0.375	< 0.003	0.415	89.7-90.4
Ca	29.2	30.4	-	0.678	0.239	0.415	106
Cd	1.233	1.241	0.99-2.0	0.406	< 0.006	0.415	96-98
Co	0.033	0.045	-	0.386	< 0.008	0.415	91-93
Cr	0.506	0.523	0.64-1.7	0.394	< 0.03	0.415	89-95
Cu	0.025	0.036	-	0.444	0.039	0.415	98
Fe	0.111	0.081	-	40.17	0.364	41.5	96
La	< 0.03	0.036	-	0.392	< 0.03	0.415	88-94
Li	< 0.008	<0.003	-	0.6917	0.3278	0.415	88
Mg	4.08	4.13	-	0.456	0.072	0.415	92
Mn	3.13	3.20	-	0.628	0.269	0.415	86
Mo	< 0.008	<0.003	-	0.400	< 0.008	0.415	94-96
Na	1389	1404	-	1218	1374	0.415	-
Ni	7.75	7.86	6.3-11	0.4194	< 0.03	0.415	95-101
P	0.272	0.383	-	< 0.1	< 0.1	-	-
Pb	1.18	1.27	0.43-2.8	0.433	< 0.1	0.415	74-104
Sb	1.17	1.22	1.3-2.8	0.383	< 0.2	0.415	48-92
Si	1.05	0.85	-	3.02	1.33	-	-
Sn	0.067	0.072	-	0.481	0.050	0.415	104
Sr	0.328	0.337	-	0.956	0.642	0.415	76
Ti	< 0.003	<0.001	-	< 0.003	< 0.003	-	-
Tl	< 0.4	0.202	-	0.581	< 0.4	0.415	50-140
V	< 0.008	<0.003	-	0.3944	< 0.008	0.415	93-95
Zn	6.33	6.57	4.3-9.7	0.858	0.522	0.415	81
Zr	< 0.008	0.003	-	0.086	< 0.008	-	-

2. Samples

Table IV.2 includes TCLP results from acid-digested extracts and from extracts analyzed without being digested. All results were well below the UTS limits.^{3,4} During acid digestion extracts were diluted to the extent that thallium could not be determined to the UTS limit; however, the detection limit for the direct analysis was low enough to show that the extract thallium concentration was below the UTS limit.

Appendix I, Table IV.2. Results from ICP-AES Analysis of the C-106 HLW Glass Sample
TCLP Extracts

Analyte	UTS (mg/L)	Digested (mg/L)				Direct (mg/L)	
		Samples		Blanks		Sample	TCLP Blank
		2a	3a	TCLP	Digestion	3b	
Ag	0.14	0.033	< 0.01	< 0.01	< 0.01	0.038	< 0.005
Al	-	0.34	0.17	< 0.04	< 0.04	0.225	0.093
B	-	0.57	0.34	0.56	0.061	0.352	0.563
Ba	21	0.45	0.32	0.60	< 0.008	0.336	0.619
Be	1.22	< 0.003	< 0.003	< 0.003	< 0.003	< 0.001	< 0.001
Ca	-	0.24	0.12	0.24	< 0.05	0.184	0.282
Cd	0.11	< 0.006	< 0.006	< 0.006	< 0.006	< 0.002	< 0.002
Co	-	< 0.008	< 0.008	< 0.008	< 0.008	< 0.003	< 0.003
Cr	0.60	< 0.03	< 0.03	< 0.03	< 0.03	< 0.009	< 0.009
Cu	-	0.039	0.042	0.014	< 0.008	0.061	0.018
Fe	-	0.36	0.29	0.30	0.16	0.182	0.006
La	-	< 0.03	< 0.03	< 0.03	< 0.03	< 0.01	< 0.01
Li	-	0.33	0.24	< 0.008	< 0.008	0.257	< 0.003
Mg	-	0.072	0.036	0.044	0.047	0.019	0.017
Mn	-	0.27	0.23	< 0.003	< 0.003	0.242	0.003
Mo	-	< 0.008	< 0.008	< 0.008	< 0.008	< 0.003	< 0.003
Na	-	1374	1394	1388	0.58	1424	1416
Ni	11	< 0.03	< 0.03	< 0.03	< 0.03	0.011	< 0.009
P	-	< 0.1	< 0.1	< 0.1	< 0.1	0.1	0.093
Pb	0.75	< 0.1	< 0.1	< 0.1	< 0.1	< 0.05	< 0.05
Sb	1.15	< 0.2	< 0.2	< 0.2	< 0.2	< 0.07	< 0.07
Si	-	1.3	0.47	0.30	0.57	0.385	0.123
Sn	-	0.050	0.061	< 0.04	< 0.04	0.058	0.052
Sr	-	0.64	0.45	0.011	< 0.003	0.471	0.012
Ti	-	< 0.003	< 0.003	< 0.003	< 0.003	< 0.001	< 0.001
Tl	0.20	< 0.4	< 0.4	< 0.4	< 0.4	< 0.1	< 0.1
V	1.6	< 0.008	< 0.008	< 0.008	< 0.008	< 0.003	< 0.003
Zn	4.3	0.52	0.31	0.62	0.047	0.302	0.631
Zr	-	< 0.008	< 0.008	< 0.008	< 0.008	< 0.003	< 0.003

B. Atomic Absorption Spectroscopy

As indicated previously, AAS analyses were performed on extracts without an independent acid-digestion step. In addition, a matrix spike was prepared for the three analytes determined by this method (arsenic, mercury, and selenium). Results from the AAS determinations have been presented in this section. Because recoveries were greater than 50 %, internal calibration quantitation was not required by TCLP protocol.

1. TCLP Standard and Spike

Table IV.3 includes TCLP results from the standard soil and results from analysis of the spiked extract. Results from the standard soil extract were within the performance assessment (PA) limits for arsenic and one of the mercury standard extracts. The cause for the large difference between the mercury standard results was uncertainties inherent in measuring concentrations close to method detection limits. Recoveries have also been included in Table IV.1. The recovery for arsenic was very high.

Appendix I, Table IV.3. Results from AAS Analysis of the Standard Soil and the Spike TCLP Extracts

Analyte	Standard (mg/L)			Spike (mg/L)			Recovery (%)
	a	b	PA Limits	Spiked	Unspiked	Added	
As	2.11	2.34	1.3-2.6	0.890	6.5×10^{-4}	0.415	214
Hg	0.017	0.0044	0.014-0.052	3.52	0.0187	4.15	84
Se	0.21	0.29	0.46-0.87	0.293	1.0×10^{-4}	0.415	70

2. Samples

Table IV.4 includes TCLP results from the sample and blank extraction fluid AAS analyses. All results were well below the UTS limits.^{3,4} Concentrations of arsenic in the blank extracts were higher than the arsenic concentrations in the samples. These concentrations were close to the method detection limits and were all at least three orders of magnitude less than the UTS limit. Similarly, mercury concentrations in the blanks were similar to the sample extract concentrations but these concentrations were also close to the method detection limits.

