Crucible-Scale Active Vitrification Testing of a Hanford Envelope C Tank 241-AN-102 Sample

by

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DISCLAIMER

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Crucible-Scale Active Vitrification Testing Envelope C, Tank 241-AN-102 (U)

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SUMMARY

As part of the Hanford River Protection Project-Waste Treatment Plant (RPP-WTP), the Savannah River Technology Center (SRTC) has produced and characterized an Immobilized Low Activity Waste (ILAW) glass from a pretreated, decontaminated Hanford Tank 241-AN-102 sample. The glass was made from a radioactive supernate that was pretreated by SRTC personnel using Sr-TRU precipitation to remove strontium and transuranic radionuclides and ion exchange to remove Cs-137 and Tc-99.

The AN-102 ILAW glass was produced from the decontaminated supernate feed referred to as 'Large C' sample. The ~ 16 L decontaminated Large C supernate (4.8 +/-0.1) Molar sodium was analyzed and the characterization results were used to provide a glass formulation recipe targeted at nominally 11.8 wt% Na_2O in glass. An 86-mL sub-sample of the characterized large C supernate was used to prepare an immobilized low-activity waste glass (ILAW) sample. The glass was vitrified from mixing this sub-sample and a blend of ten glass-forming minerals in a platinum/gold crucible and heating to 1150 °C. The glass melts were then cooled following a prescribed cooling curve. All glasses were dissolved using versions of ASTM standard glass dissolution methods involving either sodium peroxide (Na_2O_2) fusion with acid uptake, or heated digestion with a mixture of hydrofluoric (HF) and nitric (HNO_3) acids with boric acid uptake. Glasses were also dissolved by a CsOH fusion method for comparison. Measured composition of the resulting AN-102 glass waste form was close to the target composition. Compositions of standard Low Activity Reference Material LRM glass were also close to target compositions. Measured radionuclide levels in the glass indicate that the immobilized low activity wasteform is within the limits of the RP-WTP DOE/ORP contract specifications and the glass does not contain transuranic (TRU) radionuclides above the TRU wasteform limits.

No crystalline material was observed from X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses of the crushed glass. The ASTM standard Product Consistency Test (PCT) performed at 90°C on the AN-102 radioactive glass and the Low Activity Reference standard LRM glass showed similar measured releases for the B, Si, Na components. The PCT results indicate that normalized released for B, Si, and Na are well below the specification limit of 2 g glass/m^2.

INTRODUCTION

The Department of Energy, Office of River Protection is utilizing subcontractors to design, construct, and operate facilities to immobilize radioactive waste stored in underground tanks at the Hanford site near Richland, Washington [1,2]. The program is called the River Protection Project-Waste Treatment Plant (RPP-WTP). The current phase of this project is referred to as Part B-1 and includes activities for verification of technology and design of waste treatment facilities through August 24, 2000. Present studies are a continuation of previous collaborative efforts of BNFL, Inc. and the Savannah River Technology Center (SRTC) [3]. As part of the present Part B-1 SRTC demonstration, the Immobilization Technology Section (ITS) of SRTC has demonstrated, using a crucible-scale furnace, the vitrification portion of the process to producing an ILAW glass waste form from Hanford Tank 241-AN-102. This small active vitrification task evaporates and vitrifies samples from radioactive waste treatment demonstrations being performed by SRTC as part of a Work for Others (WFO) agreement [1,4].

The objectives of this work were to characterize and performance test the vitrified product and to provide RPP-WTP personnel with the results from these studies.[(1)] This is the final report for the small scale active vitrification testing on the AN-102 sample. RPP-WTP personnel have outlined the information that they are expecting from this study in a previous Task Specification document [4]. The scope of this report includes results from chemical and radionuclide analyses of 1) the Large C decontaminated supernate, 2) the dissolved glass, 3) crystalline-phase determinations in the glass by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and 4) durability Product Consistency Tests on the glass. Other tests performed on the glass include the Toxicity Characteristic Leaching Procedure (TCLP), density, and various organics, halides, anions and sulfur analyses. These latter TCLP, density and organics, halides, anions and sulfur analyses objectives and test results are described in a separate related 'Regulatory Analysis Report' prepared by ITS personnel at SRTC [5].

[(1)]Earlier work in this Part B1 program investigated sulfate removal from a portion of the AN-102 decontaminated supernate. The resulting sulfate-pretreated AN-102 supernate product was concentrated by evaporation and analyzed. Results of the analytical characterization were transmitted to VSL for glass formulation. However, it was decided by RPP-WTP personnel that sulfate removal by pretreatment was not to be pursued. Thus vitrification of the resulting glass former recipe developed by Vitreous State Laboratory for the sulfate-treated Env. C decontaminated supernate was not completed. A complete summary report on the sulfate pretreatment testing program, as well as the evaporation, characterization and glass formulation of the sulfate-treated Env. C, AN-102 sample is in preparation ("Sulfate Removal Studies for RPP Part B1", M. Hay et al., SRT-RPP-2000-00049, January, 2001).

EXPERIMENTAL

Feed Stream Characterization and Waste Glass Formulation

The goal of the Feed Stream Characterization and Waste Glass Formulation phase was to analyze the decontaminated Envelope C supernate and mix the waste form and glass-forming chemicals in a 600-mL platinum/gold crucible. The AN-102 LAW liquid was decontaminated of strontium-90 and transuranics by Sr-TRU precipitation pretreatment [6] and decontaminated of Cs and Tc by ion exchange pretreatment [7]. The pretreated AN-102 LAW supernate feed was mixed with glass-forming chemicals to complete the feed stream preparation phase. Except for preparation of supplies and glass-forming chemicals, the feed preparation was performed in radiochemical hoods within a radiological buffer area (RBA).

Decontaminated Liquid Feed Characterization

The Large C decontaminated supernate was transferred from SRTC shielded cells facility to a Radiological Buffer Area (RBA) laboratory after completion of pretreatment tasks. The supernate was transferred in 20 separate 1-Liter polybottles. Approximately 16 Liters of sample were collected. All of the supernate was composited in two similar 10-Liter polybottles within a radiological hood in the RBA. Characterization of the two replicate 10-Liter containers was pursued by SRTC personnel. Table 1 shown below summarizes the analyses performed on the Large C decontaminated supernate as well as other analyses performed in this vitrification task.

Table 1. Required Analytical Support for AN-102*

Technique	Composited, Preteated AN-102**	Sample Preparation (Glass Formers)	PCT Leach Tests	Glass Analyses
Na_2O_2 Fusion		X		X
CsOH Fusion		X		X
HNO_3 Dissolve		X		X
ICP-ES	X	X	X	X
AA(Na/K)	X (K only)			X
ICP-MS	X			X
IC	X			
Chemchek (Total Uranium by Fluorescence Spectroscopy)	X			X
Gamma-PHA	X			X
Total Alpha-PHA	X			X
Total Beta-Scint.	X			X
Liquid Scint.				X
Sr-90	X			X
Tc-99				X
TIC/TOC	X			
Density,Wt% Sol.	X			
XRD				X
SEM/EDAX				X
Ion Sel. Electrode	X			
Free OH	X			

* These analyses are to be performed at SRTC. Separate regulatory analyses are described in detail in Reference 5.

** Only Large C, AN-102 composited, pretreated supernate was characterized for this study. See text.

Waste Glass Formulation

This step prepares the appropriate amount of melter feed for the crucible vitrifications. The appropriate amounts of decontaminated AN-102 liquid waste and a nonradioactive glass-forming chemical stream were mixed directly in the crucibles. All glass-forming chemicals were comprised of various mineral compositions specified by Vitreous State Laboratory (VSL). Previous studies in Part A work used reagent grade chemicals as the glass-forming chemicals [3]. Ongoing developmental work performed with waste simulants at VSL at Catholic University and at SRTC [8] supports this task. The results from the VSL development work form the basis for the formulations used in this step. The types and forms of industrial grade glass formers used in this study are the same as those used in the VSL development to facilitate comparison of the glasses made from simulated and actual waste.

Decontaminated AN-102 supernate was analyzed by sampling each of the two similar 10-Liter carboys containing the Large C sample in duplicate according to the analyses shown in Table 1. Average results from these four replicate analyses were transmitted to VSL, who then communicated to SRTC the appropriate amounts of the waste streams, composition of the glass-forming chemicals, and appropriate amounts of the glass-forming chemicals. RPP-WTP personnel reviewed the recommendations and approved them before SRTC blended the glass formers.

As an additional quality assurance step, SRTC analyzed a representative portion of the final glass formers according to Table 1, and confirmed that analyzed components for Al, B, Cr, Fe, Li, Mg, Si, Ti, Zn, Zr agreed with as-batched values before actual mixing of the glass forming chemicals and decontaminated feed stream. The final mixtures of glass-forming chemicals and pretreated AN-102 feed were prepared in 600-mL platinum/gold crucibles for the batch tests. Balances used to measure the glass-forming chemicals, to determine the mass of each stream, etc. were standard, single-pan, top-loading analytical balances. All balances are calibrated by the SRTC standards lab using NIST-traceable standards. Calibration checks were performed with standard masses that bracket the mass of the material to be weighed prior to all weighings.

Vitrification

The goal of the Vitrification phase was to immobilize the AN-102 waste stream in glass matrix that could then be characterized and performance tested. The target melt temperature for all active crucible scale vitrifications was 1150°C. The AN-102 waste stream is a Resource Conservation and Recovery Act (RCRA) listed waste. Therefore, an offgas system was used for the vitrification testing to maintain certain listed waste component effluent levels below the allowable limits within SRTC lab facilities [4]. Evaporation and calcining occurred in the furnace at temperatures from 100°C to 900°C. Heat-up rates during these steps were nominally < 100°C/hour. The furnace temperature was then increased from 900°C to 1150°C at a nominal heat-up rate of 100°C/hour. The vitrification temperature of 1150°C was held for 4 hours. After vitrification, the glass

waste form was cooled according to a prescribed cooling schedule provided by VSL/RPP-WTP.

Waste Form Evaporation and Calcining

After mixing the glass-forming chemicals with the pretreated waste in the vitrification crucible and transferring the resulting slurry to the vitrification furnace, water from the slurry was slowly removed by evaporation. This process is described in more detail below. A detailed diagram of the furnace as configured for these studies is presented in Figure 1. The furnace used for these studies is a DelTech Model DT-29-TL-610 Top Loading Laboratory Furnace capable of 1200°C with a programmable setpoint temperature control. The furnace was initially 'baked out' and calibration-tested before use according to recommended procedures by the vendor. Thermocouples and digital readouts used for calibration of the furnace were calibrated by the SRTC standards lab with NIST-traceable standards.

An offgas collection glassware apparatus was attached to the quartz glass system within the furnace. This offgas system contained and trapped all gaseous hazardous species evolved during the slurry evaporation, calcine and vitrification steps. The offgas system contained a primary water-cooled condenser, a dry ice bath and two activated carbon beds in series. A collection pot was connected to the water condenser for final collection of all condensed phase liquid collected during the evaporation and calcine steps. As shown in Figure 1, ambient air flowed into a quartz tube through an inlet carbon filter. The quartz vessel inside of the furnace contained an alumina insert that held the vitrification crucible. Incoming air will swept through the quartz tube carrying offgas from inside the sealed quartz vessel system to the offgas system (condenser, cold trap and carbon filters). The central offgas tube exits the furnace through a 1" diameter opening cut out of the top of the furnace. All loading of equipment and samples into the furnace was performed through a top-located circular furnace door of 6" diameter (not shown in Figure 1).

The final carbon filter in the offgas system was connected to vacuum. A vacuum of nominally 2-3 inches of water was maintained on the crucible throughout the entire vitrification process. The vacuum was monitored periodically by connecting a water manometer to the air inlet. Vacuum was supplied by SRTC facility-supplied vacuum through a connection within the radiochemical hood.

Temperatures of the furnace were slowly increased to remove the water from the slurry contained in the crucible and collect it in the trap below the condenser. The nominal heatup rate was 10°C/hour. After the water was evolved from the waste form, the volatile species were driven off by slow temperature increases typically between 200 °C and 900 °C. Initial planning required use of a 250-mL crucible. However, due to increased amounts of glass product needed for all planned analyses, a 600-mL platinum/gold crucible was used.

(Offgas exits to: (1) water condenser, (2) dry ice bath, (3) carbon filters in series)

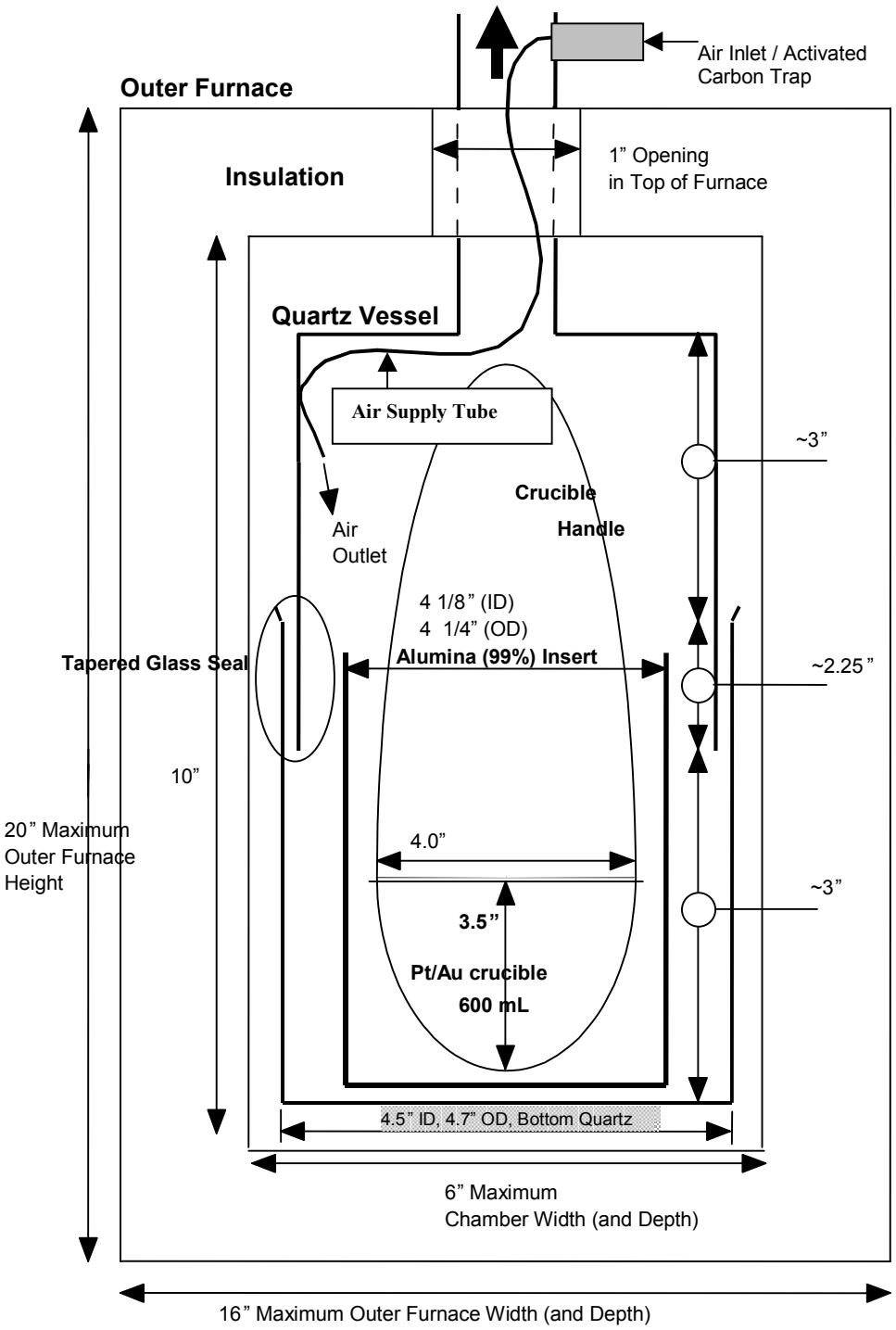


Figure 1. Small Active Crucible-Scale Furnace

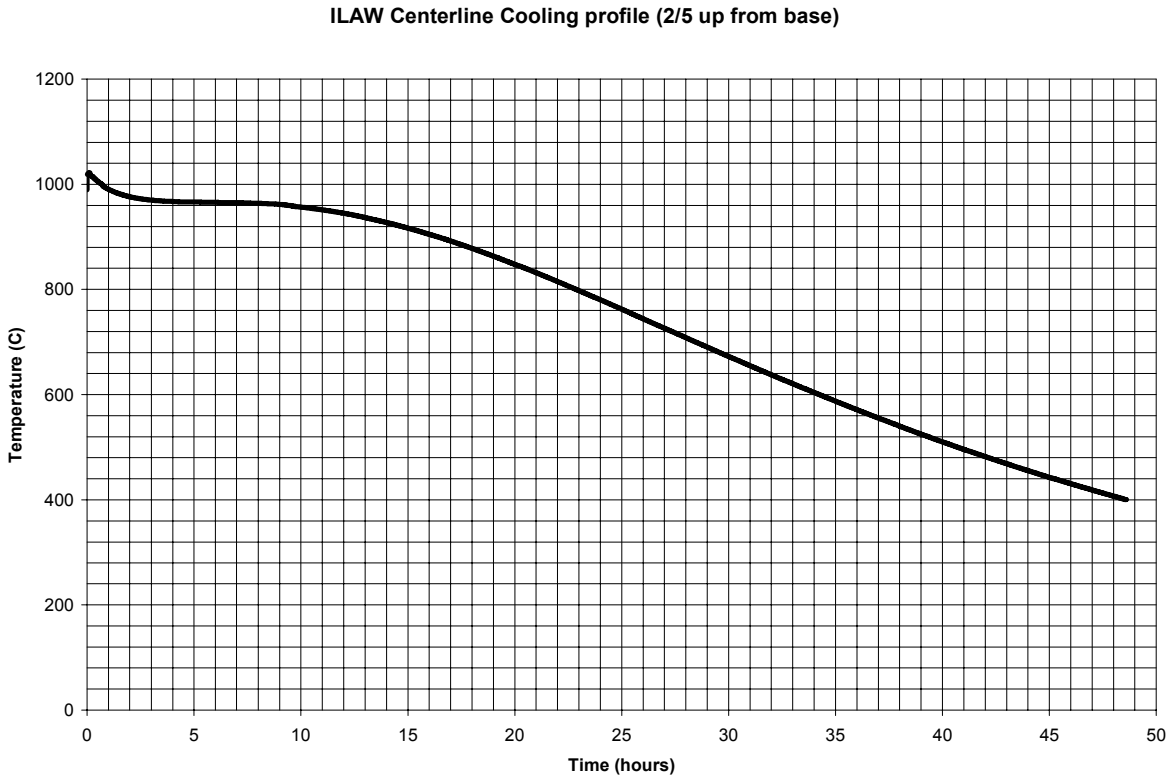
Waste Form Vitrification

The waste form was vitrified in the same furnace used to calcine the waste form. The temperature was slowly increased from 900°C, to the melt temperature of 1150°C, where it was held at this temperature for ~ 4 hours.

