Pacific Northwest National Laboratory

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Waste Form Development for a DC Arc Furnace

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September 1996

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Pacific Northwest National Laboratory
Richland, Washington 99352

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Abstract

A laboratory crucible study was conducted to develop waste forms to treat nonradioactive simulated ^{238}Pu heterogeneous debris waste from Savannah River, metal waste from the Idaho National Engineering Laboratory (INEL), and nominal waste also from INEL using DC arc melting. The preliminary results showed that the different waste form compositions had vastly different responses for each processing effect. The reducing condition of DC arc melting had no significant effects on the durability of some waste forms while it decreased the waste form durability from 300 to 700% for other waste forms, which resulted in the failure of some TCLP tests. The right formulations of waste can benefit from devitrification and showed an increase in durability by 40%. Some formulations showed no devitrification effects while others decreased durability by 200%. Increased waste loading also affected waste form behavior, decreasing durability for one waste, increasing durability by 240% for another, and showing no effect for the third waste. All of these responses to the processing and composition variations were dictated by the fundamental glass chemistry and can be adjusted to achieve maximal waste loading, acceptable durability, and desired processing characteristics if each waste formulation is designed for the result according to the glass chemistry.

Executive Summary

A study to develop waste forms within the Pacific Northwest National Laboratory (PNNL) DC Arc Furnace Project was initiated to identify waste form issues related to the processing characteristics of a DC arc process, such as the reducing melting condition, devitrification of a monolith waste form, and high waste loadings. The study was based on static laboratory crucible melts using noradioactive simulants.

Fifteen glasses and vitreous ceramics have been produced with up to 100% waste loadings on three waste streams. These wastes include ^{238}Pu heterogeneous debris waste from the Savannah River, metal waste (Metal-R) from the Idaho National Engineering Laboratory (INEL), and nominal waste (Nom-60) also from INEL. Characterization of the waste was accomplished using an optical microscope, Scanning Electron Microscope, Energy Dispersive Spectroscopy, X-Ray Diffraction, chemical analysis, and redox analysis. The U.S. Environmental Protection Agency (EPA) Toxicity Characteristic Leaching Procedure and the 7-day Product Consistency Test were utilized for durability evaluation. All of the waste forms passed the hazardous element limits for regulated disposal. Two of the waste forms failed the Land Disposal Requirements.

This study showed that different waste form compositions had very different responses for each processing variation. The reducing melting condition of a DC arc melting had no significant effects on the durability of the Nom-60 based waste form, while it decreased the waste form durability from 300 to 700% for Metal-R and Pu^{238} debris wastes, which failed some TCLP tests. The devitrification of the waste forms increased durability for Metal-R waste, exhibited no effect for Pu^{238} debris waste, and substantially decreased durability for Nom-60 waste. Increased waste loading also affected waste form behavior very differently for each of the waste forms—decreasing durability for Metal-R waste, increasing durability by 240% for ^{238}Pu debris waste, and showing no effects for Nom-60 waste.

However, these different responses to the processing and composition variations are not random and are predictable based on an understanding of glass chemistry. Most of these diverse variations are associated with the types, the amount, and the ratios of the beneficial crystals such as spinel types, to the harmful crystals such as Augite-type ($\text{Ca}(\text{Fe,Mg})\text{Si}_2\text{O}_6$), Faayalite (Fe_2SiO_4), Albite-type ($\text{Na}(\text{Si}_3\text{Al})\text{O}_8$), Clinopyroxene ($\text{Ca}(\text{Ti,Mg,Al})(\text{Si,Al})_2\text{O}_6$), Wallastonite (CaSiO_3), and Anorthite-type ($((\text{Ca,Na})(\text{Al,Si})_2\text{Si}_2\text{O}_8)$). The beneficial crystals usually have two common properties: 1) they are stable and durable of themselves, and 2) they do not consume silica, alumina, and other network formers. As a result of the formation of the beneficial crystals, the residual glassy matrix in the waste form is enriched in silica and alumina and becomes more durable. The harmful crystals usually consume silica and alumina and the formation of these crystals deplete the silica and alumina from the residual glassy matrix, resulting in reduced durability of the waste form. Preliminary discussions on how each waste form composition affected the different responses to redox, devitrification, and waste loading are presented in the report.