Appendix I, Table IV.4. Results from AAS Analysis of the C-106 HLW Glass Sample TCLP Extracts

Analyte	UTS (mg/L)	Samples (mg/L)			TCLP Blanks (mg/L)	
		2a	3a	3b	a	b
As	5.0	6.5×10^{-4}	5.5×10^{-4}	< 0.005	0.0026	0.0020
Hg	0.025	0.0187	0.0088	< 0.01	0.0055	< 0.01
Se	5.7	1.0×10^{-4}	< 0.005	< 0.005	< 0.005	< 0.005

C. Ion Chromatography

Results from the IC analyses have been given in Table IV.5. Acetate from the acetic acid extraction fluid is thought to have interfered with the fluoride measurement. Therefore, results from the ISE should be used for determining the fluoride concentrations in the extracts. Nitrate in

the samples appears to have been due to concentrated nitric acid used to adjust the extract pH. Fluoride recovery was 92 %.

Appendix I, Table IV.5. Results from IC Analysis of the C-106 HLW Glass Sample TCLP Extracts

Analyte	Samples (mg/L)			TCLP Blanks (mg/L)	
	2a	3a	3b	a	b
Formate	< 0.1	< 0.1	< 0.01	< 0.1	< 0.01
Nitrate	5.09	4.15	4.69	3.97	4.82
Nitrite	< 0.1	< 0.1	< 0.01	< 0.1	< 0.01
Oxalate	< 0.1	< 0.1	< 0.01	< 0.1	< 0.01
Phosphate	< 0.1	< 0.1	< 0.01	< 0.1	< 0.01
Sulfate	< 0.05	< 0.05	< 0.005	< 0.05	< 0.005
Chloride	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
Fluoride	0.377	0.395	0.386	0.378	0.387

D. Ion-Selective Electrode

Results from the ISE analyses have been given in Table IV.6. The chloride concentration in the TCLP blank was as high as the chloride concentrations in the samples. A matrix spike was prepared for fluoride. Assuming the fluoride concentration in the unspiked extract was 10 mg/L (unspiked sample detection limit) the recovery would have been 129 %. Assuming the unspiked extract concentration was 0, the recovery would have been 153 %.

Appendix I, Table IV.6. Results from ISE Analysis of the C-106 HLW Glass Sample TCLP Extracts

Analyte	Samples (mg/L)			TCLP Blanks (mg/L)	
	2a	3a	3b	a	b
Chloride	55.1	68.9	< 10	71.1	< 10
Fluoride	< 10	< 10	< 10	< 10	< 10

5.0 Discussion

The purpose of this section is to provide a limited discussion as to the implication of the sample results. All target analytes were monitored to concentrations well below the maximum allowable lower detection limit required by the task specification.¹ In addition, all RCRA metal results were shown to be below the UTS limits.^{3,4} Discussions have been given as to the implications of results from the standard soil extracts, from the TCLP and digestion blanks, and from the matrix spiked extract.

A. Ion-Coupled Plasma-Atomic Emission Spectroscopy

ICP-AES results have been given in Table IV.1 and IV.2. According to these results, the TCLP was successful at showing

- standard soil extract results were within certified PA limits except chromium and antimony.
- recoveries from the matrix spike were above the 50 % indicated in the TCLP method.
- target analytes were monitored to concentrations required by the task specification.¹
- the glass waste form met the UTS limits^{3,4} for the RCRA metals determined by ICP-AES.

Results from analysis of the standard-soil TCLP extract have been given in Table IV.1. Elements for which the standard had been certified were within the PA limits except chromium (18-21 % below the PA limits) and antimony (less than 10 % below the PA limits). These deviations from the PA limits are expected to have been caused by the low temperatures at which the TCLP was performed (18 to 21 °C).

Also included in Table IV.1 were results from analysis of the matrix spike. All recoveries were well above the 50 % mentioned in the TCLP, and all recoveries for all analytes except aluminum were above the 75 % generally specified by ICP-AES analyses. Note that the TCLP method required the matrix spike concentration to be “not less than five times the method detection limit”. Although all spike concentrations were above the detection limits, the lead, antimony, and thallium spikes were less than five times the acid-digested sample method detection limits.

Results from analysis of the C-106 glass waste form TCLP extract have been given in Table IV.2. All target analytes were determined to be below the levels specified in the task specification.¹ In addition, all RCRA metal concentrations were shown to be well below the UTS limits.^{3,4} Although the standard extract chromium and antimony results were slightly below the PA limits, the sample extract concentrations for these analytes were shown to be well below the UTS limits.

Some results appear to have been more an aberration of the TCLP and acid-digestion methods than of the waste form. Sodium (TCLP blank concentrations similar to the sample extract concentrations) was added as sodium hydroxide during preparation of the extraction fluid. Silicon results should be considered suspect for the acid digested extracts since nitric acid is known to dissolve silicon from quartz in the plasma. Boron, barium, calcium, iron, magnesium and zinc were also seen in the TCLP blanks at concentrations similar to the sample extracts.

B. Atomic Absorption Spectroscopy

AAS results have been given in Table IV.3 and Table IV.4. According to these results, the TCLP was successful at showing

- mercury, arsenic, and selenium were monitored to the task specification concentrations.¹
- the glass waste form met the UTS limits for mercury, arsenic, and selenium.^{3,4}
- one standard soil mercury result was within the certified PA limit, and one result was not.
- the standard soil arsenic results were within the certified PA limit.
- the standard soil selenium results were below the certified PA limit.

Results given in Table IV.4 show mercury was determined to be well below the concentration specified in the task specification¹ (0.2 mg/L). In addition, the mercury concentration was shown

to be below the UTS limit.^{3,4} Mercury results given in Table IV.4 were close to the method detection limits. As shown in Table IV.3, the mercury concentration in one of the standard extracts was within the PA limits; however, the other was below the PA limits.

Results given in Table IV.4 also show arsenic and selenium were determined to well below the concentrations specified (0.0057 and 1 mg/L respectively) and were orders of magnitude less than the UTS limits. Also shown in Table IV.4, while the arsenic concentrations in the standard extracts were within the PA limits, the selenium concentrations were below the PA limits. This was most likely due to the temperature at which the TCLP extraction was performed.

C. Ion Chromatography

Fluoride was the only target anion. The sample extract fluoride concentrations given in Table IV.5 were orders of magnitude below the maximum allowable detection limit specified in the task specification.¹ The values reported in Table IV.5 were suspected to have been due to acetate from the acetic acid extraction fluid and not actually due to fluoride. During the analysis of the matrix spike, the fluoride and the acetate peaks were resolved. Using this result and assuming the sample fluoride concentration to be 0, the fluoride recovery was determined to be 92 %.

In addition, to fluoride, the results have been given in Table IV.5 for the other anions determined by IC. All anions other than nitrate were below the method detection limits. Nitrate was present in the extraction fluids because nitric acid was used to adjust the pH as specified in the TCLP procedure. Because these were not target analytes, no matrix spike recoveries were determined. Note that these chloride concentrations were orders of magnitude less than the ISE results.