Glass Waste Form Cooling and Removal

After the melting period, the molten glass was cooled inside of the furnace according to the cooling schedule provided by VSL/RPP-WTP [LAW Cooling Curve Source: E-mail sent to T. B. Calloway (SRTC) on 11/15/99 from S. Arm (formerly with BNFL,Inc., currently with CHG), CHG Reference # is 009325]. The glass cooling profile was intended to simulate the temperature cooling profile of glass in containers planned for the RPP-WTP. The temperature range for the controlled cooling was 1100 °C to 400 °C. This cooling profile is shown in Figure 2. After the furnace program reached the lower temperature of the cooling schedule, heating of the furnace was discontinued and the system was allowed to cool to ambient temperature. The glass and offgas system were handled after the furnace vitrification system was at ambient temperature. Liquids from the condensate traps will be collected and their amounts measured by volume. No analyses were planned for any of the small active vitrification condensates resulting from crucible-scale furnace vitrification activities.

Figure 2. Cooling Curve for AN-102 Vitrification



Glass Dissolution and Analyses

Product glass for the active AN-102 sample was initially size-reduced by manually grinding the glass pieces using an agate mortar and pestle. The glass was then further pulverized to a (-) 200 mesh size using a Mixer Mill with agate cups and agate grinding ball. Samples of the standard Low Activity Reference Material LRM glass [9,10] were also ground in the Mixer Mill. Resulting glass powders were verified to be (-) 200 mesh by passing through an ASTM-certified brass sieve. These powdered glass samples were then dissolved using versions of ASTM glass dissolution procedures involving Na_2O_2/NaOH fusion with acid uptake (ASTM C 1317-95), and acid dissolution (ASTM C 1412-99). A third method for glass dissolution involving CsOH fusion was also examined in this study for both the active AN-102 glass and the LRM standard glass. This CsOH method is under investigation as a singular, comprehensive dissolution method for glass dissolution that could provide measured values for all analytes in the glass with a single dissolution method.

The peroxide fusion method used nominally 0.5 gram powdered glass samples added to 3 grams of Na_2O_2 and 2 grams of NaOH in Ni-crucibles. The resulting mixture was heated in a Thermolyne furnace at 700 °C for 15 minutes. The resulting mixture was then cooled and transferred to a 100-mL volumetric plastic flask. A volume of 25 mL of concentrated 15.7 Molar nitric acid was used to rinse the crucible and also added to the flask. The sample was then diluted to the 100-ml mark of the volumetric flask.

The acid dissolution method used nominally 0.5 gram powdered glass samples added to a wide mouth plastic bottle. Then 10 mL of 50% (~ 29 Molar) HF and 10 mL of concentrated 15.7 Molar HNO_3 were added. The bottle was capped and the mixture was heated in an oven at 105 °C for 2 hours. The mixture was then cooled, 70 mL of 0.6 Molar boric acid was added and the Teflon pressure re-sealed and heated for an additional hour. After cooling, the solution was diluted to 100 ml in a volumetric flask with deionized water.

The CsOH fusion method combines nominally 0.25 g of powdered glass with the CsOH pellet remaining after the pre-heating step (about 2.6-2.7 g) in a nickel crucible and heats at 500°C for 2 minutes. The powder form of $CsOH \cdot H_2O$ will splatter when placed in a muffle furnace at 500°C. In order to prevent the possible loss of sample from splattering, 3.0 g of $CsOH \cdot H_2O$ was first heated to 500°C in a nickel crucible for 2 minutes to remove excess moisture. The pellet that forms in the bottom of the crucible is much less deliquescent than the powder form of $CsOH \cdot H_2O$, but still must be protected from air to prevent re-absorption of moisture. After cooling the sample (preheated CsOH pellet plus glass) until warm, the fusion residue was dissolved first with de-ionized water containing 2-3 drops of 30% H_2O_2 and then 25 ml of 15.7 molar nitric acid. The acid solution was then diluted to 250 ml with de-ionized water.

All dissolved glasses were analyzed using the methods shown in Table 1 above.

Product Consistency Test

The Product Consistency Test (PCT) was performed at 90°C on the LAW glasses. The SRTC Task Plan for this work (SRTC document **BNF-003-98-0138, Rev. 0, Jan. 25, 2000**) specifies that both 90 °C and 40 °C Product Consistency Tests were to be performed on the LAW glasses. However, RPP-WTP project personnel subsequently requested that the 40 °C PCT not be performed. This change is reflected in the referenced Mod. No. M014 Section C: Statement of Work from November 2000 [1]. The durability was measured using the ASTM C-1285 standard nuclear waste glass durability test commonly referred to as the Product Consistency Test (PCT) [11]. 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. Triplicate tests were performed in sealed stainless steel vessels. The active AN-102 and Low Activity Reference standard LRM glasses were tested at 90°C +/- 2°C. 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

Feed Stream Characterization and Waste Glass Formulation

Decontaminated Feed Characterization

Decontaminated AN-102 supernate was analyzed by sampling each of the two similar 10-Liter carboys containing the Large C sample in duplicate according to the analyses shown in Table 1. No dilutions were performed on the decontaminated Large C composite supernate prior to submission to SRTC ADS for analysis. Table 2 below shows the data obtained from the duplicate analysis from each composite. Values reported in Table 2 with a less than sign, '<', represent the reported instrument detection limit value. Average values, standard deviation and % relative standard deviation determined from the four replicate values (1A, 1B, 2A and 2B) are shown for analytes that were detected above the instrument detection limit. In cases where some of the replicate analyte values were below detection limit, an upper limit average value is given. Average results from these four replicate analyses were transmitted to VSL, who then communicated to SRTC the appropriate amounts of the waste streams, composition of the glass-forming chemicals, and appropriate amounts of the glass-forming chemicals.

Review of the data shown in Table 2 indicates good agreement between the two large composite bottles. Radiochemical analysis of Cs-137 and Sr-90 shows precision in the range of 7% to 15%. Mass spectral analysis for mass-99 indicates Tc-99 precision in the two composites to be better than 1%. Other comparisons between the two composites for

the other analytical methods shown indicate good agreement between the two large composites, i.e., Na/Al from ICP-ES, TIC/TOC from wet chemical methods, K from AA and NO_3/NO_2 from IC-anions. Mass spectrometry values for Tc-99 in units of mg/L were converted to specific activity values in units of μCi/mL via Equation 1,

Equation 1:

$a = 3.5778E+05 \times (g / (t_Y \times M))$

Where:
a = activity in Curies (Ci)
3.5778E+05 = constant (Ci*years/g)
g = mass in grams
t_Y = ½ life of Tc-99 in years = 2.13E+05 years
M = mass number of Tc-99 = 99

Using the average mass spectral value for Tc-99 from Table 2 as 2.78 mg/L and solving Equation 1 above, one calculates an average specific activity for Tc-99 in the composite to be 4.72E-02 μCi/mL, via:

$a(Ci/L) = 3.5778E+05 \times (0.00278\ g/L / (2.13E+05 \times 99))$

a(Ci/L) = 4.72E-05 Ci/L, or 4.72E-02 μCi/mL

Table 2. AN-102 Large C Concentrate Composite Analyses

Decontaminated Large C Composites

	Composite 1A		Composite 1B		Composite 2A		Composite 2B		Average*	St. Dev.*	%Rel. St. Dev.*
Radiochemical											
	(uCi/mL)		(uCi/mL)		(uCi/mL)		(uCi/mL)		(uCi/mL)	(uCi/mL)	(%)
Co-60	3.81E-02		4.10E-02		4.15E-02		3.92E-02		3.99E-02	1.56E-03	3.9
Cs-137	4.54E-02		4.66E-02		6.17E-02		5.75E-02		5.28E-02	8.06E-03	15.3
Eu-154	2.93E-02		3.63E-02		3.71E-02		3.41E-02		3.42E-02	3.49E-03	10.2
Eu-155	2.27E-02		2.38E-02		2.45E-02		2.18E-02		2.32E-02	1.18E-03	5.1
Am-241	1.61E-02	<	1.38E-02		1.42E-02		2.10E-02	<	1.63E-02	NA	NA
Total Alpha	8.95E-02		6.09E-02		7.59E-02		3.45E-02		6.52E-02	2.36E-02	36.1
Total Beta	4.38		4.46		4.11		4.27		4.31	1.52E-01	3.5
Sr-90	1.41		1.21		1.41		1.37		1.35	9.52E-02	7.1
ICP-Mass Spectroscopy											
	(mg/L)		(mg/L)		(mg/L)		(mg/L)		(mg/L)	(mg/L)	(%)
mass 99	2.77E+00		2.78E+00		2.78E+00		2.81E+00		2.78E+00	1.98E-02	0.7
**Mass 99 (Tc), μCi/mL	4.69E-02		4.71E-02		4.71E-02		4.77E-02		4.72E-02	3.36E-04	0.7
mass 230	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 231	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 232 (Th)	1.51E+00		1.46E+00		1.46E+00		1.45E+00		1.47E+00	2.57E-02	1.8
mass 233	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 234 (U)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 235 (U)	8.88E-03		8.68E-03		1.07E-02		1.03E-02		9.63E-03	9.96E-04	10.3
mass 236 (U)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 237 (Np)	8.78E-02		8.93E-02		8.82E-02		8.79E-02		8.83E-02	6.76E-04	0.8
mass 238 (Pu & U)	8.64E-01		8.77E-01		9.39E-01		9.35E-01		9.03E-01	3.89E-02	4.3
mass 239 (Pu)	1.50E-02		1.54E-02		1.35E-02		1.41E-02		1.45E-02	8.64E-04	6.0
mass 240 (Pu)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 241 (Am & Pu)	7.46E-03		7.83E-03		7.09E-03		7.69E-03		7.52E-03	3.25E-04	4.3
mass 242 (Pu)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 243 (Am)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 244 (Cm)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 245 (Cm)	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
mass 246	< 3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	NA	NA
ICP-Emission Spectroscopy											
	(mg/L)		(mg/L)		(mg/L)		(mg/L)		(mg/L)	(mg/L)	(%)
Al	5.39E+03		4.51E+03		5.70E+03		5.84E+03		5.36E+03	6.01E+02	11.2
B	1.37E+01		1.14E+01		1.39E+01		1.44E+01		1.34E+01	1.32E+00	9.9
Ba	< 1.82E-01		4.69E-01	<	1.82E-01	<	1.82E-01	<	2.54E-01	NA	NA
Ca	1.12E+02		9.40E+01		1.18E+02		1.20E+02		1.11E+02	1.19E+01	10.8

Table 2, Continued

ICP-ES	Composite 1A	Composite 1B	Composite 2A	Composite 2B	Average*	St. Dev.*	%Rel. St. Dev.*
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(%)
Cd	1.99E+01	1.67E+01	2.12E+01	2.19E+01	1.99E+01	2.30E+00	11.6
Co	1.57E+00	1.95E+00	1.46E+00	1.24E+00	1.56E+00	2.96E-01	19.0
Cr	7.07E+01	5.93E+01	7.46E+01	7.64E+01	7.03E+01	7.67E+00	10.9
Cu	3.58E+00	3.39E+00	3.61E+00	3.76E+00	3.58E+00	1.53E-01	4.3
Fe	2.14E+00	2.15E+00	1.70E+00	1.73E+00	1.93E+00	2.47E-01	12.8
La	1.25E+00	2.16E+00	< 1.00E+00	< 1.00E+00	< 1.35E+00	NA	NA
Li	1.83E-01	5.34E-01	< 1.82E-01	< 1.82E-01	< 2.70E-01	NA	NA
Mg	< 9.10E-02	9.70E-02	< 9.10E-02	< 9.10E-02	< 9.25E-02	NA	NA
Mn	8.16E-01	7.83E-01	7.91E-01	8.00E-01	7.98E-01	1.42E-02	1.8
Mo	2.25E+01	1.81E+01	2.21E+01	2.32E+01	2.15E+01	2.28E+00	10.6
Na	1.10E+05	1.07E+05	1.16E+05	1.13E+05	1.12E+05	3.54E+03	3.2
Ni	1.23E+02	1.03E+02	1.33E+02	1.36E+02	1.24E+02	1.53E+01	12.3
P	7.05E+02	5.71E+02	7.30E+02	7.42E+02	6.87E+02	7.88E+01	11.5
Pb	4.40E+01	3.92E+01	4.73E+01	4.71E+01	4.44E+01	3.75E+00	8.4
Si	4.35E+01	1.21E+01	3.91E+01	4.09E+01	3.39E+01	1.46E+01	43.1
Sn	1.19E+01	9.82E+00	1.10E+01	1.12E+01	1.10E+01	8.72E-01	7.9
Sr	1.27E+02	1.02E+02	1.37E+02	1.40E+02	1.26E+02	1.70E+01	13.5
Tc	2.83E+00	2.79E+00	2.41E+00	2.05E+00	2.52E+00	3.64E-01	14.4
Ti	3.05E-01	7.63E-01	< 1.82E-01	< 1.82E-01	< 3.58E-01	NA	NA
V	6.07E-01	1.29E+00	< 2.73E-01	< 2.73E-01	< 6.10E-01	NA	NA
Zn	1.40E+00	1.25E+00	1.30E+00	1.22E+00	1.29E+00	7.83E-02	6.1
Zr	< 3.64E-01	< 3.64E-01	5.05E-01	< 3.64E-01	< 3.99E-01	NA	NA
IC Anions							
Cl	1.60E+03	1.56E+03	1.56E+03	1.59E+03	1.57E+03	1.83E+01	1.2
F	7.69E+02	7.71E+02	7.77E+02	7.46E+02	7.66E+02	1.36E+01	1.8
Formate	4.58E+03	4.65E+03	4.60E+03	4.45E+03	4.57E+03	8.46E+01	1.9
NO2	3.65E+04	3.66E+04	3.62E+04	3.56E+04	3.62E+04	4.72E+02	1.3
NO3	8.56E+04	8.69E+04	8.65E+04	8.48E+04	8.59E+04	9.53E+02	1.1
SO4	5.59E+03	5.51E+03	5.48E+03	5.32E+03	5.47E+03	1.12E+02	2.1
PO4	2.68E+03	1.73E+03	1.73E+03	2.64E+03	2.20E+03	5.38E+02	24.5
Oxalate	1.15E+03	1.00E+03	9.98E+02	1.12E+03	1.07E+03	7.91E+01	7.4
sp. G. (g/mL)	1.2255	1.2283	1.229	1.2323	1.23E+00	2.79E-03	0.2
Insol. solids(wt%)	< 0.002	< 0.002	< 0.002	< 0.002	< 2.00E-03	NA	NA
soluble solids (wt%)	30.98	30.58	32.04	32.43	3.15E+01	8.70E-01	2.8
Free OH (M)	1.1531	1.207	1.2879	1.3229	1.24E+00	7.70E-02	6.2
Atomic Absorption							
	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(%)
K	1.06E+03	9.39E+02	8.98E+02	9.11E+02	9.51E+02	7.21E+01	7.6
As	2.84E-01	2.36E-01	2.14E-01	2.85E-01	2.55E-01	3.55E-02	13.9
Se	2.19E-01	2.65E-02	9.65E-02	2.25E-01	1.42E-01	9.70E-02	68.4
Hg	< 2.40E-02	< 1.98E-02	< 1.76E-02	< 1.65E-02	< 1.95E-02	NA	NA
Total U	< 1.00E+00	< 1.00E+00	< 1.00E+00	< 1.00E+00	< 1.00E+00	NA	NA

Table 2, Continued

Ion Selective Electrode	Composite 1A	Composite 1B	Composite 2A		Composite 2B		Average*	St. Dev.*	%Rel. St. Dev.*
	(mg/L)	(mg/L)	(mg/L)		(mg/L)		(mg/L)	(mg/L)	(%)
Cl	179.6	188.5	213.2		218.34		2.00E+02	1.88E+01	9.4
F	119.4	105.85	93.78	<	10	<	8.23E+01	NA	NA
TIC/TOC									
	(mg/L)	(mg/L)	(mg/L)		(mg/L)		(mg/L)	(mg/L)	(%)
TIC	5782	5618	6294		5656		5.84E+03	3.12E+02	5.3
TOC	11432	11617	11814		10732		1.14E+04	4.71E+02	4.1

Notes:

* Average (Composite 1A, 1B, 2A, 2B), 1-sigma Standard Deviation (Composite 1A, 1B, 2A, 2B) and % Relative Standard Deviation (%RSD calculated from [(St.Dev./Average)*100])

(NA) = Not Applicable due to one or more replicate analyte values determined below the instrument detection limit.