An understanding of glass chemistry enables us to develop different formulations to achieve maximum waste loading, acceptable durability, and desired processing characteristics for treating diverse wastes and to optimize processing parameters in terms of redox control, production of monolith or gem final products. A brief discussion of each waste and associated recommendations based on the preliminary study are summarized as follows:

- In treating Metal-R wastes, the waste form durability can be reduced by 400 to 700%, the melt viscosity can also be lowered by more than 20%, and the tendency of the waste form to devitrify can be increased when it is produced in a reducing environment such as the DC arc process. The devitrified waste form is actually more durable than the quenched glass, suggesting that the waste form produced by treating Metal-R wastes with the DC arc melting process can be poured into a large container without concerns of devitrification and slow cooling. The final product passes the TCLP tests. The increased waste loading from 34 to 100% resulted in only a slight decrease in waste form durability.

The recommendation for treating Metal-R waste is at 100% waste loading in the DC arc process and pour the resulting waste form into a large container to reduce the overall waste volume. Moderate increases in oxygen concentration in the processing atmosphere during plasma processes are recommended to enhance the long-term durability of the product, but are not required to pass TCLP tests.

- In treating the Savannah River ^{238}Pu heterogeneous debris wastes, the reducing conditions during the DC arc process can decrease chemical durability 330% for glass and 400% for vitreous ceramics, lower the viscosity of the melts, and decrease the tendency for glass to devitrify. The devitrified product has similar durability as the glass because the formation of spinel crystals does not affect glassy matrix durability. However, the reduced melting condition deteriorated the waste form durability more in the case of vitreous ceramics than in the case of glass. Increasing waste loading in the case of ^{238}Pu heterogeneous wastes actually increased waste form durability under oxidizing conditions by 240% and also increased the tolerance of the waste form to reducing conditions. The lower waste loading failed the TCLP test under reducing conditions.

The recommendation for treating the ^{238}Pu heterogeneous debris waste is to process 100% waste directly without other additives. To achieve the desired chemical durability, it may be necessary either to provide a more oxidizing atmosphere (which may be difficult with the DC arc process) or to pour the final product into small size gems to reduce devitrification (which is achievable in the DC arc process).

- In treating Nom-60 waste, waste loading can be as high as 100% and it can be conveniently melted under DC arc conditions with a viscosity of less than 8 Pa.s at 1425°C. The reducing melting condition, such as that in DC arc melting, has shown insignificant effects on waste form durability in terms of both TCLP and PCT results. The reducing condition, on the other hand, can enhance melting rates by decreasing viscosity about 20%. The reducing condition is also expected to decrease the volatilities of some elements such as Tc. On the other hand, the data suggest that devitrification of the waste form has had much greater effects on glass durability, increasing sodium release by 300% and causing the waste form to fail TCLP tests for lead release. This is an important issue facing every high temperature process.

The recommendation for treating Nom-60 wastes with DC arc is to 1) treat the waste directly at 100% waste loading, and 2) avoid waste form devitrification by pouring the melted waste form into small gems to quench the melt and minimize devitrification.

1.0 Introduction

Graphite electrode arc furnaces have been used in the steel industry since the turn of the century. A refined DC arc furnace for treating waste materials has been developed by a team consisting of the Pacific Northwest National Laboratory (PNNL), Massachusetts Institute of Technology (MIT), two industrial partners T&R Associates and Electro-Pyrolysis, Inc. (EPI) [Surman-1994, -1993].

The graphite electrode DC electric arc is used to deliver thermal energy into the material to be processed. Use of the current-controlled graphite electrode DC arc provides the high-temperature environment required for melting a range of heterogeneous waste forms (temperatures in excess of 1700°C can be produced in the material to be processed). In addition, the high-temperature region around the DC arc will destroy organic species and vapors that evolve from the material being processed. The volume of off-gas produced in the pyrolytic DC arc treatment of solid waste is greatly reduced relative to combustion treatment--typically by more than a factor of 10. Since the gas is substantially reduced, there are fewer entrained particulates in the off-gas, and the size of the off-gas scrubbing equipment can be reduced substantially.

Graphite electrodes offer important advantages for buried waste treatment applications. The graphite electrode can be operated in a submerged mode concentrating the heat in rather than above the material to be melted. Graphite electrodes are robust. These electrodes, are well suited to the requirements of remote operation. Graphite electrodes do not have to be water cooled, in contrast to metal electrode plasma torches, thus eliminating any possibility of an accident that could cause pressurized water to contact molten waste form or metal. Moreover, short DC arcs, which are highly effective in transferring thermal energy into the material to be processed, can be used. Graphite arc electrodes also permit the use of higher levels of DC arc current, providing the capability to transfer large amounts of the heat into the material to be processed. These factors make possible the high processing rates required for full-scale implementation of this technology at the INEL site.