D. Ion-Selective Electrode

Although fluoride was the only anion that indicated in the task specification,¹ both fluoride and chloride were determined by ISE. As shown in Table IV.6, the fluoride concentrations were determined to be below the method detection limits in all samples and blanks. The fluoride recovery was determined to be between 129 % and 153 %. Although chloride was detected in two samples, it was detected at a similar concentration in one of the blanks. The chloride did not, therefore, appear to have come from the waste form.

6.0 Conclusion/Summary

TCLP analysis was used successfully to characterize the Tank C-106 glass waste to provide the following information:

- Concentrations of all target analytes to below the maximum allowable lower limit of detection.
- Proof that all RCRA metal concentrations were below the UTS limits in the TCLP extract.
- Results for metal analytes were similar regardless of whether an acid-digestion was performed.
- Low extraction temperatures caused 4 analytes to be biased slightly low in the TCLP standard.
- Recoveries were greater than 50 % for all target analytes and greater than 75 % for most.

All target analytes were measured to below the maximum allowable lower limit of detection (MALLD) given in the task specification.¹ Except thallium and arsenic, the target analytes concentrations were determined to less than a tenth of the MALLD. Thallium could only be determined to approximately half the MALLD, and arsenic was determined to almost a tenth of the MALLD.

All RCRA metals were determined to be below the UTS limits.^{3,4} Except mercury, all RCRA metals were less than half the UTS limits. The mercury results are suspected of being higher than the actual concentration in the extract. Mercury was expected to have been lost from the waste form during vitrification. In addition, the reported concentration of mercury in one of the blanks was about a third of the sample result, and these values were close to the method detection limit.

ICP-AES result showed that for the most part metal results were similar for the extracts regardless of whether samples were subjected to an acid-digestion prior to analysis. Although required by the SW-846 method² prior to metal analyses, common laboratory practice is to adjust the sample pH and analyze the sample without performing an acid-digestion. The results from this study support this practice.

Most of the analytes were present in the standard extract at concentrations that met the PA limits. Antimony, chromium, and selenium were below the PA limits in all the standard extracts. In addition, mercury was below the PA limits in one of the standard extracts. Antimony, chromium, and selenium concentrations were so low in the sample extracts that such deviations (most likely due to the temperature of the extraction) would not have caused the sample extracts to exceed the UTS limits.^{3,4}

For all target analytes, matrix spike recoveries were greater than the 50 % specified in the TCLP method. Except for silver and mercury, the recoveries were greater than 75 %. Recoveries for arsenic, fluoride, and probably thallium were greater than 125 %. Although these recoveries are not generally considered acceptable, the effect on the TCLP results are not considered to make a significant difference on these results.

7.0 TCLP References

1. "Vitrification of HLW Envelope D Sample C-106 and Product Testing," Gary Smith and Stuart Arm, **RPP-WTO Development Task Specification Number TS-W375HV-PR00005, Rev. 2**, January 10, 2000.
2. "Test Methods for Evaluating Solid Waste-Physical/Chemical Methods," U. S. Environmental Protection Agency, **EPA SW-846**.
3. "Dangerous Waste Regulations," Washington Department of Ecology, **Chapter 173-303 Washington Administrative Code**, November 1995.
4. "Land Disposal Restrictions," U. S. Environmental Protection Agency, **40CFR268**.

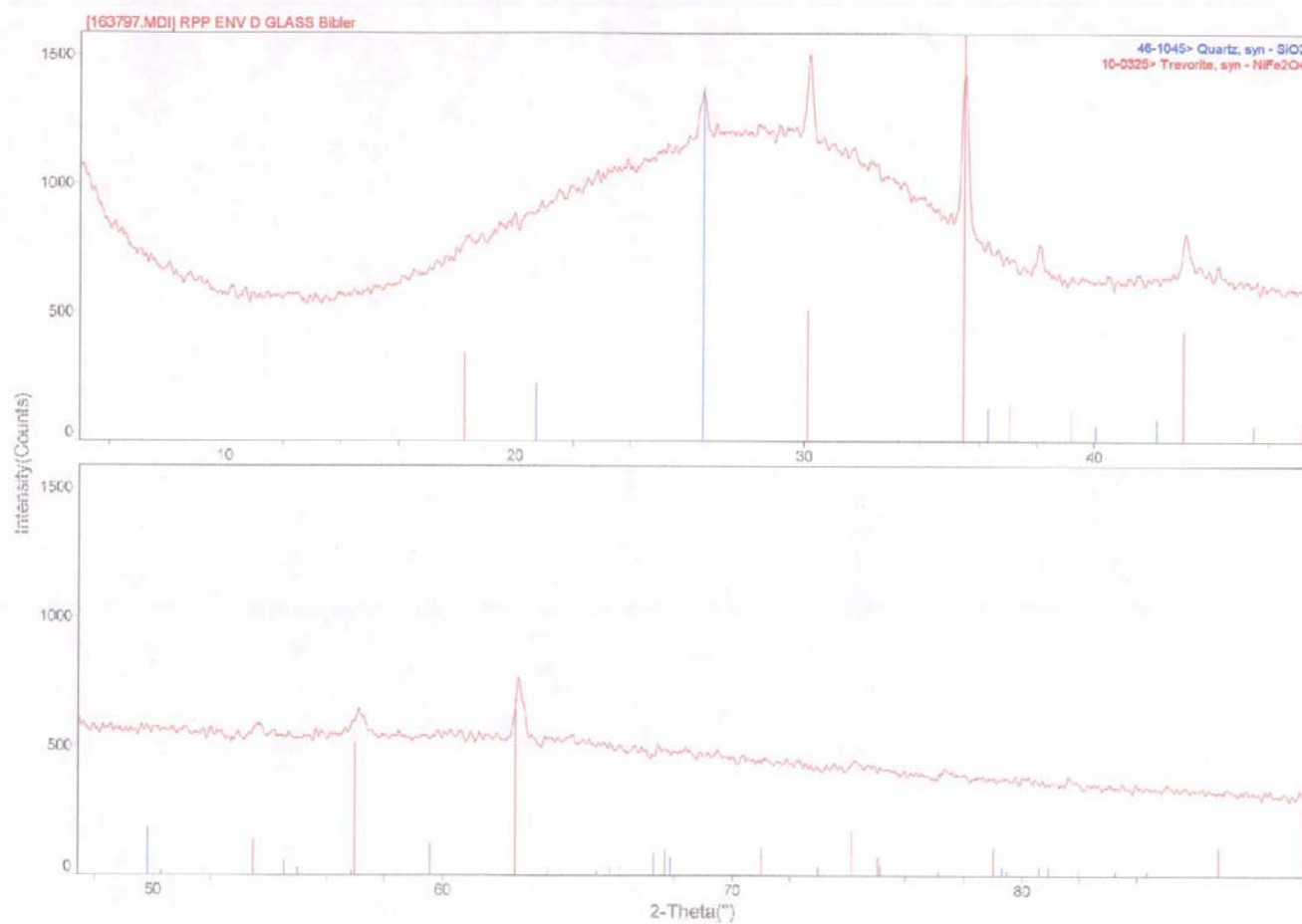
Appendix J. Microscopy and Crystallinity Phase Determination