* * Tc-99 specific activity (μCi/mL) calculated from ICP-MS value. See text.

Waste Glass Formulation

Results of the AN-102 Large C composite analyses were transmitted to the VSL to develop a glass formers recipe for the waste stream. The recipe uses a mixture of ten different minerals and added sugar as a reductant. The target waste loading for this AN-102 glass is nominally 11.8 wt% Na_2O. This waste loading is lower than the original Specification C Statement of Work target of ~ 20 wt% Na_2O due to the use of a non-sulfate pretreated supernate as feed. The current Modification 014 Specification C Statement of Work specifies a waste loading for Env. C of > 10 wt% based on Na_2O. Appendix A contains details of the glass recipe provided to SRTC from VSL. The sodium and sulfur target loading in the glass is specified as 11.8 and 0.36 wt% as Na_2O and SO_3, respectively. The product of these two numbers is 4.248, which is close to the upper target limit value of 5. The product of the sodium and sulfur oxide components equal to or less than 5 is referred to as the 'rule of 5' guidance in formulating the sulfate-containing wastes. This target product value of 5 is an upper limit value and is not strictly used as the actual target value for glass formulation. Concurrence with this particular glass recipe was also obtained from RPP-WTP personnel. The recipe shows that ten different minerals were to be used as the glass former mixture.

Glass Former Blend Preparation and Analysis

One batch of glass formers was prepared for this study using the recipe for the AN-102 glass supplied by VSL. The blend of glass formers identified as 9382-129 was weighed on a calibrated balance and blended with an automatic shaker for ten minutes. The blend was split into smaller representative samples with a Quantachrome Sieving Rotary Riffler. The riffler device consists of a central container that when filled, is shaken at a high frequency causing the contents to be slowly fed into a rotating tray of eight equal sized stainless steel pans. Splitting a mixture with the riffler device ensures that the resulting portions are representative of the starting batch of material. The glass former blend mixtures were split into $1/8^{th}$ samples and seven samples were recombined. One $1/8^{th}$ sample was split into $1/64^{th}$ samples. A $1/64^{th}$ sample was again split into eight parts for ADS chemical analysis. The glass former blend was prepared as a one-half VSL batch as shown in Table 3. More detailed information on the glass formers used can be found in Reference #8.

Table 3. Glass Former Blend NB#-9382-129 *

Glass Former Identification	Batch Wt. (grams)	Actual Wt. (grams)
Kyanite, Kyanite Mining. Raw–325	65.69	65.70
Boric Acid, USBorax, Gran.	119.76	119.79
Wollastonite, NYCO-325, NY	89.15	89.18
Fe2O3, Alfa Aesar, (VSL)	39.95	39.96
Li2CO3, Cyprus Foote	45.04	45.07
Olivine, Unimin, #180	20.93	20.98
Silica, US SCS-75, MCOK.	215.12	215.13
Titania, Chemalloy, (VSL)	7.75	7.78
Zinc Oxide, ZCA, (VSL)	19.95	19.95
Zircon Flour, Amer. Miner. (VSL)	30.86	30.86
Sugar, (local)	10	Added Later
Total	654.17	654.20

*One Half VSL No. AN102 Recipe

The glass former blend was analyzed by SRTC/ADS personnel using Na_2O_2 fusion. The analytical data and the calculated blend composition were compared. Table 4 below shows the target weight percent elemental compositions of the blend, the average analyzed elemental compositions and the statistical data indicating percent bias for each result. The percentage difference accounts for the precision in each triplicate analysis vs. the target. In general, the Na_2O_2 fusion provided reliable results and all values were found to have a difference of less than +/- 5% with the exception of zirconium and titanium, which were analyzed to be slightly higher than the target (8% and 10% bias, respectively. These data compare well to previous glass former dissolution data presented in the Env. A, AN-103 technical report for use of Na_2O_2 fusion [12]. Other dissolution methods such as Acid dissolution and CsOH dissolution were not pursued for the Env. C AN-102 glass former batch minerals due to their previous performance in attempts to dissolve the glass former batch minerals in the Env. A, AN-103 work [12]. Previous use of these methods provided low bias results indicating incomplete dissolution of the glass former minerals [12].

Table 4. Statistical Analysis Comparing Calculated Glass Former Elemental Composition to Analyzed

9382-129 Batch

Element	Target (Ele.Wt%)	Na_2O_2 Average (of 3), Ele.Wt%	% Difference
Al	2.92	2.96	1.41
B	3.23	3.20	1.05
Ca	4.66	4.78	2.68
Cr	0.00	.02	
Fe	4.59	4.68	1.91
Li	1.29	1.33	3.13
Mg	0.94	0.96	2.00
Si	22.0	22.8	3.35
Ti	0.78	0.83	10.0
Zn	2.45	2.57	4.85
Zr	2.32	2.51	8.02

Vitrification

Mixing of Glass Formers and Waste Streams

Portions of the glass former batch of minerals were mixed with known volumes of either simulant AN-102 supernate or the active AN-102 pretreated supernate according to the recipe provided for the radioactive AN-102 glass (See Appendix A). The simulant solution recipe is shown in Table 5. This simulant was made to contain only the six salts, NaOH, $Al(OH)_3$, Na_2SO_4, Na_2CO_3, $NaNO_3$ and $NaNO_2$. The simulant derives from the previously shown Large C composite characterization data (See Table 2) and was targeted to be a simple representative salt solution for use as the simulant glass feed.

Crucible-Scale Vitrification

Several crucible-scale vitrification experiments were conducted during the course of this study. Table 6 documents the details of all tests. The initial vitrification Tests #1 - #3 involved simulant supernate and were conducted in a Thermolyne furnace capable of 1200°C and did not use the sealed quartz offgas system. These tests used a mixed slurry (simulant waste supernate and glass formers and sugar) and used open crucibles in a

static mode, i.e., no stirring of the crucibles was performed after the initial mixing of supernate and glass formers and sugar. The primary goal of these crucible-scale vitrification tests were to determine if foaming of the slurries within the crucibles during evaporation/calcining was significant, i.e., determine if the material remained completely inside the crucibles during the melt. Initially, we attempted to produce 135 grams of AN-102 simulant glass in a 600 mL crucible. The 135 gram glass target was determined from approximately ½ of the total glass made for regulatory analyses in previous Envelope A, AN-103 testing [12]. The Test #1 was discontinued at about 750°C due to excessive overflow of the mixture over the top of the 600-mL crucible. The next Test #2 attempted produce 120 grams of product glass in the 600-mL crucible. Intermittent observations of the crucible melt in this test indicated some foaming of the contents at levels above the top of the 600-mL crucible. However, the material did not foam over and a product amount of 120.7 grams of glass was produced. Final Test #3, targeting a product of 110 grams of glass, showed successful containment of the crucible material throughout the melt process. It was thus decided to use the 110 g glass batch target as the proper crucible loading for the radioactive AN-102 crucible vitrification tests.

Radioactive AN-102 crucible vitrification Tests #4-#5 took place in the custom designed Deltech furnace with sealed quartz offgas containment. The target mass of glass shown in the last column of Table 6 derives from the calculated glass mass from the glass former recipe (Appendix A). Table 7 shows the calculated specific activity values expected from blending the specified volume of AN-102 decontaminated supernate (86 mL) to make the product glass (111 g). Average radionuclide values were used from Table 2 composite supernate characterization data.

Figure 3 shows temperature vs. time and condensate volume vs. time plots for the active AN-102 vitrification test. The temperature plot in Figure 3 indicates a slow increase in temperature to ~ 250 °C, followed by a more rapid temperature increase up to 700 °C. From 700 °C to 800 °C, the temperature was increased very slowly to prevent overflow of the dried material in the 600 mL crucible. A rapid increase in temperature was used from 800 °C to the melt temperature of 1150 °C, followed by 4 hours of melting at 1150 °C. Note that the controlled canister cooling segment shown in Figure 3, in the temperature range of 1022 °C to 400 °C, was maintained to ensure that the actual cooling matched that of the targeted cooling curve previously shown in Figure 2 to within +/- 5°C within this 1022 °C to 400 °C temperature range. Also note from Figure 3 that no further significant condensate was collected after about 6/20/00 11:30 at the ~ 700 °C temperature, i.e., the condensate volume data shows no further significant increase after this time and temperature. The very initial increase in condensate of about 10 mL occurring below the 100 °C temperature range is most likely due to moisture condensing out of the humid air that was pulled though the offgas system by the vacuum. The vacuum was decreased after this initial condensate volume was noticed and the condensate volume remained unchanged until further heatup caused actual evaporation of the slurry sample in the crucible.

Table 5. Simulant Salt Solution for AN-102 Vitrification Simulant Testing

Target Species	**Concentration**	**Salt**
	mg/L	
Al	5,361	$Al(OH)_3$
CO_3^{2-}	39,700	Na_2CO_3
OH^-	21,000	NaOH
NO_3^-	85,942	$NaNO_3$
NO_2^-	36,218	$NaNO_2$
Na	111,514	Na_2CO_3, NaNOH, $NaNO_3$, $NaNO_2$, Na_2SO_4
SO_4^{2-}	5,475	Na_2SO_4

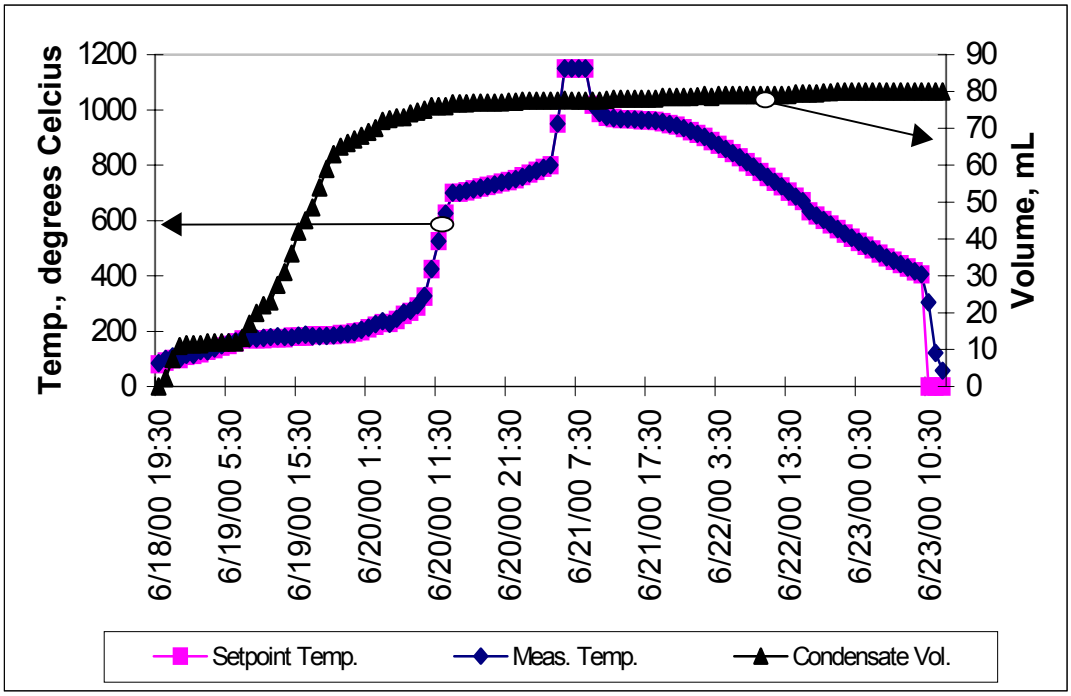
Table 6. Details of Crucible-Scale Vitrification Experiments

Test/Date	Date	Furnace	Mass Glass Formers & Sugar (g)	Volume Liquid (mL)	Crucible	Target Glass Mass (g)
Simulant AN102 Vitrification Tests						
#1	6/13/00	Thermolyne	134.5 2 g sugar	106	600-mL Pt/Au	135
#2	6/14/00	Thermolyne	119.5 1.8 g sugar	94.2	600-mL Pt/Au	120 Actual = 120.7 g
#3	6/15/00	Thermolyne	109.5 1.65 g sugar	86.35	600-mL Pt/Au	110 Actual = 110 g
Active AN-102 Tests						
#4,	6/18/00	Deltech	109.5 1.65 g sugar	86	600-mL Pt/Au	110
#5,	6/25/00	Deltech	109.5 1.65 g sugar	86	600-mL Pt/Au	110

Table 7. Expected Average Radionuclide Specific Activities in AN-102 Glass

Radionuclide	Average Concentration (uCi/mL) (from Table 2)	Volume Liquid (mL)	Mass Glass (g)	Expected Specific Activity in Glass (uCi/g)
Cs-137	5.28E-02	86	111	4.09E-02
Sr-90	1.35	86	111	1.04
Tc-99	4.72E-02	86	111	3.66E-02

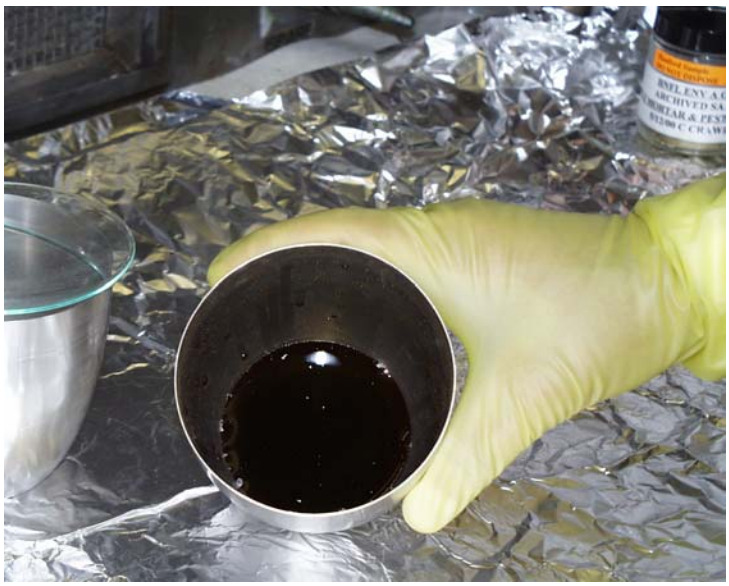
Figure 3. Temperature and Condensate Volumes vs. Time for AN-102 Active Vitrification Tests.



Glass Characterization

Powdered glass samples of the AN-102 glass were analyzed by XRD and SEM/EDAX analysis. The crushed glass samples were obtained from excess glass that was ground and sieved for the PCT. The results are shown in Appendix B. No crystalline phases were observed in the powdered glass specimens as examined by either XRD or SEM. These analytical results agree with observations of the AN-102 glass by visual inspection that showed the glass to be dark and shiny with no evidence of any secondary phases present. Digital photographs of the top portion of one of the AN-102 glass crucible vitrifications are shown in Figure 4 below. The XRD spectrum of the Envelope C AN-102 glass shown in Figure B1 of Appendix B, indicates presence of a peak that is identified as a Cr/Fe/Ni component. This peak is likely due to presence of steel remnants in the powdered glass from use of a tungsten carbide blade grinder with steel compartment. Also, some of the high magnification images and EDAX data indicate presence of steel contaminates in the glass that was prepared for PCT using a tungsten carbide blade grinder with steel compartment (See Figures B6 and B8 of Appendix B). It should be noted that the steel contaminants do not interfere with the PCT since the PCT is carried out in stainless steel vessels. Similar microstructure results to those discussed above for this AN-102 Envelope C glass were reported in the previous SRTC technical report on the AN-103 Envelope A glass [12].

Figure 4. Digital Photographs of the AN-102 Crucible Melt



Radioactive AN-102 and the LRM glasses prepared for dissolution for analytical characterization were prepared using an agate ball/mill grinder. Dissolved glasses were analyzed by ICP-ES, AA(K) and Total Uranium methods to determine the inorganic components present in the glass matrix. Results for the two different glasses studied are shown in Table 8 (Radioactive AN-102) and Table 9 (LRM). Oxide values were summed to obtain the totals for all dissolution methods performed on all glasses. As noted in both Tables 8 and 9, sodium and nickel values were indeterminate for the

peroxide fusion method that used sodium as reagent and were performed in nickel crucibles. Nickel crucibles were also used in the CsOH fusions. Boric acid was used as reagent in the acid dissolutions preventing boron to be determined in these samples. Note that no analytical values were obtained from the SRTC analyses of the glasses for the elementals Cl, F and S. These values were determined in separate Regulatory analyses and are reported in Reference 5.

Table 8 shows elemental weight percent of all analyzed components in the active AN-102 glass. The elemental values were converted to their oxide components by multiplying by an oxide conversion factor. The target oxide composition shown in Table 8 is taken from the VSL glass recipe discussed earlier (Appendix A). The peroxide fusion data, the CsOH dissolution data and acid dissolution data were averaged and compared to the target. The last column in Table 8 indicates that the average composition of the glass determined by both peroxide fusion, CsOH fusion and acid dissolution is within 97% to 114% of the target for major elements present in the glass at 0.5 wt% or more on an oxide basis. This comparison of analyzed oxide to target values is nominally within the 10% analytical uncertainty of the measurements used to determine the various components in the glass. Elemental analysis was not performed for chlorine, fluorine and sulfur in the glass analyzed in SRTC. Values shown in Table 8 for these elements represent the maximum amounts that could be present from waste feed concentrations.