Glass Analyses by X-ray Diffraction Analyses (XRD) and Scanning Electron Microscopy (SEM)/ Energy Dispersive X-ray Analyses (SEM/EDAX)

The Envelope D radioactive glass was ground to a powder for the PCT durability testing using a Techmar tungsten blade grinder with stainless steel grinding compartment. Glass produced in this process was used for XRD and SEM/EDAX analyses. The ground glass was sieved through brass sieves to render three different fractions of glass powders. The glass fines sieved through the 200-mesh sieve are the -200-mesh powders that were not used in the PCT. The glass powder sieved through the 100-mesh sieve that remained on the 200-mesh sieve is referred to as the -100 to + 200-mesh powder that was used in the PCT. The ground glass remaining on the 100-mesh sieve is the + 100-mesh glass. The -200-mesh fine powdered glass was used for XRD analyses. The ground glass remaining on the 100-mesh sieve, i.e., the + 100-mesh glass, was used for the SEM and EDAX analyses.

The X-ray diffraction pattern of the Envelope D glass is presented in Figure 1. Two different crystalline structures were initially identified from the XRD spectra as quartz (blue peak at 2Theta (°) of 27°) and the nickel-iron spinel trevorite (red peaks at 2Theta (°) of 30°, 35°, 43°, 57° and 63°). The quartz crystal, SiO_2 , could derive from trace quartz contaminants flaked off from the quartz containment vessel used in making the glass in the Deltech furnace. The trevorite crystal, NiFe_2O_4 , is in the spinel family of crystals with generalized formula $((\text{Fe},\text{Ni},\text{Mn})(\text{Fe},\text{Cr})_2\text{O}_4)$. Spinel crystals have been identified in previous vitrification studies involving Hanford RPP High-Level Waste streams (See References # 5d,e and #22 in main text). Further analysis of the original XRD spectrum indicated possible presence of an additional face centered cubic (FCC) crystalline structure as indicated by the XRD spectra peaks located at ~ 38° and ~ 45°. Figure 2 shows this structure tentatively assigned to metallic silver. Other possible constituents for these peaks include reduced copper and possibly trace tungsten metal from the grinder blade.

Appendix J, Figure 1. XRD Spectra of Envelope D Glass Powder



Appendix J, Figure 2. XRD Spectra of Envelope D Glass Powder

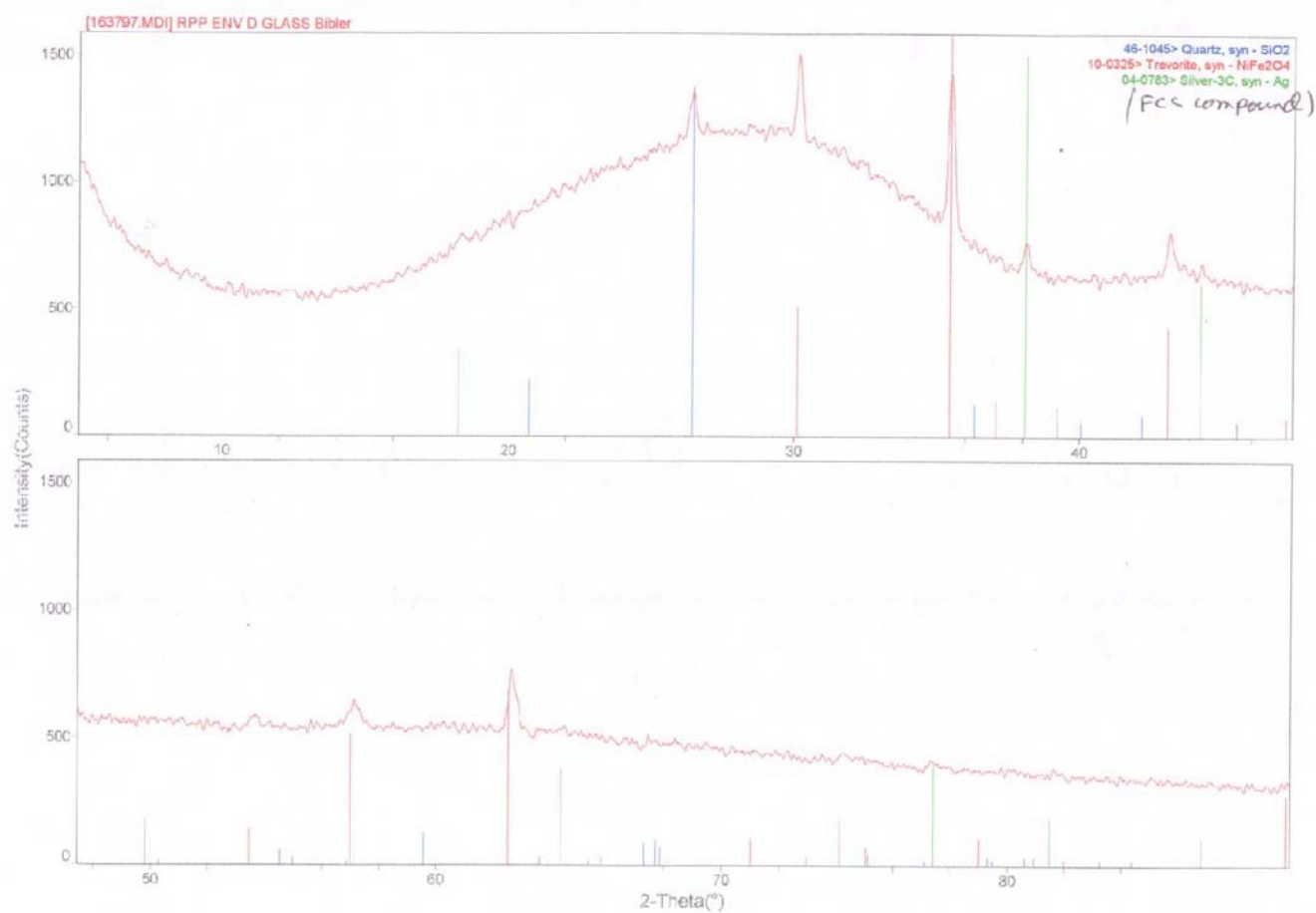


Table 1 contains summary information on the SEM microscopic images of the +100-mesh powdered glass samples derived from grinding the Envelope D glass. Images obtained from secondary electron and backscattered electron microscopy were obtained at magnifications of 10X, 50X, 400X and 1000X. Generally the SEM technique uses backscattered electrons (BSE), or incident electrons, to indicate potential density differences in the image particles. Use of secondary electron (SE) imaging that involves actual electrons from the matrix material provides topography images of the matrix. Figures 3-5 show the SEM images. Figure 3a and 3b show backscattered electron images of glass magnified to 10X and 50X, respectively. Two individual pieces of glass are identified in Figure 3b as glass particle #1 and glass particle #2. Figures 4a-c show further images of the glass particle #1. Figure 4a and 4b show 400X magnified images using backscattered electrons (4a) and secondary electrons (4b). Figure 4c shows further magnification of glass particle #1 at 1000X. Several individual spots on Figures 4a and 4c are identified. These spots are discussed below with respect to the EDAX spectra. Figures 5a and 5b are 400X magnified images of the glass particle #2. The individual spots shown in Figure 5a are also discussed below.

Appendix J, Table 1. Summary Information on Microscopy Data

Figure	SEM Image	Technique	Magnification	EDAX
3a	068	BSE	10-X	
3b	069	BSE	50-X	
4a	070	BSE	400-X	6 a-b,7 a-b
4b	071	SE	400-X	
4c	072	BSE	1000-X	8a
5a	075	BSE	400-X	8b
5b	076	SE	400-X	

Energy dispersive X-ray analyses were obtained for the bulk glass matrix and the light-shaded matrix particles shown in Figure 4a (SEM Image #070). These EDAX spectra are shown in Figures 6 and 7. The EDAX pattern shown in Figure 6a shows the bulk glass matrix material (indicated as 'A' in Figure 4a) to be comprised of the elemental components of the glass matrix, Sr, Si, Mn and Fe. The EDAX pattern shown in Figure 6b shows the light-shaded matrix particles (indicated as 'B' in Figure 4a) to be comprised of Sr, Mn, Fe and Ni. The EDAX

pattern shown in Figure 7a shows the light-shaded matrix particles (indicated as 'C' in Figure 4a) to be comprised of Sr, Cr, Mn, Fe and Ni. The EDAX pattern shown in Figure 7b shows the light-shaded matrix particles (indicated as 'D' in Figure 4a) to be comprised of Sr, Cr, Fe and Ni. All of these 'B', 'C' and 'D' spots are likely associated with the crystalline phase identified by XRD as trevorite, or NiFe_2O_4 . The Cr and Ni elements associated with these spots could also be indicative of trace steel particles in the glass fines that come from the steel compartment inside of the Techmar grinder used to grind the glass.

Additional EDAX patterns are shown in Figures 8a and 8b for the spots labeled as 'E' in Figure 4c (image # 072) and as 'F' in Figure 5a (image # 075). Note that these spots are relatively much smaller in size to the other identified spots in the Figures 4c (spots 'B' and 'C') and 5a (spots 'C'). The tungsten (W) identified in Figure 8a likely derives from the tungsten blade used to grind the glass. The Zn and Cu identified in Figure 8b likely derives from the brass sieves used to sieve the glass powders.

It is possible to estimate the amount of crystallinity associated with the glass by using the SEM image from Figure 4a, labeled as SEM image # 070. There appears to be about 10 different crystalline spots on the surface of this glass piece. Measurements using a ruler on the original SEM image show the spots to be in the 3 to 4 mm cross-sectional size range. The entire glass piece measures to be about 100 mm x 80 mm. Using these data with the equation below, an estimate of about 1.5 % surface crystallinity is obtained for this glass piece. If this single glass piece is representative of the entire glass and the crystalline material is distributed throughout the glass at the same level as is shown on the glass piece surface in SEM image #070, then this estimate should be accurate to within about +/- 10 to 20%.

% crystallinity on surface of glass in SEM image # 070 = (crystal area / glass area) * 100

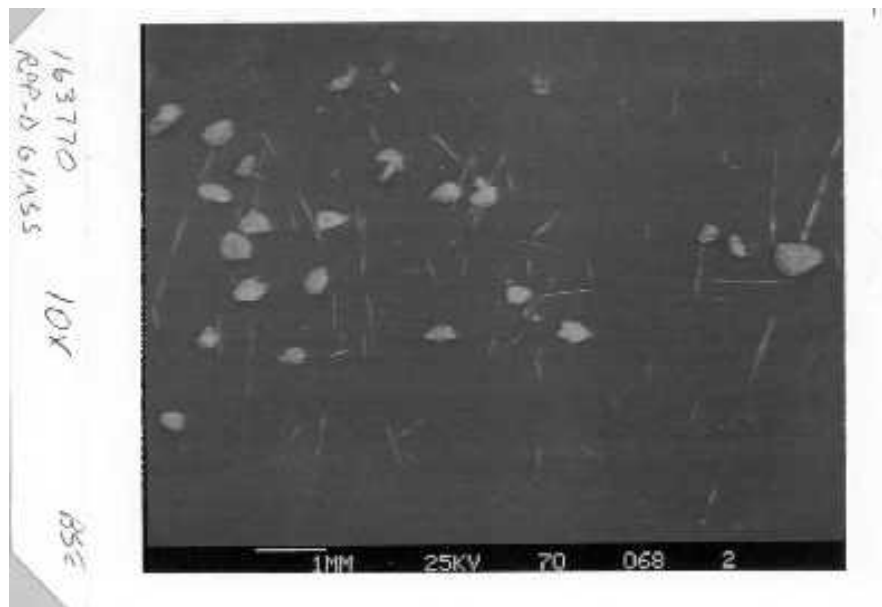
where,

crystal area = $\sim (3.5 \text{ mm})^2/\text{spot} \times 10 \text{ spots} = 122.5 \text{ mm}^2$

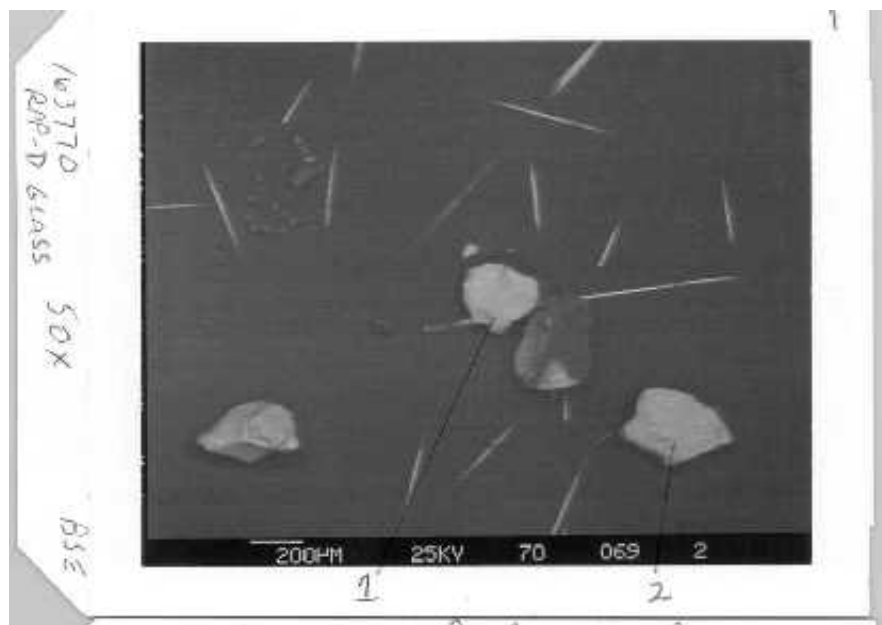
glass area = $\sim 100\text{mm} \times 80 \text{ mm} = 8,000 \text{ mm}^2$