Table 9 shows similar data for the standard LRM glass. The target oxide composition shown in Table 9 is taken from averaging all of the reported round-robin analytical data reported by Ebert and Wolf [9a]. The peroxide fusion data, the CsOH data and acid dissolution data were averaged and compared to the target. The last column in Table 9 indicates that the average composition of the glass determined by both peroxide fusion and acid dissolution for major components > 0.5 wt% as oxide in the glass is within 99% to 111% of the target excluding potassium. Previous analysis of the LRM glass [12] indicates better agreement for analyzed K vs. target than the present data.

Table 8. Elemental and Oxide Composition of Active AN-102 Glass

AN102 Radioactive Glass									
Element	Peroxide Fusion %Elemental	CsOH %Elemental	Acid %Elemental	Oxide	Peroxide Fusion % Oxide	CsOH % Oxide	Acid % Oxide	Target % Oxide	% of Target
Al	3.51	3.47	3.10	Al2O3	6.64	6.55	5.86	6.15	103
B	3.36	3.22	[1]	B2O3	10.81	10.37	[1]	10.13	105
Ba	<0.01	0.01	<0.01	BaO	<0.01	0.01	<0.01	0.00	
Ca	4.71	4.44	4.24	CaO	6.59	6.22	5.94	6.42	97
Cd	0.01	0.01	0.00	CdO	0.01	0.01	0.00	0.00	
Cr	0.12	0.06	0.03	Cr2O3	0.18	0.08	0.04	0.02	
Cu	0.02	0.02	0.01	CuO	0.03	0.03	0.01	0.00	
Fe	5.29	5.00	4.50	Fe2O3	7.57	7.15	6.43	6.49	109
K	0.15	[3]	0.07	K2O	0.18	[3]	0.09	0.09	
La	<0.02	0.07	0.01	La2O3	<0.02	0.08	0.01	0.00	
Li	1.29	1.27	1.24	Li2O	2.78	2.73	2.67	2.74	99
Mg	1.01	0.98	0.95	MgO	1.67	1.62	1.58	1.52	107
Mn	0.11	0.04	0.02	MnO2	0.17	0.06	0.04	0.00	
Mo	0.01	0.01	0.00	MoO3	0.02	0.02	0.01	0.00	
Na	[2]	9.13	8.98	Na2O	[2]	12.31	12.11	11.80	103
Ni	[2]	[2]	0.02	NiO	[2]	[2]	0.03	0.01	
P	0.09	0.09	0.10	P2O5	0.12	0.11	0.13	0.13	
Pb	<0.05	0.12	0.02	PbO	<0.05	0.13	0.02	0.00	
Si	23.13	23.48	22.76	SiO2	49.48	50.23	48.70	46.75	106
Sn	0.03	0.04	0.02	SnO2	0.04	0.06	0.03	0.00	
Ti	0.77	0.80	0.74	TiO2	1.29	1.33	1.24	1.13	114
V	0.01	0.03	0.01	V2O5	0.02	0.05	0.01	0.00	
Zn	2.63	2.53	2.44	ZnO	3.27	3.15	3.04	3.03	104
Zr	2.47	2.39	2.39	ZrO2	3.34	3.22	3.23	3.03	108
U	<0.01	[3]	<0.01	UO2	<0.01	--	<0.01	0.00	
Cl [4]				Cl	0.12	0.12	0.12	0.12	
F [4]				F	0.06	0.06	0.06	0.06	
S [4]				SO3	0.36	0.36	0.36	0.36	
				Totals:*	107.07	106.09	102.35	99.99	

Notes:

(1) Boric acid used to dilute to mark in acid dissolved glass.
(2) Sodium used in NaOH/Na2O2 fusion in Ni-crucibles. Ni-crucibles used in CsOH fusion.
(3) Total Uranium and potassium not measured in CsOH dissolved glass.
(4) These and other elements not measured in AN-102 glasses analyzed at SRTC. See Regulatory Analyses Technical Report for analyzed values [5]. Target oxide values for these elements included in oxide Totals.

*Total oxide wt% values for Peroxide Fusion method calculated by adding average sodium values from CsOH and Acid methods, and nickel values from Acid Dissolution data. Total oxide wt% values for CsOH calculated by adding nickel value from Acid Dissolution data. Total oxide wt% values for Acid Dissolution method calculated by adding boron value from average of Peroxide Fusion data and CsOH data.

Table 9. Elemental and Oxide Composition of LRM Standard Glass

LRM Standard Glass									
Element	Fusion %Elemental	CsOH %Elemental	Acid %Elemental	Oxide	Fusion % Oxide	CsOH % Oxide	Acid % Oxide	Target % Oxide	% of Target
Al	5.00	5.48	4.83	Al2O3	9.45	10.35	9.13	9.54	101
B	2.39	2.53	[1]	B2O3	7.71	8.15	[1]	7.90	100
Ba	<0.01	0.01	<0.01	BaO	<0.01	0.01	<0.01	0.00	
Ca	0.37	0.41	0.36	CaO	0.52	0.58	0.50	0.54	99
Cd	0.15	0.15	0.14	CdO	0.17	0.18	0.16	0.16	
Cr	0.17	0.19	0.14	Cr2O3	0.25	0.28	0.20	0.19	
Cu	0.01	0.01	0.06	CuO	0.01	0.01	0.08	[5]	
Fe	1.03	1.11	0.97	Fe2O3	1.48	1.59	1.39	1.42	105
K	1.04	[3]	0.94	K2O	1.25	[3]	1.13	1.48	80
La	<0.10	0.04	0.01	La2O3	<0.12	0.05	0.01	0.02	
Li	0.05	0.05	0.05	Li2O	0.10	0.12	0.10	0.11	
Mg	0.06	0.07	0.06	MgO	0.10	0.12	0.11	0.10	
Mn	0.10	0.07	0.06	MnO2	0.16	0.11	0.09	0.08	
Mo	0.10	0.08	0.07	MoO3	0.15	0.11	0.10	0.10	
Na	[2]	16.14	15.02	Na2O	[2]	21.76	20.24	20.03	105
Ni	[2]	[2]	0.15	NiO	[2]	[2]	0.19	0.19	
P	0.24	0.26	0.27	P2O5	0.55	0.59	0.62	0.53	111
Pb	<0.23	0.17	0.10	PbO	<0.25	0.18	0.11	0.10	
Si	24.79	28.01	25.64	SiO2	53.04	59.92	54.84	54.26	106
Sn	<0.42	0.03	0.02	SnO2	0.54	0.04	0.03	0.03	
Ti	0.07	0.07	0.06	TiO2	0.12	0.12	0.10	0.11	109
V	<0.01	<0.01	<0.01	V2O5	<0.02	<0.02	<0.00	[5]	
Zn	0.05	0.01	0.02	ZnO	0.06	0.02	0.02	[5]	
Zr	0.71	0.78	0.75	ZrO2	0.96	1.06	1.02	0.93	109
U	<0.01	[3]	<0.01	UO2	<0.01	[3]	<0.01	0.00	
Cl [4]				Cl	0.80	0.80	0.80	0.80	100
F [4]				F	1.00	1.00	1.00	1.00	100
S [4]				SO3	0.20	0.20	0.20	0.20	100
				Totals:*	100.18	107.55	100.11	99.80	

Notes:

(1) Boric acid used to dilute to mark in acid dissolved glass.
(2) Sodium used in NaOH/Na2O2 fusion in Ni-crucibles. Ni-crucibles used in CsOH fusion.
(3) Potassium and Total Uranium not measured in CsOH dissolved glass.
(4) These and other elements not measured in AN-102 glasses analyzed at SRTC. See Regulatory Analyses Technical Report for analyzed values [5]. Target oxide values for these elements included in oxide Totals.
(5) Components not added to LRM glass, See reference 9a.

*Total oxide wt% values for Peroxide Fusion method calculated by adding average sodium values from CsOH and Acid methods, and nickel values from Acid Dissolution data. Total oxide wt% values for CsOH calculated by adding nickel value from Acid Dissolution data. Total oxide wt% values for Acid Dissolution method calculated by adding boron value from average of Peroxide Fusion data and CsOH data.

Dissolved glasses were analyzed by various radiochemical methods indicated in Table 1 shown previously. All reported analytical data is shown in Table 10 for the glasses dissolved by peroxide fusion. Similar data is shown in Table 11 for glasses dissolved by acid dissolution. All results were reported in units of dpm/mL for these samples. These values were converted to specific activities of radionuclides in the glasses (μCi/g glass) using Equation 2 below:

Equation 2:

dpm/mL x (mL/grams glass) x (1 min/60 s) x (1Ci/3.7E+10 dps) x 1E+06 μCi/1Ci

The exact amounts of each glass weighed out in the dissolution process to produce the 100-mL solutions are noted at the bottom of Tables 10 and 11. All radionuclide values reported in Tables 10 and 11 were analyzed directly except for Y-90 and Ba-137m. Yttrium-90 (half-life = 2.671 days) is a 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. Barium-137m 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.

Tables 10 and 11 indicate that Strontium-90 and Yyttrium-90 are the predominant radionuclides in the active AN-102 glass at about 1 μCi/g levels. Cesium-137 and Ba-137m and Tc-99 are present at nominally much lower specific activities (~ 0.02 to ~ 0.03 μCi/g) in the active AN-102 glass. Conversion of the tabulated radionuclide values from units of μCi/g glass to Ci/m^3 can be calculated using the measured density of both the Env. C glass = 2.87 g/cm^3 (Reference 5) and LRM glass = 2.52 g/cm^3 (Reference 9b) per Equation 3 below.

Equation 3:

Env. C glass:
1 μCi/g x 2.87 g/cm^3 x 1 Ci/ 1E+06 μCi x $(100\ cm/1\ m)^3$ = 2.87 Ci/m^3

LRM glass:
1 μCi/g x 2.52 g/cm^3 x 1 Ci/ 1E+06 μCi x $(100\ cm/1\ m)^3$ = 2.52 Ci/m^3

Section C, Specification 2.2.2.8 of the RPP-WTP-DOE/ORP contract indicates that average radionuclide concentration limitations shall be less than 3 Ci/m^3 for Cs-137, < 20 Ci/m^3 for Sr-90 and < 0.1 Ci/m^3 for Tc-99 for the ILAW glasses[1]. The average cesium concentration as design basis is actually 0.3 Ci/m^3. Per communication with Mike Johnson of RPP/WTP, this latter value of 0.3 Ci/m^3 is the more recent design basis value put in place to reduce the level of shielding needed in a LAW vitrification plant.

Consideration of previous Table 7 data for estimated values of the Cs-137, Sr-90 and Tc-99 radionuclides in the AN-102 glass and analyzed data from Tables 10 and 11 indicate these radionuclide levels are below the specified limits for the LAW Envelope C glass. Analyzed values for these radionuclides presented in Tables 10 and 11 are near the estimated values from Table 7. Analyzed values for Cs-137 and Sr-90 give better agreement vs. estimated values than does the analyzed Tc-99 values vs. estimated values. Analyzed values for Tc-99 were somewhat lower than the value estimated from the feed Tc-99 concentration. Volatilization during open-crucible vitrification or during aggressive glass dissolutions (peroxide fusion and acid digestion) could account for this observation. A singular value for Tc-99 (radiochemical counting value of 1.6E-01 μCi/g from Table 11 acid dissolved glass) is an order of magnitude higher than all other reported radiochemical and mass spectral values for the AN-102 glass. Comparison of this Tc-99 value with the predicted value from Table 7 (3.66E-02 μCi/g) also indicates that this value is likely inaccurate. One explanation for this high Tc-99 analyzed value could be interference from the relatively higher amounts of the beta decaying Sr and Y radionuclides present in the glass. It was noted in the radiochemical analysis of this sample that evidence of non-Tc-99 beta energies were present, which would contribute to the high bias value in this case.

Conversion of the units shown in Table 10 and Table 11 as μCi/g to units of ηCi/g indicates that the transuranic (TRU) content of the AN-102 glass samples is ~ 22 ηCi/g measured total alpha (0.022 μCi/g), or ~ 24 ηCi/g (0.024 μCi/g) from summation of the Pu-238, 239 and 240, Am-241 and Cm-244 isotope alpha-emitters. These values are at least 4X lower than the DOE/ORP contract limit of 100 ηCi/g [1]. Similarly, Table 11 shows that the TRU content of the AN-102 glass sample is ~ 22 ηCi/g measured total alpha, or ~ 20 ηCi/g from summation of the Pu/Am/Cm alpha-emitters. According to the Code of Federal Regulations (CRF) (10 CFR 61.56, Waste Classification, Table 1), a limit of 3,500 ηCi/g is provided for the beta-emitter radioisotope Pu-241. Both Tables 10 and 11 indicate that Pu-241 was not detected in either dissolved AN-102 glass to the detection level of < 2.9 ηCi/g. Overall agreement between various radionuclides analyzed by peroxide fusion (Table 10) and acid dissolution (Table 11) is very good.

Table 10. Radionuclide Analyses for Peroxide Fusion Dissolved Glasses

Radionuclide Analyses for Peroxide Fusion Dissolved Glasses

Radionuclide		AN-102 Radioactive Glass (dpm/mL)	AN-102 Radioactive Glass (uCi/g)	AN-102 Radioactive Glass $Ci/m^{3\,\#}$		LRM Standard Glass (dpm/mL)	LRM Standard Glass (uCi/g	LRM Standard Glass $Ci/m^{3\,\#}$		Reagent Blank (dpm/mL)	Reagent Blank (uCi/mL)
Total Alpha		241	2.17E-02	6.23E-02	<	6.62	5.93E-04	1.49E-03	<	4.51	2.03E-06
Total Beta		3.54E+04	3.19E+00	9.15E+00	<	202	1.81E-02	4.56E-02	<	13.5	6.08E-06
Sr-90		1.27E+04	1.14E+00	3.28E+00	<	65.2	5.84E-03	1.47E-02	<	65.2	2.94E-05
Estimated ##			1.04								
Y-90		1.27E+04	1.14E+00	3.28E+00	<	65	5.84E-03	1.47E-02	<	65.2	2.94E-05
Tc-99	<	137	1.23E-02	3.54E-02	<	6.38	5.72E-04	1.44E-03		16.4	7.39E-06
Tc-99(ICP-MS) †		5.4 ug/L	1.83E-02	5.26E-02	<	0.09	3.04E-04	7.65E-04	<	0.08	1.36E-06
Estimated ##			3.66E-02								
Pu-238	<	7.16	6.45E-04	1.85E-03	<	0.556	4.98E-05	1.26E-04	<	2.64	1.19E-06
Pu-239/240	<	3.57	3.22E-04	9.23E-04	<	6.2	5.55E-04	1.40E-03	<	1.05	4.73E-07
Pu-241	<	--	2.92E-03	4.19E-05	<	--	3.38E-03	4.28E-05	<	--	1.26E-05
Am-241		157.0	1.41E-02	4.06E-02		13.5	1.21E-03	3.05E-03		8.39	3.78E-06
Cm-244		99.7	8.98E-03	2.58E-02		45	4.03E-03	1.02E-02		9.62	4.33E-06
Gamma Spectroscopy											
Cs-137		3.82E+02	3.44E-02	9.89E-02	<	48.6	4.35E-03	1.10E-02	<	30.1	1.36E-05
Estimated ##			4.09E-02								
Ba-137m		3.63E+02	3.27E-02	9.39E-02	<	46.2	4.14E-03	1.04E-02	<	28.6	1.29E-05
Co-60		3.80E+02	3.42E-02	9.83E-02	<	12.6	1.13E-03	2.84E-03	<	48.5	2.18E-05

AN-102 Radioactive Glass =ADS#717 = 0.4999 grams in 0.1 L
LRM Standard Glass= ADS#720 = 0.5028 grams in 0.1 L

Notes:

Specification limits (Reference 1): < 3 Ci/m^3 for Cs-137, < 20 Ci/m^3 for Sr-90 and < 0.1 Ci/m^3 for Tc-99; Densities used for Env. C glass (2.87 g/cm^3) and LRM glass (2.52 g/cm^3) from References 5 and 9b, respectively.

Estimated values from Table 7.

† Tc-99 ICPMS Instrument Detection Limit (IDL) =0.02 ug/L for non-diluted sample; ICPMS values for Tc-99 in ug/L converted to uCi/g in glass using the mass and volume data shown at bottom of Table 10 and using Equation 1.