% crystallinity = $(122.5 \text{ mm}^2 / 8,000 \text{ mm}^2) * 100 \sim 1.5 \%$

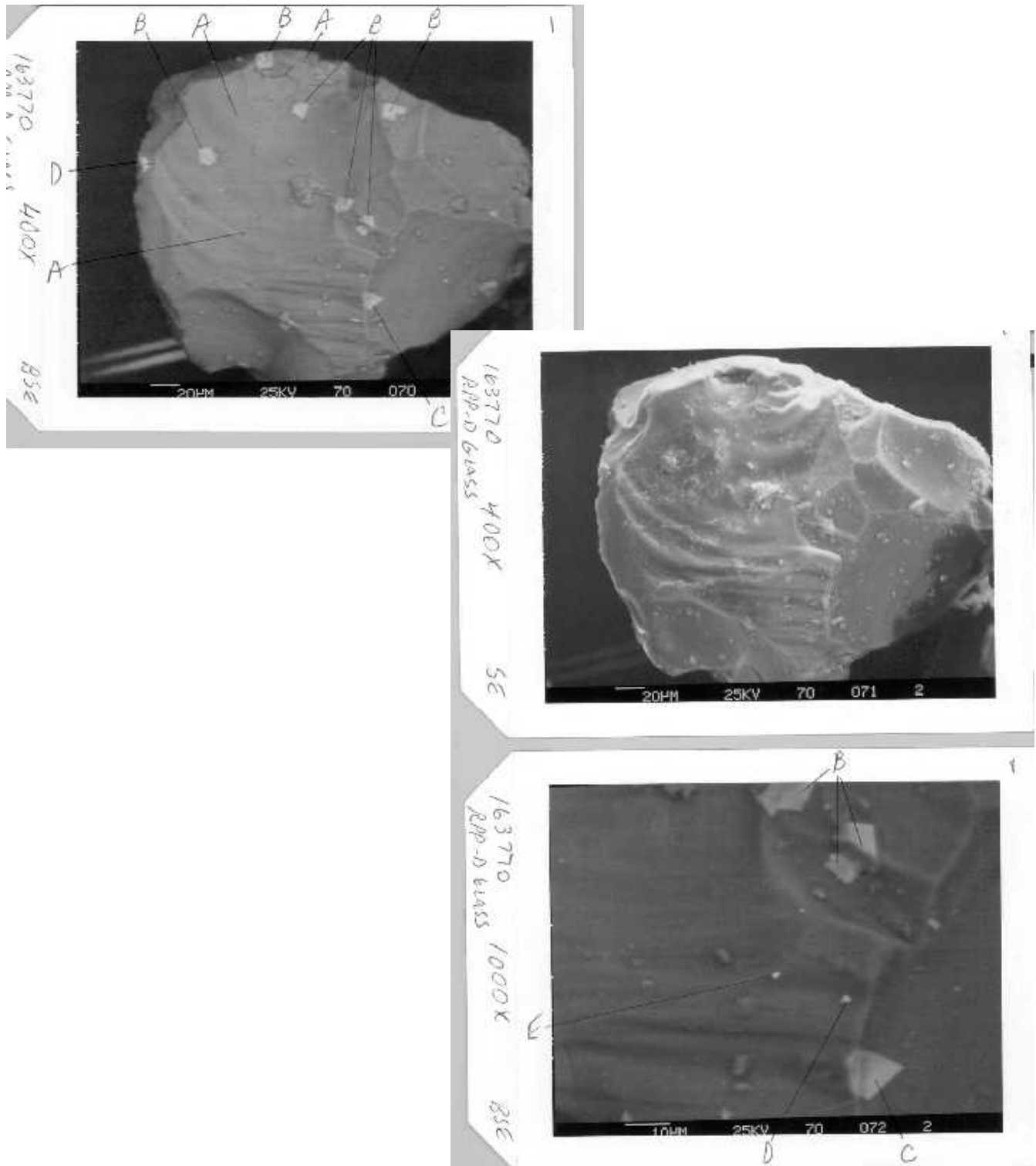
Appendix J, Figure 3. (a) SEM Image of D Glass at 10X Magnification



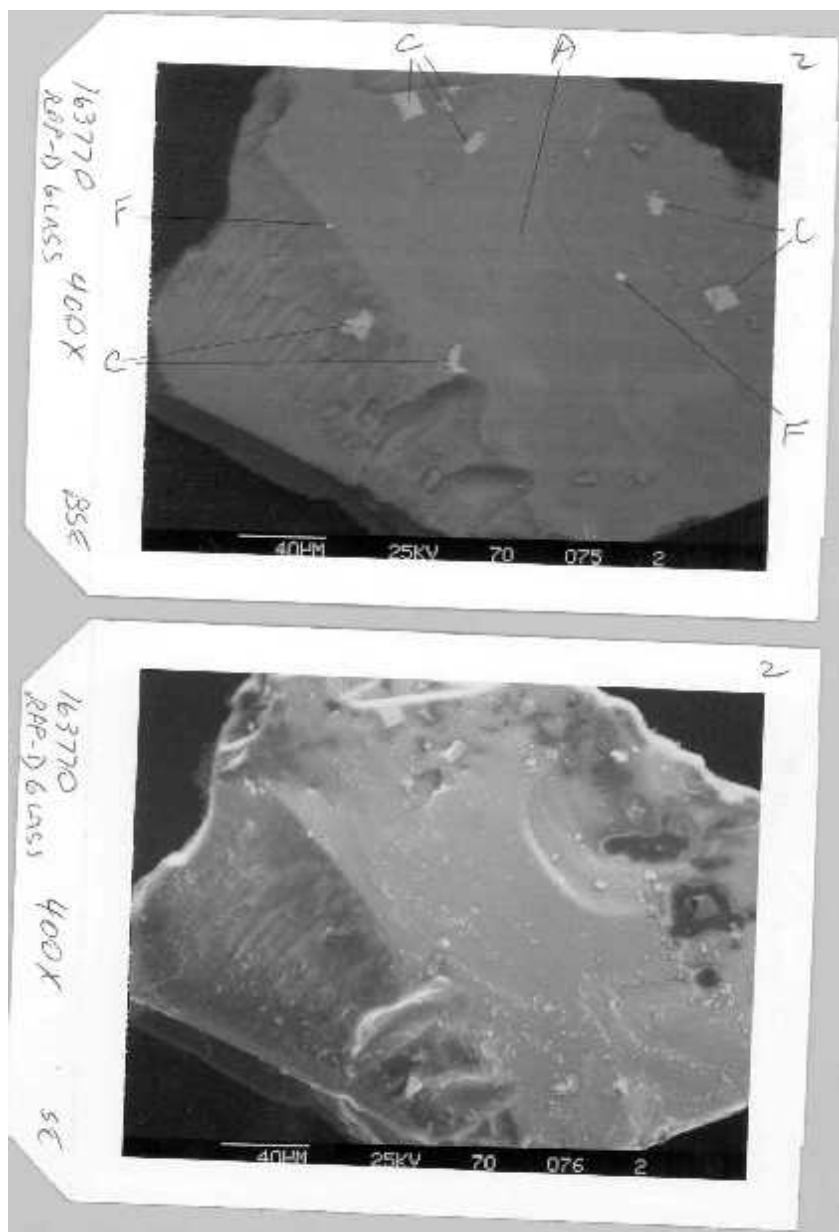
Appendix J, Figure 3(b) SEM Image of D Glass at 50X Magnification