Table 11. Radionuclide Analyses for Acid Dissolved Glasses

Radionuclide Analyses for Acid Dissolved Glasses

Radionuclide		AN-102 Radioactive Glass (dpm/mL)	AN-102 Radioactive Glass (uCi/g)	Ci/m$^{3\,\#}$		LRM Standard Glass (dpm/mL)	LRM Standard Glass (uCi/g)	Ci/m$^{3\,\#}$		Reagent Blank (dpm/mL)	Reagent Blank (uCi/mL)
Total Alpha		248.0	2.22E-02	6.37E-02	<	5.6	5.05E-04	1.27E-03	<	5.9	2.68E-06
Total Beta		3.38E+04	3.03E+00	8.68E+00	<	14.3	1.29E-03	3.25E-03	<	14	6.44E-06
Sr-90		1.21E+04	1.08E+00	3.11E+00	<	65.2	5.88E-03	1.48E-02	<	65.2	2.94E-05
Estimated ##			1.04								
Y-90		1.21E+04	1.08E+00	3.11E+00	<	65.2	5.88E-03	1.48E-02	<	65.2	2.94E-05
Tc-99	<	1820	1.63E-01	4.68E-01	<	3.91	3.52E-04	8.88E-04		3.92	1.77E-06
Tc-99(ICP-MS) †		4.9 ug/L	1.65E-02	4.74E-02	<	0.43	1.46E-03	3.68E-03	<	0.42	7.13E-06
Estimated ##			3.66E-02								
Pu-238	<	7.16	6.41E-04	1.84E-03	<	0.849	7.65E-05	1.93E-04	<	0.07	3.15E-08
Pu-239/240	<	3.57	3.20E-04	9.17E-04	<	0.351	3.16E-05	7.97E-05	<	0.0183	8.24E-09
Pu-241	<		2.90E-03	8.32E-03	<		1.72E-03	4.33E-03	<		5.78E-06
Am-241		136	1.22E-02	3.49E-02	<	19.8	1.78E-03	4.50E-03	<	12.1	5.45E-06
Cm-244		82.40	7.38E-03	2.12E-02	<	6.98	6.29E-04	1.59E-03	<	8.76	3.95E-06
Gamma Spectroscopy											
Cs-137		3.77E+02	3.38E-02	9.70E-02	<	45.7	4.12E-03	1.04E-02	<	51.4	2.32E-05
Estimated ##			4.09E-02								
Ba-137m		3.59E+02	3.21E-02	9.21E-02	<	43.4	3.91E-03	9.86E-03	<	48.8	2.20E-05
Co-60		3.59E+02	3.21E-02	9.23E-02	<	43.4	3.91E-03	9.86E-03	<	56.1	2.53E-05

AN-102 Radioactive Glass = ADS#709 = 0.5032 grams in 0.1 L
LRM Standard Glass = ADS#712 = 0.4997 grams in 0.1 L

Notes:

Specification limits (Reference 1): < 3 Ci/m^3 for Cs-137, < 20 Ci/m^3 for Sr-90 and < 0.1 Ci/m^3 for Tc-99; Densities used for Env. C glass (2.87 g/cm^3) and LRM glass (2.52 g/cm^3) from References 5 and 9b, respectively.

Estimated values from Table 7.

† Tc-99 ICPMS Instrument Detection Limit (IDL) =0.02 ug/L for non-diluted sample; ICPMS values for Tc-99 in ug/L converted to uCi/g in glass using the mass and volume data shown at bottom of Table 11 and using Equation 1.

Results of 'Association of Standards and Test Methods' (ASTM) Test C 1285 – 97 Leach Test 'Product Consistency Test' (PCT) on AN-102 ILAW Glass

The two tables in this section show the results of the standard ASTM C 1285 –97 test on the radioactive AN-102 glass. This test is commonly called the Product Consistency Test (PCT) and is performed at 90°C [11]. The procedure for PCT-A of the ASTM C 1285-97 was strictly followed for this test. Triplicate samples of the AN-102 glass and, as prescribed by the procedure, triplicate blanks were used. The standard glasses, Low Activity Reference Material (LRM) [9,10] and Analytical Reference Material (ARM) [14] were also leached in the test with the AN-102 glass.

In the contract, SRTC was required to subject the AN-102 glass to the PCT and report the results for B, Si, and Na for the AN-102 glass. Section 2.2.2.17.2 of Mod. No. M013 of the contract specifies that in the PCT, the glass shall have a normalized mass loss less than 2 g/m^2 (2 grams of glass per square meter of exposed surface area of glass tested in a 90°C PCT) based on each of the elements B, Si, and Na. The LRM [9,10], and the standard (ARM) glass [14] were also tested with the AN-102 glass to confirm that the test conditions for the PCT were properly controlled. Table 12 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. The raw data that is the bases of these averages are in Appendix C. The last row of the table presents the consensus results of the PCT of a round robin on the LRM glass involving six different laboratories [10]. As can be seen, the concentrations measured in this test for LRM glass were very close to the consensus concentrations.

Table 12. 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)	0.029	0.046	0.318	6.66
ARM(a)	18.3	65.5	38.7	10.2
AN-102(a)	19.4	57.1	62.9	10.6
LRM(a)	25.4	80.1	160.8	10.8
LRM(c)	26.7	82.0	159.7	11.7

(a) Based on triplicate tests.

(b) Initial pH of the leach water was 6.57

(c) Published consensus values for LRM glass. [10]

The results for the blanks indicate that contamination of the leachates from possible impurities in the water or 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 [15]. This comparison is part of the ASTM procedure. The results were between the lower and upper control limits (See Appendix C for PCT data sheet on ARM glass) indicating that all 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 (see Appendix C) 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 12 the AN-102 glass appears slightly more durable that the LRM 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. The specification for ILAW glass is that the normalized mass losses based on B, Si, and Na, shall each be <2 grams of glass per square meter of exposed surface area of glass tested in a 90°C PCT for 7 days [1]. 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 Gaussion [11]. 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 $1.12X10^{-4}$ 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

$$NR_i = (C_i/C_{ig})/0.02X10^3$$

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 13 presents the normalized releases calculated from the PCT data and the measured composition of the AN-102 glass (see Table 8). Table 13 presents the averages and standard deviations based on triplicate tests. The normalized releases for all three elements are less than the upper limit of 2 g glass/m^2 specified in section 2.2.2.17.2 of the Section C, Statement of Work [1]. Thus the glass meets this specification. Table 13 also

shows similar normalized results for the LRM glass calculated from the PCT data and the measured composition of the LRM glass (see Table 9).

Table 13. Normalized Mass Losses (g glass/m^2) Based on B, Si, and Na, For AN-102 Glass in a 90°C PCT

	AN-102 Glass	LRM Glass
Element	**Normalized Release[a]**	**Normalized Release[a]**
B	0.298±0.004	0.520±0.005
Si	0.125±0.002	0.155±0.001
Na	0.350±0.014	0.536±0.017

(a) Based on triplicate Product Consistency Tests.

The normalized releases for the AN-102 ILAW glass in Table 13 are very close to those determined for AN-103 ILAW glass and reported earlier [12]. For that glass, the normalized releases for B, Si, and Na were 0.37 g/m^2, 0.17 g/m^2, and 0.40 g/m^2, respectively.

CONCLUSIONS

The experiments presented in the technical report support use of the technology being proposed by RPP-WTP personnel for pretreatment and immobilization of pretreated Hanford tank 241-AN-102 waste. The AN-102 active waste stream was immobilized into a durable LAW waste glass that meets the specifications set forth in Reference [1]. This demonstration was successful at producing an active AN-102 glass based on formulations provided by VSL. Resulting glass compositions were very similar to the target compositions for the two glasses (active AN-102 and standard LRM) examined.

Analyzed activities from radioactive AN-102 glass for Cs-137, Sr-90 and Tc-99 indicate these radionuclides are present below the average target values in the specification. The transuranic concentrations of the AN-102 glass are well below the contract specification limit for TRU-containing waste. X-ray diffraction and microscopy analyses of active AN-102 glass show this waste form to be amorphous with no evidence for the presence of crystals.

The ASTM standard Product Consistency Test (PCT) performed at 90°C on the AN-102 radioactive glass and the Low Activity Reference standard LRM glass showed similar measured releases for the B, Si, Na components. The PCT results indicate that normalized released for B, Si, and Na are well below the specification limit of 2 g glass/ m^2.

CONTROLS AND QUALITY ASSURANCE

QA and QC programs applied to the testing described in this technical report include SRTC procedures for control of measurement and testing equipment (M&TE), tracking of radioactive samples, control of laboratory notebooks, and routine ADS QA and QC [16-18]. The QA program applied by SRTC for preparation and analysis of the AN-102 glass sample complies with the requirements of NQA-1. Data sheets for the chemicals used in glass formulation are included in Appendix D of this report.

Analytic standards were required for all analyses performed for this study. Use of these standards is part of routine ADS QA and QC and are part of the procedures in **Manual L16.1** for the operating the analytical instrument.

All M&TE used to perform the evaporation and vitrification experiments was used within the specified calibration period. Calibrations were verified as required for each mass balance instrument. A record of the calibration was routinely maintained in the logbook designated for that piece of equipment.

All personnel who performed steps of the evaporation and vitrification testing were trained on the ITS procedure for operating the evaporation apparatus and furnace. In addition, they were trained on calibrating and operating equipment used in these steps. Training records were maintained for all personnel working on this project.

All laboratory data obtained in the tasks described in this technical report are included as permanent record in Charles L. Crawford's WSRC laboratory notebooks**: WSRC-NB-98-00196** and **WSRC-NB-99-00182**. Associated data to these two WSRC laboratory notebooks is also kept as permanent record in the three-ring binders labeled as**: LAW Envelope C, Sample AN-102, Vitrification and Product Testing, Charles L. Crawford.** Certain data related to the glass dissolution preparation and the Product Consistency Tests are maintained as permanent record in Daro M. Ferrara's WSRC laboratory notebook: **WSRC-NB-2000-00060**.

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17. Westinghouse Savannah River Company, "**WSRC L1 Savannah River Technology Center Procedures Manual**," current revision.

18. Westinghouse Savannah River Company, "**WSRC L16.1 Analytical Development Section Analytical Operating Procedures Manual**," current revision.

Appendix A. Glass Recipe for Active AN-102 Glass Product

Recipe for glass LAWC21 using High Sulfate Active sample

23-May-00

Envelope AN-102 Constituents	calculated M	Evapor. feed AN-102 active mg/L	GLASS Oxides	Conversion to wt% " Oxides"	AN102 wt% As glass	AN102 wt% in glass @ 11.8% Na2O	Glass Former Mix	**LAWC21 this target** for AN102	Additives this sample		Assay	Ratio	Target Weight (g)	other oxides present	
		5/15/00 email	**Loading**		100%	13.43%	100%	0.1343	86.57%				Additives	% Fe2O3	%SiO2
Ag			Ag2O	0.00	0.00	0.0000		0.0000							
Al		**5361**	Al2O3	3.03	5.92	0.795	6.18	6.1454	5.350	**[1]**	0.990	0.540	**131.37**	1.16%	43.70%
B		**13.4**	B2O3	0.01	0.03	0.0034	11.70	10.1323	10.129	**[2]**	0.986	0.563	**239.52**		
Ba		**0**	BaO	0.00	0.00	0.0000		0.0000							
Ca		**111**	CaO	0.05	0.09	0.0122	7.40	6.4185	6.406	**[3]**	0.993	0.475	**178.29**	0.40%	51.00%
Cd		**19.9**	CdO	0.01	0.01	0.0018		0.0018							
Co		**1.6**	CoO	0.00	0.00	0.0002		0.0002							
Cr		**70.3**	Cr2O3	0.06	0.12	0.0161		0.0161							
Cu		**3.6**	CuO	0.00	0.00	0.0004		0.0004							
Fe		**1.9**	Fe2O3	0.00	0.00	0.0002	7.50	6.4931	6.493	**[4]**	0.998	1.000	**79.89**		
K		**951**	K2O	0.34	0.67	0.090		0.0899							
La		**1**	La2O3	0.00	0.00	0.0001		0.0001							
Li		**0**	Li2O	0.00	0.00	0.0000	3.17	2.7444	2.744	**[5]**	0.99	0.404	**90.07**		
Mg		**0**	MgO	0.00	0.00	0.0000	1.75	1.5150	1.515	**[6]**	0.990	0.480	**41.85**	7.68%	42.52%
Mn		**1**	MnO2	0.00	0.00	0.0001		0.0001							
Mo		**21.5**	MoO3	0.01	0.02	0.0025		0.0025							
Na	4.9E+0	**111514**	Na2O	45.01	87.88	**11.80**		11.8000							
Ni		**124**	NiO	0.05	0.09	0.0124		0.0124							
Pb		**44.4**	PbO	0.01	0.03	0.0038		0.0038							
Sn		**11**	SnO2	0.00	0.01	0.0011		0.0011							

Env. AN-102 Recipe, Continued															
Si		**34**	SiO2	0.02	0.04	0.0057	54.00	46.7547	46.749	**[7]**	0.997	1.000	**430.23**		
Sr		**126**	SrO	0.04	0.09	0.0117		0.0117							
Ti		**0**	TiO2	0.00	0.00	0.0000	1.30	1.1255	1.125	**[8]**	0.954	1.000	**15.49**	0.71%	0.91%
W		**0**	WO3	0.00	0.00	0.0000		0.0000							
Zn		**1.3**	ZnO	0.00	0.00	0.0001	3.50	3.0302	3.030	**[9]**	0.997	1.000	**39.90**		
Zr		**0**	ZrO2	0.00	0.00	0.0000	3.50	3.0301	3.030	**[10]**	0.990	0.651	**61.72**		33.00%
Br		**0**	Br	0.00	0.00	0.0000		0.0000							
Cl		**1575**	Cl	0.47	0.92	0.1236		0.1236							
F		**766**	F	0.23	0.45	0.0601		0.0601							
PO4		**2196**	P2O5	0.49	0.96	0.1288		0.1288		**Total Sodium Moles**			**5.00 moles**		
SO4	57.0E-3	**5475**	SO3	1.37	2.67	0.3582		0.3582		**Expected Glass yield**			**1313 g**		
NO2	0.79	**36218**	NO2	10.84						**Sum of Additives (g)**			**1308 g**		
NO3	1.39	**85942**	NO3	25.73						**Sugar as added reductant (decreased for TOC)**			**20 g**		
OH			OH	0.00											
CO3		**39700**	CO3	11.89											
oxalate		**1067**	C	0.32											
formate			C	0.00											
SUM			SUM	100.00	100.00	13.43	100.00	100.00	86.572						

Notes:

[1] Kyanite (Al2SiO5) 325 Mesh	Kyanite Mining
[2] H3BO3 (Technical Granular)	US Borax
[3] Wollanstonite NYAD 325 Mesh, Calcium metaSilicate, CaSiO3	NYCO Minerals
[4] Fe2O3 (Iron III oxide, -325 Mesh)	Alfa Aesar-Johnson Matthey
[5] Li2CO3 (Chemetall Foote Co. Tech.gr.	Cyprus Foote Mineral Co.
[6] Olivine (Mg2SiO4) 325 Mesh (#180)	UNIMIN Corp.
[7] SiO2 (Sil-co-Sil 75)	US SILICA
[8] TiO2 (Rutile Airfloated)	Chemalloy
[9] ZnO (Kadox-920)	Zinc Corp. of America
[10] Zircon ZrSiO4 (Flour) Mesh 325	American Mineral

Appendix B. X-ray Diffraction and Scanning Electron Microscopy Data for AN-102 Glass

The X-ray diffraction pattern of the radioactive AN-102 glass is presented in Figure B1. No peaks were observed for any crystalline materials indicating that these amorphous glass powders contain no crystalline material above the nominal 0.5 vol% detection limit of the X-ray diffraction technique. A few small peaks in the XRD spectrum are attributed to the Cr/Fe/Ni oxides. This identification is likely due to trace steel particles in the glass powders that were prepared from grinding the AN-102 active glass in a tungsten blade grinder with steel compartment.

Figure B1. XRD Pattern from Analysis of AN-102 Active Glass

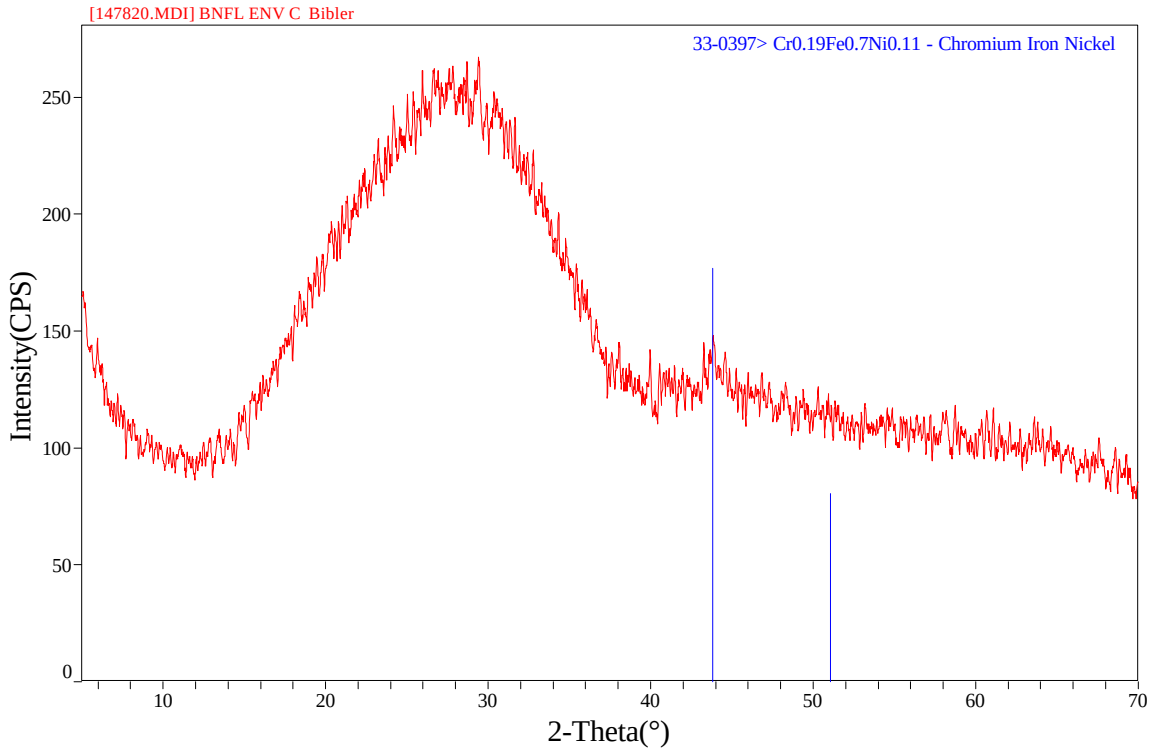


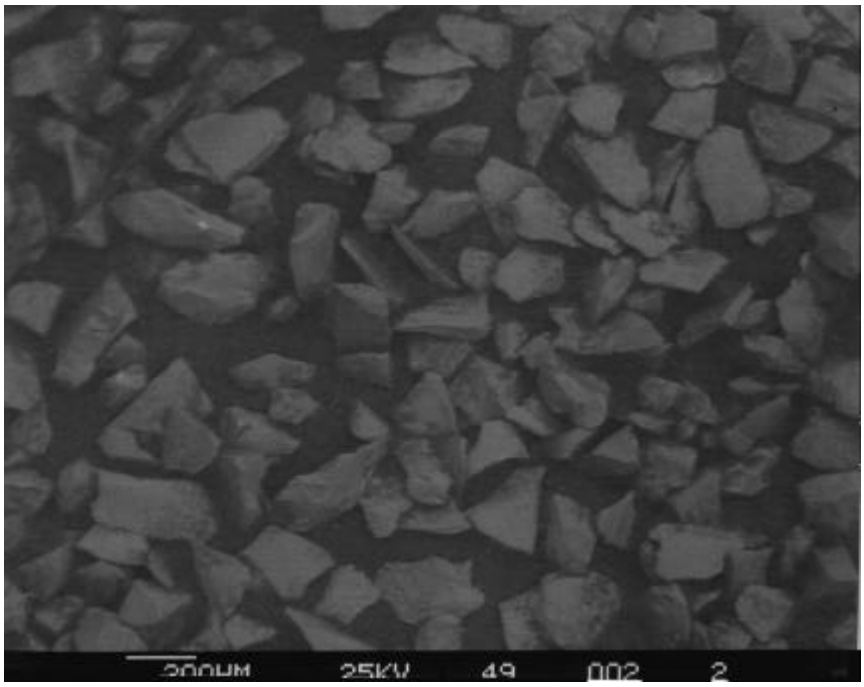
Table B1 contains summary information on the SEM microscopic images of the powdered glass samples derived from grinding the AN-102 radioactive glass. These glass powders were obtained during preparation of the glass for PCT durability tests using a Techmar tungsten blade grinder with stainless steel grinding compartment. Images obtained from secondary electron and backscattered electron microscopy were obtained at magnifications of 50X and 250X. 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.