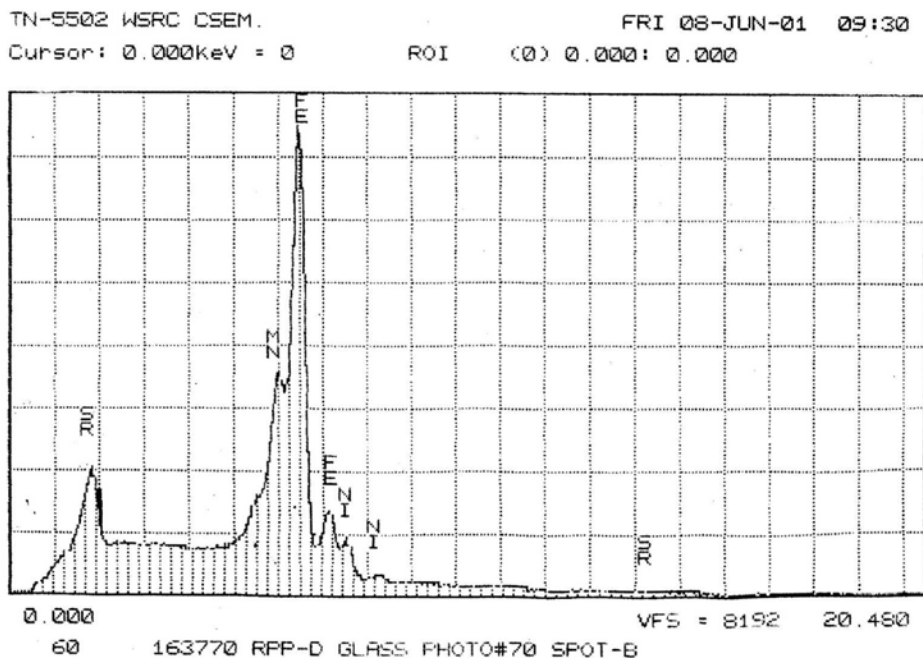
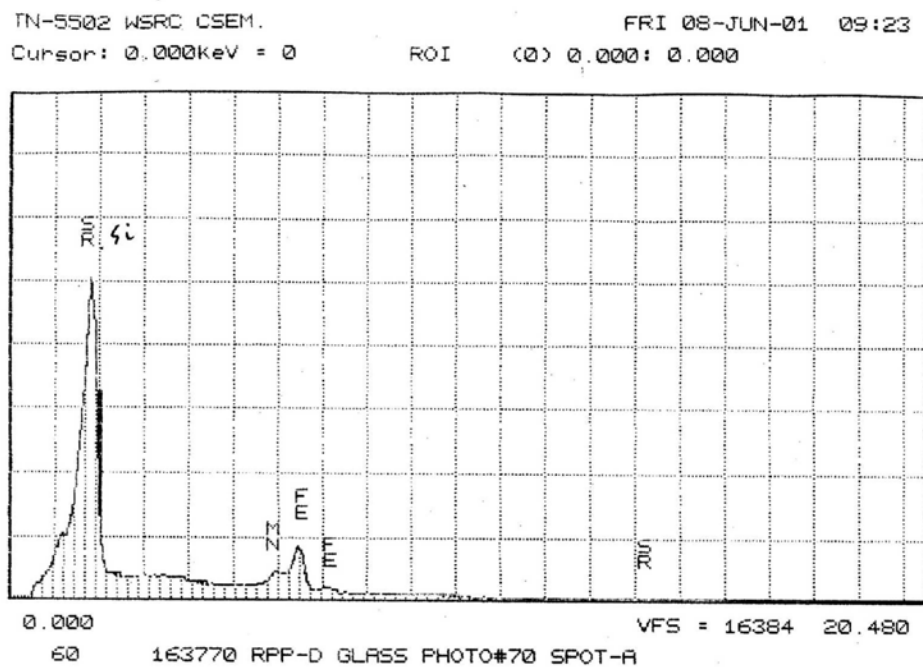
Appendix J, Figure 4. (a)-Top, (b)- Middle and (c)-Bottom: Additional SEM Images of D Glass



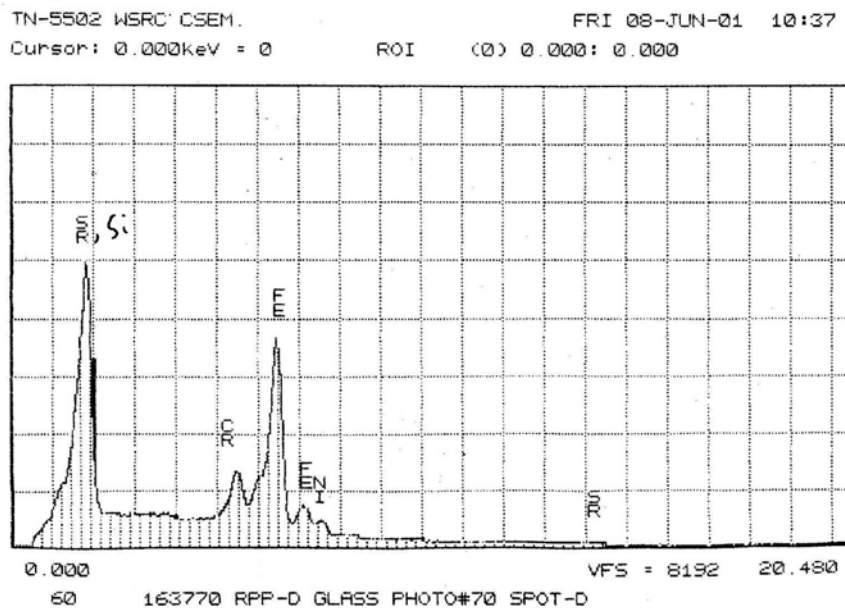
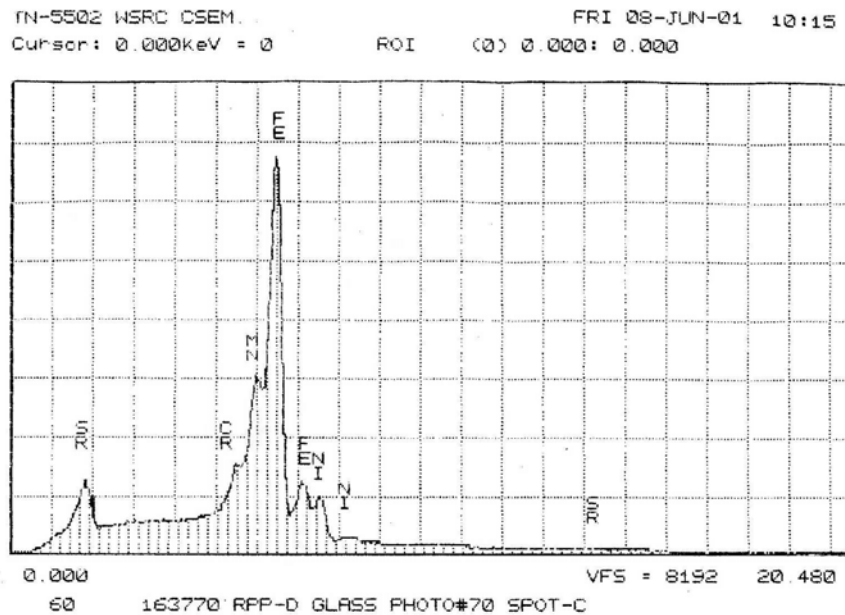
Appendix J, Figure 5. SEM Images of the Particle #2 D Glass, (a)-Top and (b) - Bottom



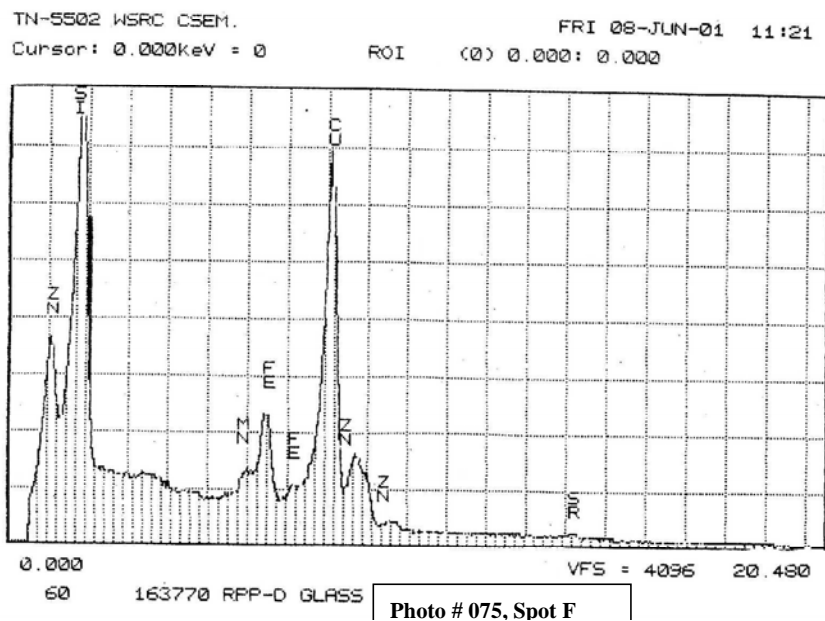
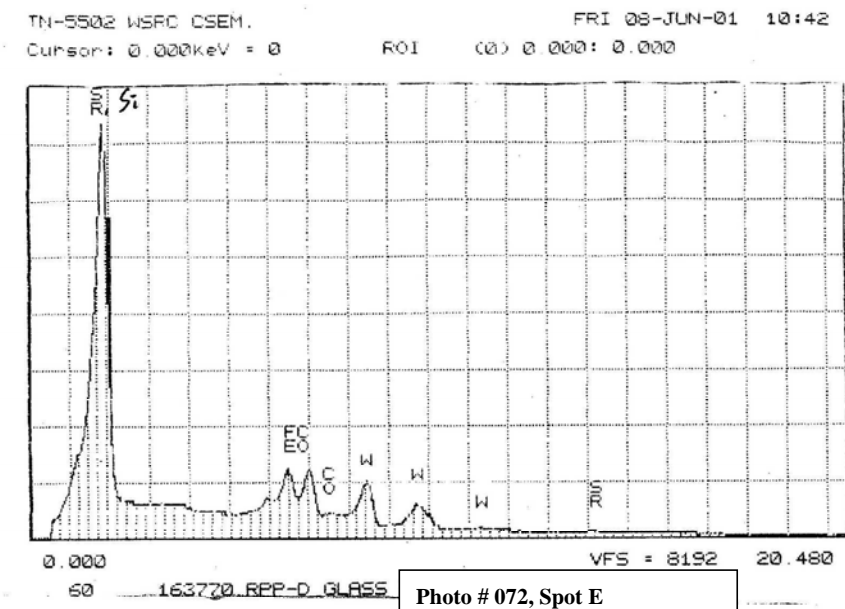
Appendix J, Figure 6 (a) EDAX Spectra Spot 'A', (b) EDAX Spectra Spot 'B'



Appendix J, Figure 7 (a) EDAX Spectra Spot 'C', (b) EDAX Spectra Spot 'D'



Appendix J, Figure 8 (a) EDAX Spectra Spot 'E', (b) EDAX Spectra Spot 'F'



X. References

- ¹ “Vitrification of HLW Envelope D Sample C-106 and Product Testing,” G. Smith and S. Arm, **RPP-WTO Development Task Specification Number TS-W375HV-PR00005, Rev. 2**, January 10, 2000.
- ² Note: This work was initiated under the Tank Waste Remediation System Privatization Contract with DOE:
TWRS Privatization, **Contract No. DE-AC27-96RL13308**, Mod. No. M014, Section C: Statement of Work, November 2000. (See: <http://www.hanford.gov/doe/contracts/de-ac06-96rl13308/conformed/index.html>)
The TWRS Contract was replaced by the Waste Treatment Plant DOE Contract in early 2001 with DOE: WTP Contract, **Contract No. DE-AC27-01RV14136**, Mod. No. M008, Section C: Statement of Work, September 2001. (See: <http://www.hanford.gov/orp/contracts/de-ac27-01rv14136/toc.html>)
- ³ Technical and Development Support to TWRS Design, K.A. Johnson and M.E. Johnson, **K0104_077_PRC**, December 1997.
- ⁴ Westinghouse Savannah River Company, Work for Others Agreement, **WFO-98-003**, 1998.
- ⁵ (a) Westinghouse Savannah River Company, Work for Others Agreement, **WFO-96-004**, 1996; (b) “Production of a High-Level Waste Glass from Hanford Waste Samples”, C.L. Crawford, D.M. Ferrara, B.C. Ha, and N.E. Bibler, **Proceedings of Spectrum ’98**, Denver, Colorado, September 13-18 1998, Vol. 1 pp. 581-588; (c) “Vitrification of Three Low-Activity Radioactive Waste Streams from Hanford”, D.M. Ferrara, C.L. Crawford, B.C. Ha, and N.E. Bibler, **Proceedings of Spectrum’98**, ibid. pp 706-713; (d) “Vitrification of Four Radioactive Hanford Waste Samples”, D.M. Ferrara, C.L. Crawford, B. C. Ha, N. E. Bibler and A. S. Choi, **SRTC-BNFL-023, Rev. 2, Jan. 5, 1998**; (e) “Hanford High Level Waste Glass – Additional Results”, N. E. Bibler, D. M. Ferrara and C. L. Crawford, **SRTC-BNFL-026, Rev. 0, Jan. 19, 1998**.
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