Energy dispersive X-ray analyses were obtained for the matrix particles shown in Figure B7 (SEM Image #4). The EDAX pattern shown in Figure B5 shows this material to be comprised of the elemental components of the glass matrix, including Na, Mg, Al, Si, Zr, K, Ca, Ti, Fe and Zn. SEM image in Figure B6 shows images of the bulk glass matrix particles and relatively smaller particles with apparent different densities than the bulk matrix. These lighter shaded particles were examined with EDAX to produce the pattern shown in Figure B8. This EDAX pattern indicates presence of Cr and Ni that is indicative of trace stainless steel particles derived from grinding of the glass. These apparent steel trace contaminants in these analyzed powdered glasses do not interfere with the PCT durability testing since the PCT is conducted in stainless steel containers. Also, is should be noted that separate powdered glass samples obtained from agate ball/mill grinding were analyzed for glass characterization.

Table B1. Summary Information on Microscopy Data

Figure	SEM Image	Technique	Magnification	EDAX
B2	2	BSE	50-X	
B3	1	SE	50-X	
B4	3	SE	250-X	
B5	4	BSE	250-X	
B6	7	BSE	250-X	
B7		BSE	--	See Fig. B5
B8		BSE	--	See Fig. B6

Figure B2 showing SEM image 002



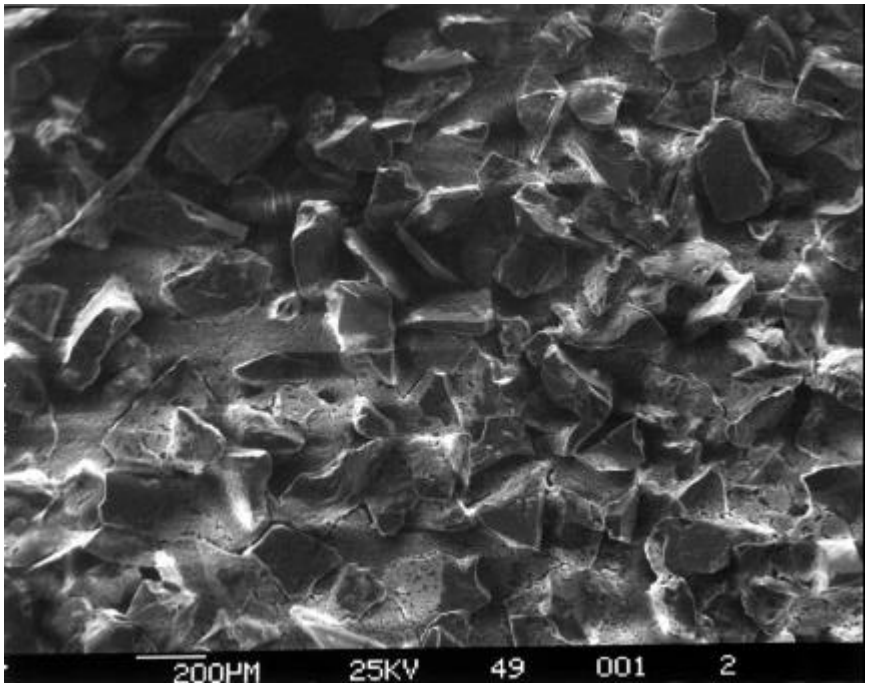
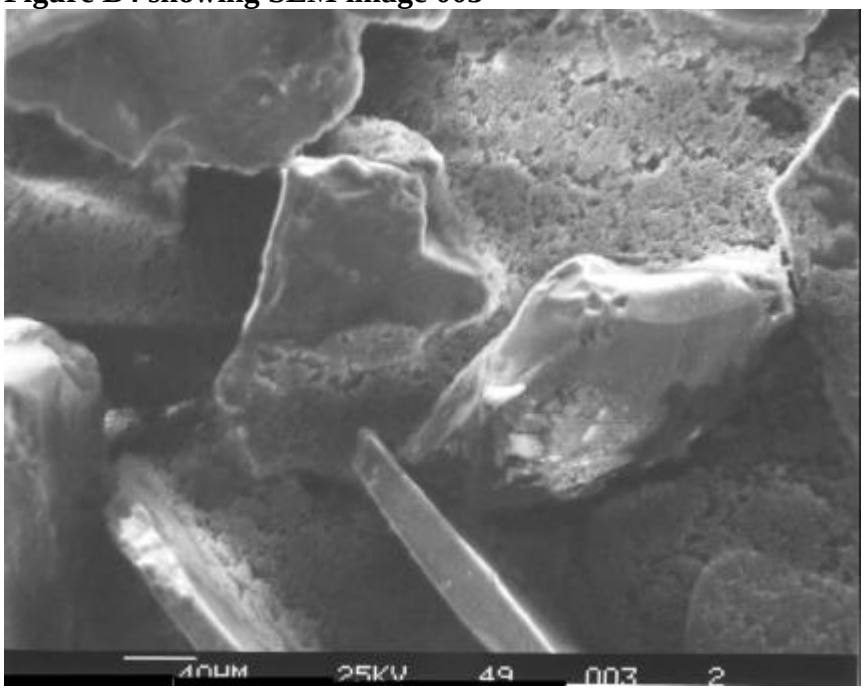


Figure B3 showing SEM image 001.

Figure B4 showing SEM image 003



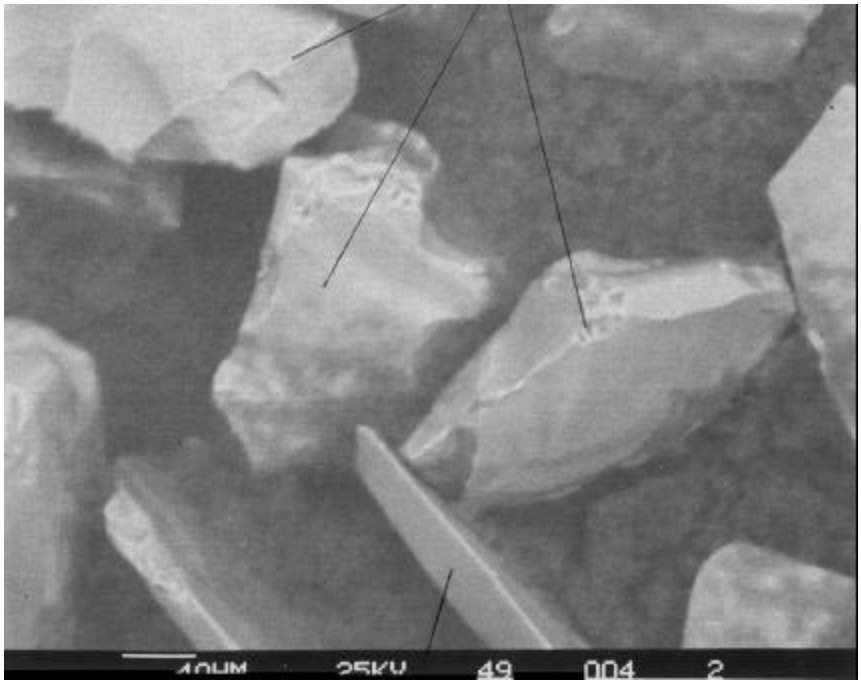
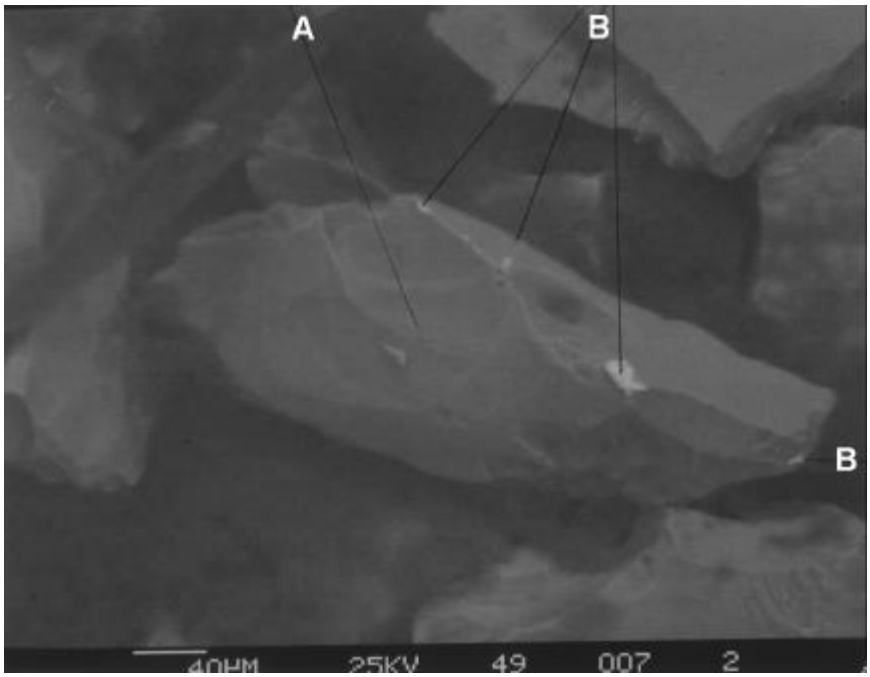


Figure B5 showing SEM image 004.

Figure B6 showing SEM image 007



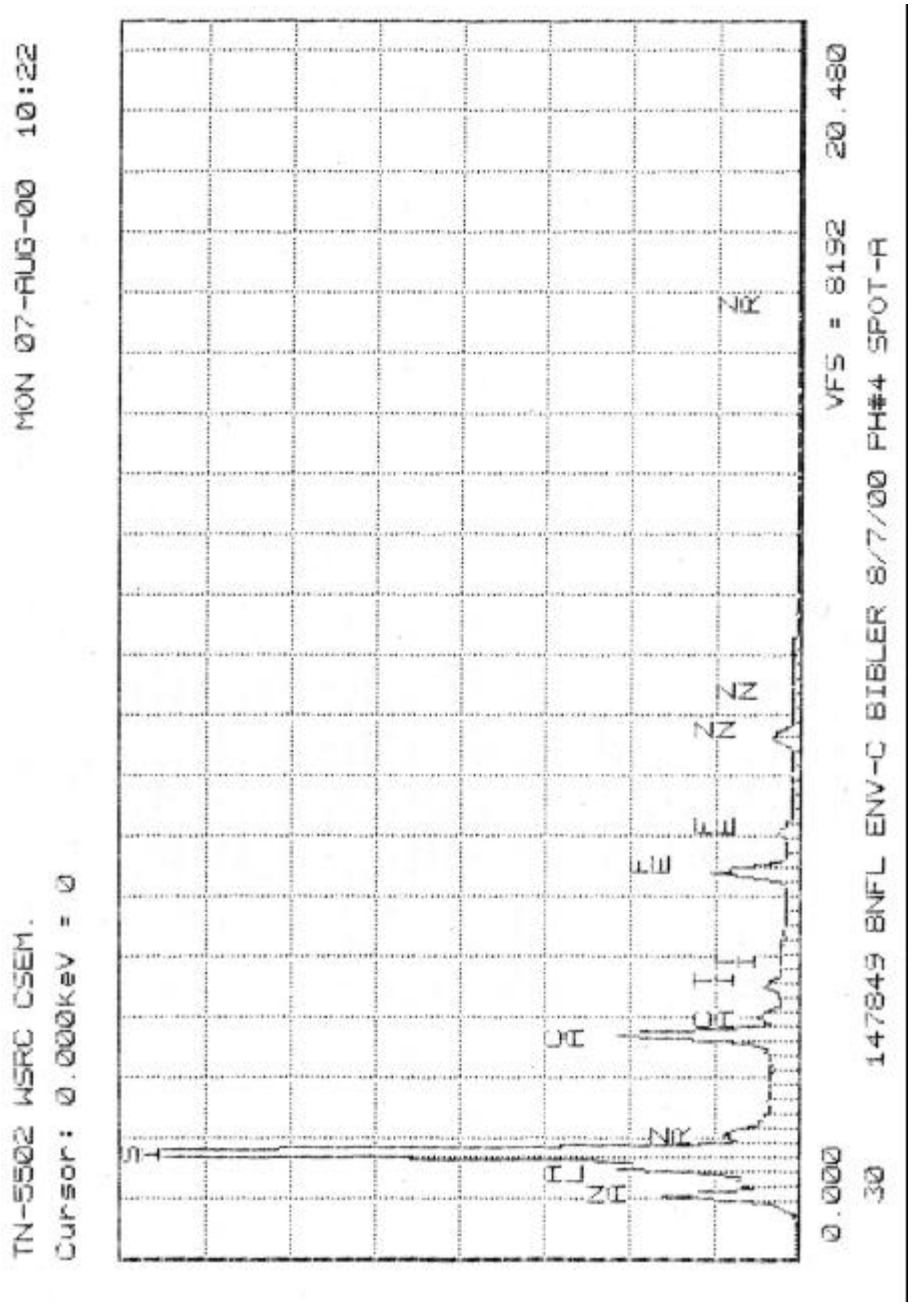


Figure B7: EDAX Pattern from Particles Shown in SEM Image 004 (Figure B5).

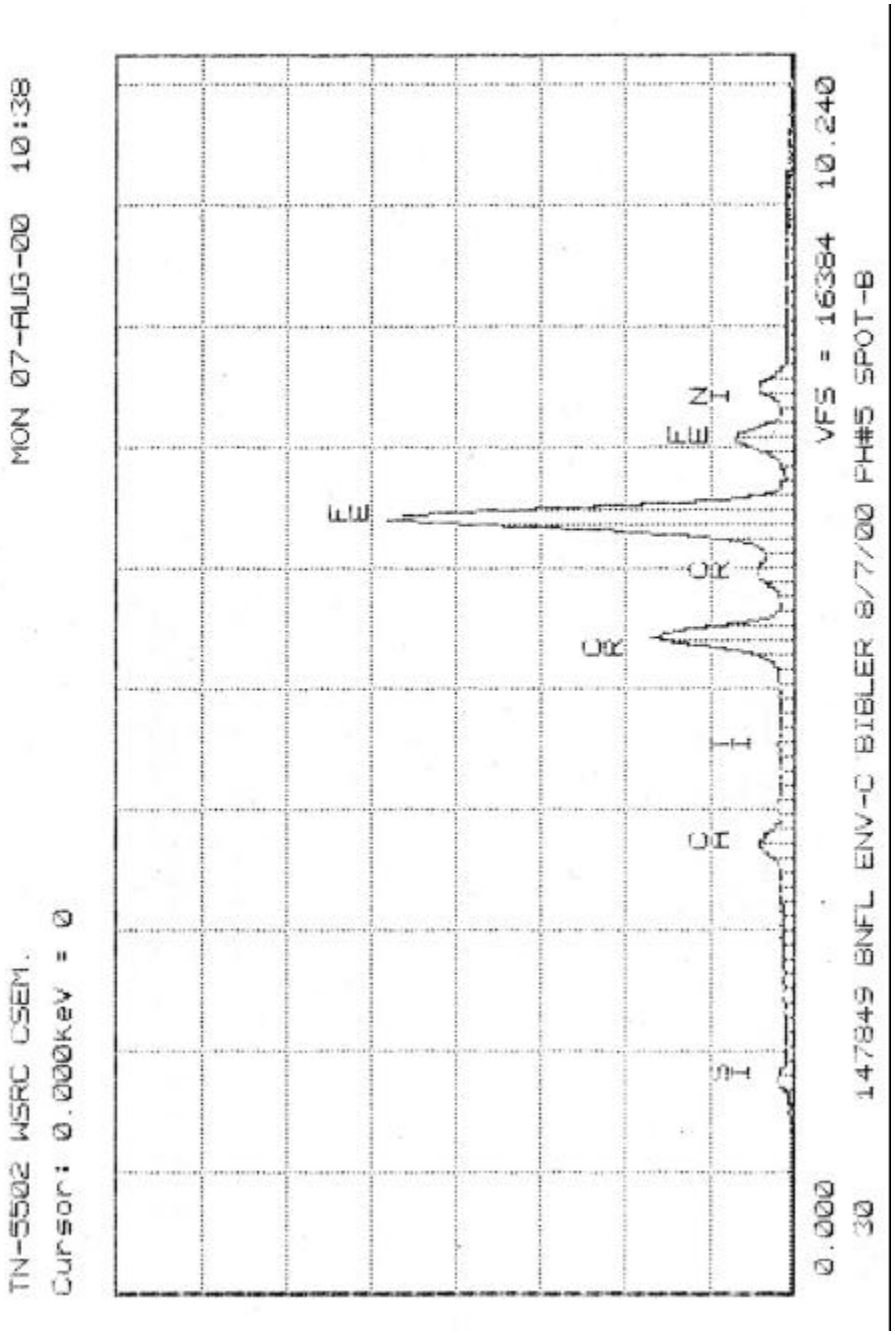


Figure B8. EDAX Pattern from Particles Indicated by 'B', Shown in SEM Image 007 (Figure B6).

Appendix C, Page C-1

Product Consistency Test (PCT) Data

AN-102 PCT SPREADSHEETS - AN 102 GLASS

APPENDIX C, PAGE C-2, WSRC-TR-2000-00371, SRT-RPP-2000-00022, Rev. 0, Formerly BNF-003-98-0271, RPP-WTP Doc. TRPT-W375-00-00028

PROCEDURE ASTM-1285-97 **PROCEDURE CALLS FOR 0.02 M2 PER GRAM OF GLASS AND 10 ML ASTM WATER PER GRAM OF GLASS**

DATA AND RESULTS FOR 7 DAY PCT TEST

TEST NAME 90C PCT WITH BNFL AN 102 GLASS
GLASS. LOW LEVEL RADIOACTIVE AN102 GLASS

DATE IN OVEN

DATE IN OVEN 7/26/00 TIME IN OVEN 10:19
DATE 0UT OF OVEN 8/2/00 TIME OUT OF OVEN 10:19

LEACHATE DILUTION FACTORS:

BLANKS: 10 ML SPL AND 0.10ML CONC HNO3 DF =10.1/10 = 1.01
ARM AND STANDARDS : 6ML SPL/0.1 ML CONC HNO3/4 ML ASTM H2O DF=1.683
ENV C AND LRM: 3ML SPL/7ML HOH/0.1ML CONC HNO3 DF=3.367

INITIAL pH = 6.57

RAW EXPERIMENTAL DATA:

SAMPLE NAME	EMPTY	WEIGHTS W/GLASS	GLASS	WEIGHT W/H2O	INITIAL WEIGHT IN PCT	FINAL WEIGHT IN PCT	INIT. VOL.(ML)	WATER LOSS	FINAL pH	SRTC ANALYTICAL NUMBER	RESULTS (ppm) FOR ACIDIFIED LEACHATES. B	Si	Na	Li	Al
P452 Blank-C-1	119.499	N.A.	N.A.	136.027	337.558	337.569	16.000	-0.011	6.66	3-147788	0.031	0.059	0.602	0.016	<.016
P453 Blank -C-2	119.742	N.A.	N.A.	136.259	338.582	338.596	16.000	-0.014	6.94	3-147793	0.033	0.037	0.198	0.011	<.016
P454 Blank -C-3	120.948	N.A.	N.A.	137.478	338.984	338.982	16.000	0.002	6.39	3-147798	0.023	0.043	0.155	0.008	<.016
BLANK AVERAGE											0.029	0.046	0.318	0.012	
SAMPLES															
P431 BNFL -AN 102-1	119.734	N.A.	N.A.	338.57	338.565	338.569	NA	-0.004	10.5	3-147789	5.763	17.063	19.356	2.777	0.690
P432 BNFL-AN 102-2	120.566	N.A.	N.A.	339.481	339.474	339.477	NA	-0.003	10.6	3-147794	5.838	17.098	18.967	2.790	0.644
P433 BNFL-AN 102-3	119.975	N.A.	N.A.	338.855	338.847	338.852	NA	-0.005	10.6	3-147799	5.674	16.718	17.935	2.596	0.662

CALCULATED RESULTS: WATER LOSS, pH VALUES, AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS

SAMPLE NAME	GLASS WEIGHT	INIT. VOL.(ML)	FINAL VOL.(ML)	% LOSS	pH VALUES INITIAL	pH VALUES FINAL	CONCENTRATIONS (PPM) B	Si	Na	Li	Al
P431 BNFL -AN 102-1	1.686	16.860	16.871	-0.024	6.6	10.5	19.4	57.4	65.0	9.3	2.3
P432 BNFL-AN 102-2	1.639	16.390	16.404	-0.018	6.6	10.6	19.6	57.5	63.7	9.4	2.2
P433 BNFL-AN 102-3	1.632	16.320	16.318	-0.031	6.6	10.6	19.1	56.3	60.2	8.7	2.2
AVERAGE							**19.4**	**57.1**	**62.9**	**9.2**	**2.2**
STANDARD DEVIATION							**0.3**	**0.7**	**2.5**	**0.4**	**0.1**
REL. STD. DEVIATION (%)							**1.4**	**1.2**	**3.9**	**4.0**	**3.5**
NORMALIZED CALCULATIONS:		ELEMENTAL WEIGHT PERCENT IN GLASS					3.248	22.76	8.982	1.282	**3.268**
		NORMALIZED MASS LOSS (GRAMS GLASS / METER SQUARED)					**0.298**	**0.125**	**0.350**	**0.357**	**0.034**
		NORMALIZED MASS LOSS STANDARD DEVIATION					**0.004**	**0.002**	**0.014**	**0.014**	**0.001**
		NORMALIZED MASS LOSS PLUS 2 SIGMA					**0.30**	**0.13**	**0.36**	**0.36**	**0.04**

QUALITY ASSURANCE INFORMATION:

LAB#=B-111
PH METER SER.#=81201792 BUFFERED TO PH 7AND 10.
BALANCE SER.#=1119180391
OVEN SER.#OV3-30228
FILTER SIZE:.45 MICRON

STANDARD RESULTS:
EXPIRATION DATE 2/28/01
LOT NO. 2517

	LIMS NO.	ICP RESULTS (PPM) B	Si	Na	Li	Al
S-1	3-147787	11.70	29.77	49.85	6.09	2.25
S-2	3-147792	11.88	30.11	50.39	6.21	2.29
S-3	3-147797	11.51	29.25	47.17	5.76	2.19
S-4	3-147802	11.57	29.33	47.20	5.76	2.21
RESULTS CORRECTED FOR DILUTION FACTOR OF 1.68						
S-1		**19.69**	**50.11**	**83.90**	**10.25**	**3.79**
S-2		**19.99**	**50.67**	**84.81**	**10.45**	**3.85**
S-3		**19.37**	**49.22**	**79.38**	**9.70**	**3.68**
S-4		**19.48**	**49.36**	**79.44**	**9.69**	**3.72**
STANDARD COMPOSITION (PPM)		**20.00**	**50.00**	**81.00**	**10.00**	**4.00**

RESEARCHER: NED BIBLER

AN-102 PCT SPREADSHEETS - LRM GLASS

APPENDIX C, PAGE C-3, WSRC-TR-2000-00371, SRT-RPP-2000-00022, Rev. 0, Formerly BNF-003-98-0271, RPP-WTP Doc. TRPT-W375-00-00028
PROCEDURE ASTM-1285-97
DATA AND RESULTS FOR 7 DAY PCT TEST

TEST NAME 90C PCT WITH BNFL ENVELOPE C GLASS
GLASS. LRM GLASS

DATE IN OVEN

DATE IN OVEN 7/26/00 TIME IN OVEN 10:19
DATE 0UT OF OVEN 8/2/00 TIME OUT OF OVEN 10:19

LEACHATE DILUTION FACTORS:
BLANKS: 10 ML SPL AND 0.10ML CONC HNO3 DF =10.1/10 = 1.01
ARM AND STANDARDS : 6ML SPL/0.1 ML CONC HNO3/4 ML ASTM H2O DF=1.683
ENV C AND LRM: 3ML SPL/7ML HOH/0.1ML CONC HNO3 DF = 3.367

INITIAL pH = 6.57

RAW EXPERIMENTAL DATA:

SAMPLE NAME	EMPTY	WEIGHTS W/GLASS	GLASS	WEIGHT W/H2O	INITIAL WEIGHT IN PCT	FINAL WEIGHT IN PCT	INIT. VOL.(ML)	WATER LOSS	FINAL pH	SRTC ANALYTICAL NUMBER	RESULTS (ppm) FOR ACIDIFIED LEACHATES. B	Si	Na	Li	Al
P452 Blank-C-1	119.499	N.A.	N.A.	136.027	337.558	337.569	16.000	-0.011	6.66	3-147788	0.031	0.059	0.602	0.016	<.016
P453 Blank -C-2	119.742	N.A.	N.A.	136.259	338.582	338.596	16.000	-0.014	6.94	3-147793	0.033	0.037	0.198	0.011	<.016
P454 Blank -C-3	120.948	N.A.	N.A.	137.478	338.984	338.982	16.000	0.002	6.39	3-147798	0.023	0.043	0.155	0.008	<.016
BLANK AVERAGE											0.029	0.046	0.318	0.012	
SAMPLES															
P434 LRM -C-1	121.066	122.721	1.655	139.273	340.717	340.721	16.552	-0.004	10.76	3-147790	7.575	23.783	49.484	0.028	4.484
P435 LRM-C-2	120.277	121.929	1.656	138.498	340.467	340.473	16.569	-0.006	10.80	3-147795	7.452	23.576	46.628	0.027	4.410
P436 LRM-C-3	120.614	122.269	1.658	138.856	340.431	340.439	16.587	-0.008	10.86	3-147800	7.565	24.027	47.151	0.025	4.502
ANALYTICAL DETECTION LIMITS															

CALCULATED RESULTS: WATER LOSS, pH VALUES, AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS

SAMPLE NAME	GLASS WEIGHT	INIT. VOL.(ML)	FINAL VOL.(ML)	% LOSS	pH VALUES INITIAL	pH VALUES FINAL	CONCENTRATIONS (PPM) B	Si	Na	Li	Al
P434 LRM -C-1	**1.655**	16.552	16.556	-0.024	**6.97**	10.76	25.51	80.08	166.61	0.09	15.10
P435 LRM-C-2	**1.652**	16.569	16.575	-0.036	**6.97**	10.80	25.09	79.38	157.00	0.09	14.85
P436 LRM-C-3	**1.655**	16.587	16.595	-0.048	**6.97**	10.86	25.47	80.90	158.76	0.08	15.16
AVERAGE							**25.36**	**80.12**	**160.79**	**0.09**	**15.03**
STANDARD DEVIATION							**0.23**	**0.76**	**5.12**	**0.01**	**0.16**
REL. STD. DEVIATION (%)							**0.91**	**0.95**	**3.18**	**5.73**	**1.09**
NORMALIZED CALCULATIONS:	ELEMENTAL WEIGHT PERCENT IN GLASS						2.44	25.82	14.99	0.047	**5.13**
	NORMALIZED MASS LOSS						**0.520**	**0.155**	**0.536**	**0.096**	**0.147**
	NORMALIZED MASS LOSS STANDARD DEVIATION						**0.005**	**0.001**	**0.017**	**0.005**	**0.002**
	NORMALIZED MASS LOSS PLUS 2 SIGMA						**0.52**	**0.16**	**0.54**	**0.11**	**0.15**

QUALITY ASSURANCE INFORMATION:

LAB#=B-111
PH METER SER.#=81201792 BUFFERED TO PH 7AND 10.
BALANCE SER.#=1119180391
OVEN SER.#OV3-30228
FILTER SIZE:.45 MICRON

STANDARD RESULTS:
EXPIRATION DATE 2/28/01
LOT NO. 2517

	LIMS NO.	B	Si	Na	Li	Al
		ICP RESULTS (PPM)				
S-1	3-147787	11.70	29.77	49.85	6.09	2.25
S-2	3-147792	11.88	30.11	50.39	6.21	2.29
S-3	3-147797	11.51	29.25	47.17	5.76	2.19
S-4	3-147802	11.57	29.33	47.20	5.76	2.21
RESULTS CORRECTED FOR DILUTION FACTOR OF 1.683						
S-1		**19.69**	**50.11**	**83.90**	**10.25**	**3.79**
S-2		**19.99**	**50.67**	**84.81**	**10.45**	**3.85**
S-3		**19.37**	**49.22**	**79.38**	**9.70**	**3.68**
S-4		**19.48**	**49.36**	**79.44**	**9.69**	**3.72**
STANDARD COMPOSITION (PPM)		**20.00**	**50.00**	**81.00**	**10.00**	**4.00**

RESEARCHER: NED BIBLER

AN-102 PCT SPREADSHEETS - ARM GLASS

APPENDIX C, PAGE C-4, WSRC-TR-2000-00371, SRT-RPP-2000-00022, Rev. 0, Formerly BNF-003-98-0271, RPP-WTP Doc. TRPT-W375-00-00028
PROCEDURE ASTM-1285-97
DATA AND RESULTS FOR 7 DAY PCT TEST

TEST NAME 90C PCT WITH BNFL ENVELOPE C GLASS
GLASS. ARM GLASS

DATE IN OVEN

DATE IN OVEN 7/26/00 TIME IN OVEN 10:19
DATE 0UT OF OVEN 8/2/00 TIME OUT OF OVEN 10:19

INITIAL pH = 6.57

LEACHATE DILUTION FACTORS:
BLANKS: 10 ML SPL AND 0.10ML CONC HNO3 DF =10.1/10 = 1.01
ARM AND STANDARDS : 6ML SPL/0.1 ML CONC HNO3/4 ML ASTM H2O DF=1.683
ENV A AND LRM: 3ML SPL/7ML HOH/0.1ML CONC HNO3 DF = 3.367

RAW EXPERIMENTAL DATA:

SAMPLE NAME	EMPTY	WEIGHTS W/GLASS	GLASS	WEIGHT W/H2O	INITIAL WEIGHT IN PCT	FINAL WEIGHT IN PCT	INIT. VOL.(ML)	WATER LOSS	FINAL pH	SRTC ANALYTICAL NUMBER	RESULTS (ppm) FOR ACIDIFIED LEACHATES. B	Si	Na	Li	Al
P452 Blank-C-1	119.499	N.A.	N.A.	136.027	337.558	337.569	16.000	-0.011	6.66	3-147788	0.031	0.059	0.602	0.016	<.016
P453 Blank -C-2	119.742	N.A.	N.A.	136.259	338.582	338.596	16.000	-0.014	6.94	3-147793	0.033	0.037	0.198	0.011	<.016
P454 Blank -C-3	120.948	N.A.	N.A.	137.478	338.984	338.982	16.000	0.002	6.39	3-147798	0.023	0.043	0.155	0.008	<.016
BLANK AVERAGE											0.029	0.046	0.318	0.012	
SAMPLES															
P437 ARM-C-1	119.378	121.036	1.659	137.631	338.799	338.807	16.595	-0.008	10.24	3-147791	10.836	39.036	23.834	9.098	3.091
P438 ARM -C-2	119.748	121.402	1.658	137.989	339.486	339.490	16.587	-0.004	10.23	3-147796	11.106	39.002	23.087	8.707	2.936
P439 ARM -C-3	120.103	121.757	1.659	138.364	339.524	339.532	16.607	-0.008	10.26	3-147801	10.768	38.779	22.612	8.540	3.001

CONTROL CHART VALUES FOR CONCENTRATIONS IN ARM GLASS PCT

	B	Si	Na	Li
HIGH	22.7	73.4	43.6	16.3
LOW	12.9	49.0	28.9	10.8

CALCULATED RESULTS: pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS

SAMPLE NAME	GLASS WEIGHT	INIT. VOL.(ML)	FINAL VOL.(ML)	% LOSS	pH VALUES INITIAL	pH VALUES FINAL
P437 ARM-C-1	**1.658**	16.595	16.603	-0.0663	**6.57**	10.24
P438 ARM -C-2	**1.654**	16.587	16.591	-0.0844	**6.57**	10.23
P439 ARM -C-3	**1.654**	16.607	16.615	0.0120	**6.57**	10.26

CONCENTRATIONS (PPM)

	B	Si	Na	Li	Al
P437 ARM-C-1	18.2	65.7	39.8	15.3	5.2
P438 ARM -C-2	18.7	65.6	38.5	14.6	4.9
P439 ARM -C-3	18.1	65.2	37.7	14.4	5.1
AVERAGE	**18.3**	**65.5**	**38.7**	**14.8**	**5.1**
STANDARD DEVIATION	**0.30**	**0.23**	**1.04**	**0.48**	**0.13**
REL. STD. DEVIATION (%)	**1.64**	**0.36**	**2.68**	**3.26**	**2.59**

NORMALIZED CALCULATIONS:

ELEMENTAL WEIGHT PERCENT IN GLASS

NORMALIZED MASS LOSS (GRAMS GLASS/LITER)	**NOT APPLICABLE**
NORMALIZED MASS LOSS STANDARD DEVIATION	**NOT APPLICABLE**
NORMALIZED MASS LOSS PLUS 2 SIGMA	**NOT APPLICABLE**

QUALITY ASSURANCE INFORMATION:

LAB#=B-111
PH METER SER.#=81201792 BUFFERED TO PH 7AND 10.
BALANCE SER.#=1119180391
OVEN SER.#OV3-30228
FILTER SIZE:.45 MICRON

STANDARD RESULTS:
EXPIRATION DATE 2/28/01
LOT NO. 2517

	ADS. NO.	ICP RESULTS (PPM) B	Si	Na	Li	Al
S-1	3-147787	11.70	29.77	49.85	6.09	2.25
S-2	3-147792	11.88	30.11	50.39	6.21	2.29
S-3	3-147797	11.51	29.25	47.17	5.76	2.19
S-4	3-147802	11.57	29.33	47.20	5.76	2.21

RESULTS CORRECTED FOR DILUTION FACTOR OF 1.68

	B	Si	Na	Li	Al
S-1	**19.69**	**50.11**	**83.90**	**10.25**	**3.79**
S-2	**19.99**	**50.67**	**84.81**	**10.45**	**3.85**
S-3	**19.37**	**49.22**	**79.38**	**9.70**	**3.68**
S-4	**19.48**	**49.36**	**79.44**	**9.69**	**3.72**
STANDARD COMPOSITION (PPM)	**20.00**	**50.00**	**81.00**	**10.00**	**4.00**

RESEARCHER: NED BIBLER

Appendix D

Data Sheets for Glass Forming Minerals

COMPLETE CHEMICAL ANALYSIS ON KYANITE CONCENTRATES

	Kyanite (Raw)	Mullite (Calcined)
Ignition loss	.21	- -
Alumina	54.00 - 60.06	54.17 - 60.06
Silica	43.70 - 37.64	43.73 - 37.84
Iron Oxide	.40 - 1.16	.36 - .90
Titania	.52 - 1.65	.52 - .98
Lime	.03	.03
Magnesia	.01	.01
Alkalies	.42	.42
	99.98	99.97

Composition - Raw Kyanite $3AL_2O_3$-$3SiO_2$
Calcined Kyanite (Mullite) $3AL_2O_3$-$2SiO_2$

Physical Properties - a. Streak - uncolored
b. Hardness - 4 to 7 Mohr's Scale
c. Specific Gravity Kyanite 3.5 to 3.7
Mullite 2.9 to 3.1
d. Lustre - vitreous to pearly
e. Color - dark grey to sandy
f. Particle shape - bladed (elongated)

Pyrometric Cone Equivalent - Cone 36 to 37
The Calcined Kyanite (Mullite) has been completely converted at a temperature of 3000 deg. F by Kyanite Mining Corp.

Mines - Dillwyn, Virginia (East Ridge Mountain)
Dillwyn, Virginia (Willis Mountain)
Kyanite Grinding Plants - East Ridge
Gieseke
Pamplin
Mullite Production & Grinding Plants - Dillwyn
Cullen
East Ridge

TYPICAL SCREEN ANALYSES ON VIRGINIA KYANITE & MULLITE

KYANITE PRODUCTS

35 Mesh %		48 Mesh %		100 Mesh %		200 Mesh %		325 Mesh %	
On	35 - 9.2	On	48 - 10.0	On	100 - 6.9	On	200 - 9.8	On	325 - 10.1
	48 - 21.2		100 - 29.9		150 - 11.1		325 - 19.6		-325 - 89.9
	100 - 41.4		150 - 13.5		200 - 17.6		-325 - 70.6		
	150 - 16.8		200 - 9.3		325 - 23.9				
	200 - 8.7		325 - 10.7		-325 - 39.5				
	325 - 2.5		-325 - 26.6						
	-325 - .1								
Total	99.9		100.0		99.0		100.0		100.0

MULLITE (Calcined Kyanite) PRODUCTS

35 Mesh %		48 Mesh %		100 Mesh %		200 Mesh %		325 Mesh %	
On	35 - 16.9	On	48 - 4.9	On	100 - 5.8	On	200 - 9.0	On	325 - 9.8
	48 - 21.6		100 - 16.1		150 - 11.6		325 - 18.1		-325 - 90.1
	100 - 38.1		150 - 11.6		200 - 16.7		-325 - 72.0		
	150 - 13.2		200 - 10.0		325 - 27.1				
	200 - 6.9		325 - 18.7		-325 - 38.7				
	325 - 2.6		-325 - 36.7						
	-325 - .6								
Total	99.9		98.0		99.9		99.1		99.9

20 MULE TEAM®

Product Specification
B-0310-U

Boric Acid

Technical Granular

May 1, 1998

Orthoboric Acid
H_3BO_3
CAS No. 10043-35-3

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 %	56.25-56.80
Equivalent H_3BO_3 %	99.9-100.9
SO_4 ppm	≤350
Cl ppm	≤18
Fe ppm	≤6

56.52

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.

BORAX

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

Wolastonite = Calcium Metasilicate, $CaSiO_3$

Wollastonite

Key Benefits and Properties

Production Benefits of Using Wollastonite in Sanitaryware*

- Shrinkage maintained to comply with mold standards
- Reduced vitrification temperature
- Increased impact resistance
- Increased drying temperature
- Reduced drying time
- Reduced HF emissions
- Improved fired strength

*wollastonite with a very high aspect ratio

Typical Physical and Chemical Properties of Wollastonite

Appearance	White
Particle Shape	Acicular
Molecular Weight	116
Specific Gravity	2.9
Refractive Index	1.63
pH (10% slurry)	9.9
Water Solubility (g/100cc)	0.0095
Density (lbs./solid gallons)	24.2
Mohs Hardness	4.5
Coefficient of Expansion (mm/mm/°C)	6.5×10^{-6}
Melting Point (°C)	1540

Chemical Composition

CaO	47.5
SiO_2	51.0
Fe_2O_3	0.4
Al_2O_3	0.2
MnO	0.1
MgO	0.1
TiO_2	0.02
L.O.I. (1000°C)	0.68

MATERIAL SAFETY DATA SHEET

Alfa Aesar (A Johnson Matthey Company)
Johnson Matthey Catalog Company, Inc.
30 Bond Street
Ward Hill, MA 01835-0747
Emergency Phone-(978) 521-6300
CHEMTREC-(800) 424-9300
Web Site: www.alfa.com

SECTION 1-IDENTIFICATION

Product Code: 12375 **Revision Date:** 5/14/96
Product Name: Iron (III) oxide
Synonyms: Ferric oxide
Red iron oxide
Iron red
Iron sesquioxide
Chemical Family: Metal oxide
CAS#: 1309-37-1
Molecular Formula: Fe_2O_3

SECTION 2-INGREDIENTS

Chemical: Iron (III) oxide

CAS#	%	PEL	TLV
1309-37-1	100	10mg Fe/m^3-fume	5mg Fe/m^3-dust/fume

SECTION 3- PHYSICAL DATA

Boiling Point: Not applicable
% Volatiles: Not applicable
Solubility in Water : Insoluble
Specific Gravity (H_2O=1): 5.24
Freezing/Melting Point: 1565°C (decomposes)
Evaporation Rate (butyl acetate=1): Not applicable
Vapor Density (air=1): Not applicable
Vapor Pressure : Not applicable
Appearance and Odor: Reddish-brown powder; odorless
Other: No data

SECTION 4-FIRE AND EXPLOSION HAZARD DATA

Flash Point:(°F) Not applicable
Flammable Limits in Air, % by volume: **Lower** Not applicable
Upper Not applicable
Autoignition Temperature: No data

TECHNICAL DATA

OLIVINE Refractory Grades
HAMILTON, WA

CHEMICAL ANALYSIS

Mean Values. These Do Not Represent A Specification.

		Mean Percent by Weight
Magnesium Oxide	(MgO)	48.01
Silicon Dioxide	(SiO_2)	42.52
Iron Oxide	(Fe_2O_3)	7.68
Calcium Oxide	(CaO)	0.02
Chromium Oxide	(Cr_2O_3)	0.13
Aluminum Oxide	(Al_2O_3)	0.19
Potassium Oxide	(K_2O)	.01
Sodium Oxide	(Na_2O)	.02
Nickel Oxide	(NiO)	.37
Loss on Ignition	(LOI)	1.05

ORDERING INFORMATION

Shipping Point: HAMILTON, WA

Availability: BULK, 50 LB., 100 LB., AND BULK BAGS
TRUCK AND RAIL

UNIMIN

FOR PRODUCT INFORMATION AND CUSTOMER SERVICE: **UNIMIN CORPORATION**
U.S. and CANADA 800-243-9004 • FAX 800-243-9005
WORLDWIDE 203-966-1306 • FAX 203-972-1378

Silica Sand • Ground Silica • Feldspar • Nepheline Syenite • High Purity Quartz • Olivine • Microcrystalline Silica • Bentonite Clay • Dolomite

GRADE NUMBERS INDICATE RELATIVE VALUES ON RESULTS. THEY ARE NOT A SPECIFICATION OR WARRANTY OF PERFORMANCE.

HEALTH HAZARD WARNING: Prolonged inhalation of dust associated with the materials described in this data sheet can cause delayed lung injury. This material contains Nickel. IARC has determined Nickel compounds are carcinogenic to humans and the National Toxicological Program (NTP) has determined that Nickel may reasonably be anticipated to be a carcinogen. Avoid creating dust when handling, using or storing. Follow OSHA or other applicable governmental Safety and Health Standards. Current Material Safety Data Sheet containing safety information is available and should be consulted before usage.

Notice: While information contained herein is correct to the best of our knowledge, Unimin Corporation herein disclaims any warranties as to the accuracy of the same. Recommendations or suggestions are made without guarantee or representation as to result, since conditions of use are beyond our control. All materials are sold to Unimin Corporation standard terms and conditions of sale and on the condition that buyer shall make his own tests to determine the suitability of such product for buyer's purpose. No statement contained herein shall be construed as a recommendation to infringe any patent.

ham-ref (4/97)

Olivine/Olivine Containing

SIL-CO-SIL® GROUND SILICA FROM U.S. SILICA COMPANY

New Name	75				63			53			
Old Name	200 Mesh	200 Mesh	200 Mesh	200 Mesh				270 Mesh	270 Mesh		
Mine	Pacific, MO	Columbia, SC	Mill Creek, OK	Berkeley Springs, WV				Berkeley Springs, WV	Mill Creek, OK		
Sieve Analysis											
Cum. % + 100 Mesh	Trace	Trace	Trace	0.1				0.1	Trace		
Cum. % + 200 Mesh	1.6	1.5	1.5	0.7				0.3	0.5		
Cum. % + 325 Mesh	12.0	13.0	13.0	12.0				6.0	7.0		
Particle Size											
Median (Microns)	17.0	16.0	15.0	16.0				15.0	12.0		
Average (Microns)	6.0	6.0	6.0	6.0				5.5	5.5		
Specific Surface Area, (cm^2/g)	3800	3800	3800	3800				4200	4200		
Oil Absorption, (lbs/100 lbs)	20.5	22.0	20.0	24.0				25.0	21.0		
Hegman Grind	1	1	1	1				2½	3		
Apparent Density, (lbs/cu. ft.)											
Bulk Density, Tapped	93	94	93	94				90	89		
Bulk Density, Untapped	55	57	55	58				56	51		
Optical Properties											
Reflectance, Green Iris	88.0	86.0	88.0	85.0				85.0	89.0		
Reflectance, Blue Iris	85.0	82.0	87.0	82.5				83.0	87.0		
Reflectance, Amber Iris	89.0	87.5	90.0	88.0				89.0	90.0		
Yellowness	.047	.055	.035	.060				.060	.033		
Brightness (457 µm)	86.0	84.0	86.0	84.0				84.0	87.0		
pH	7.2	6.0	7.0	6.8				6.8	7.2		
Chemical Analysis, %											
SiO_2	99.7	99.5	99.7	99.6				99.6	99.7		
Fe_2O_3	.018	.025	.020	.025				.025	.020		
Al_2O_3	.100	.200	.090	.100				.100	.090		
TiO_2	.012	.035	.012	.020				.020	.012		
CaO	.01	<.01	.035	.015				.015	.035		
MgO	<.01	<.01	<.01	<.01				<.01	<.01		
L.O.I	.145	.150	.140	.200				.200	.140		

ASTM C-371-56
SEDIGRAPH 50% Point ASTM C-958
Fisher Subsieve ASTM B-330
Derived from Fisher avg. particle size (see above)
ASTM D-1483

ASTM D-1210
U.S. Silica
HunterLab Colorimeter ASTM E-306
Photovolt
5% Slurry
ASTM C-146-72

Chemalloy Company, Inc. P.O. Box 350 Bryn Mawr, PA 19010-0350
Tel. No. 610-527-3700 Fax No. 610-527-3878

Chemalloy

"Over a Quarter Century of Quality and Service"

January 1, 1999

Rutile
(Premium Grade - Rutile Ore)

Chemical Analysis

	Specification	Typical
TiO_2	94.0% min.	95% - 96%
SiO_2	1.5% max.	0.55%
ZrO_2*	1.5% max.	0.6%
Fe_2O_3*	1.0% max.	0.6%
V_2O_5	1.0% max.	0.5%
Cr_2O_3	0.5% max.	0.18%
S	0.02% max.	0.01%
P	0.02% max.	0.015%

*Airfloated product is 2.5% max.

Physical Description

30 x 200 Mesh	40 x 200 Mesh
100% minus 30 Mesh	100% minus 40 Mesh
15% max. minus 200 Mesh	15% max. minus 200 Mesh
60 x 200 Mesh	**Airfloated**
100% minus 50 Mesh	100% minus 80 Mesh
1% max. on 60 Mesh	90% min. minus 325 Mesh
15% max. minus 200 Mesh	

Price Schedule

PLEASE CHECK BRYN MAWR OFFICE FOR CURRENT PRICE

30, 40, 60 x 200 Mesh - - - FOB Cleveland, OH
Airfloated - - - - - - - - FOB Conshohocken, PA

Minimum Order Charge - - - $100.00 Each Shipment

Packaging

"Hex" Cardboard Boxes - 30, 40, 60 x 200 Mesh Only - 3,500 lbs. net weight - bulk
Multiwall Bags--Screened Products - 100 lbs. net weight in 3,000 lb. pallet units
Multiwall Bags-Milled (Airfloated)- 50 lbs. net weight in 3,000 lb. pallet units
Steel Drums - 1,000 lbs. net weight - $0.05 per pound Extra
Wooden Pallet Boxes-Type A - for bagged or bulk shipments - $60.00 per box Extra

Terms

Net 30 Days

PRICES SUBJECT TO CHANGE WITHOUT NOTICE

HMIS No.: 1*-0-0-E
CAS No.: 13463-67-7
Revision No.: 45
Supersedes: 9/1/98

Page 58

ZINC CORPORATION OF AMERICA
300 FRANKFORT ROAD
MONACA, PENNSYLVANIA 15061
(412) 774-1020

KADOX-920 ZINC OXIDE

DESCRIPTION

KADOX-920 is a high purity French Process zinc oxide, providing a surface area and reactivity between KADOX-911 and KADOX-930. KADOX-920 is also available in coated (KADOX-920C), pelleted (KADOX-920P), and granular (KADOX-920G) forms.

USES

RUBBER — KADOX-920 is used extensively in various rubber products, such as mechanical goods, insulated wire, footwear, and tires, where uniform activation, moderate reinforcement, and good dispersion are desired.

OTHER USES — KADOX-920 is used in the production of ceramics, rayon, resinates, textiles, zinc chromates, and phosphate solutions where a zinc oxide of high purity is required.

TECHNICAL DATA

Representative Physical Properties

Mean Particle Size (microns)	0.21
Surface Area (sq. meters/gram)	5.0
Specific Gravity	5.6
Apparent Density (lb./ft.^{3})	35.
Through 325 Mesh	99.99%
Specifications	ASTM D-79 ASTM D-4295

Representative Chemical Properties

ZnO	99.8%
PbO	.001%
CdO	.005%
CuO	<.0005%
MnO	<.0005%
Fe_2O_3	.001%
H_2O Soluble Salts	.02%

CYPRUS Foote Mineral

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

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.

QA ANALYST [signature] DATE 4/10/95

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

FOOTE LITHIUM

American Minerals, Inc.
901 E. Eighth Avenue, Suite #200
King of Prussia, Pennsylvania 19406

ZIRCON
Zirconium Silicate

ZIRCON

Zircon Sand and Flour for all applications. Large stockpiles maintained assuring consistent high quality and reliable supply of different grades and grinds for various industries.

APPLICATIONS

- Steel
- Refractory
- Foundries
- Ceramics
- Glass
- Chemical
- Abrasive

SPECIFICATION

Chemical:

Constituent -	Typical Range - Wt.%	
ZrO_2 (+HfO_2)	66.0% Typ.	65.0 Min.
Fe_2O_3	0.06 - 0.09	
TiO_2	0.07 - 0.14	
Al_2O_3	0.1 - 0.4	
SiO_2	32.0 - 32.5	
Free Silica	0.01 - 0.2	
U + Th	400 ppm.	500 ppm. Max.

Physical:

Data:

Specific Gravity	4.6-4.8
Bulk Density	170-180 ibs./ft.^{3}
L.O.I.	0.15-0.25
Melting Point	2200^{0}C
Hardness (MOHS)	7.5
Angle of repose	30^0

Sand:	Typical	'B' Grade	'C' Grade
	40	0	0
	50	0	1.0
	70	0.20	28.0
	100	8.00	55.0
	140	50.00	14.0
	200	41.00	1.5
	270	1.30	0.5
	PAN	0	0
		AFS 108-115	AFS 68-74

Flour:	Grind	Typical Wt.%	Mesh
	200	95	-200
	325	95	-325

Special Grinds Available

'B' Grade and Flour available as Calcined Zircon.

Tel. (610) 962-5050 • Fax (610) 962-5056 • Telex 881075