The use of graphite electrodes and graphite crucibles in the process also provides a reducing condition for the production of the waste forms. It has been shown that the redox condition of waste form processing may affect their chemical durability and processing properties such as viscosity [Feng-1990, Buechele-1991]. The processed waste form can be poured into a large container to form a large monolith where the waste form is subjected to slow

cooling and devitrification. The uncontrolled devitrification can substantially reduce glass durability by two orders of magnitude [Kim-1995, Li-1996]. The controlled devitrification can in fact enhance waste form durability substantially [Feng-1996]. The waste form can also be made into small gems where glass is quenched without much devitrification, but the waste form occupies large volumes due to voids among the small gems.

The waste form development task within the DC plasma arc program is to develop waste form formulations for various waste streams targeted for treatability testing by the DC plasma arc technique. It is also to assess how the reducing condition in DC plasma and devitrification of the products affect waste form processing characteristics and waste form durability. This information is valuable for the design of optima processing conditions and optima formulations that achieve maximum waste loading, acceptable chemical durability, and proper melting temperatures and viscosities. The initial development of vitreous waste forms focuses on three separate waste streams with high metal contents.

2.0 Experimental

The waste forms were prepared in crucibles. The oxidizing melts were produced with oxides in air while the reducing condition is achieved by melting a mixture of the powders of the oxidized waste form and carbon powders. The devitrified waste form is referred to vitreous ceramic waste form while the quenched amorphous waste form is referred as glass in this report.

2.1 Glass and Vitreous Ceramic Preparation

The glasses were batched and melted according to Pacific Northwest National Laboratory (PNNL) Procedure PSL-417-GBM ("Procedure for Glass Batching and Melting, Rev. 0"). For each glass, the chemical components to be varied were batched together as oxides and carbonates. Using an Angstrom grinding machine, each batch was mixed in a grinding cell for 5 min to achieve a homogeneous mixture.

About 500 g of each formulation was melted in a platinum crucible under a lid (to reduce volatilization) using an electrically heated resistance furnace (Deltech DT-31). Furnace temperature was controlled by a Honeywell controller/programmer and monitored by one S-type thermocouple on the controller and a second, independent S-type thermocouple. The thermocouples were located in the middle of the furnace hot zone. Variation in temperature readout from the two thermocouples was $\pm 1.0^{\circ}\text{C}$ with respect to the preset melting temperatures.

The glass was removed from the furnace after one hour of melting. The glass was then ground to small powders. The powders were remelted at melting temperature for another hour and quenched into a glass. If the remelted glass was heat treated to promote devitrification after the one-hour of remelting, a vitreous ceramic waste form was formed. If the remelting was performed on the mixture of glass powders and carbon powders, a reduced waste form was formed.

2.2 Viscosity Estimates

Viscosity was estimated by comparing the pouring behavior of the melt with that of the viscosity standards.

2.3 Durability Tests

The chemical durability of a waste form is a measure of its capability to resist corrosion when in contact with an aqueous solution or water vapor. The durability of the glass/vitreous ceramics was measured by two tests: the toxicity characteristic leaching procedure (TCLP) and the product consistency test (PCT).

2.3.1 Toxicity Characteristic Leaching Procedure Test

The U.S. Environmental Protection Agency (EPA) toxicity characteristic leaching procedure (TCLP) (EPA 1988) was used to determine if arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver leached. The leachate concentrations, obtained after 18 hours of reaction of vitreous ceramic powders at 22°C in a sodium acetate buffer solution, were measured. This test was used to satisfy EPA regulatory requirements for RCRA-controlled toxic metals and to compare the performance of the waste form developed here in the TCLP test to that of other waste forms.

2.3.2 Product Consistency Test

The product consistency test (PCT) is based on previous research (Jantzen 1992) and was adapted as PNNL Procedure MCC-TP-19 ("Leaching Test Using PCT Test Method, Rev. 0"). The test was conducted using deionized water in Teflon™ containers at 90°C. New containers were baked at 200°C for one week to drive off residual fluorine and then washed thoroughly according to the PCT procedure. The glass/vitreous ceramics was ground in a tungsten-carbide grinding cell and then sieved through +200 -100 mesh stainless-steel sieves to obtain particle

sizes between 75 and 150 μm (equivalent spherical diameters). The large particles remaining on the top of the 100-mesh sieve were crushed repeatedly until all the waste form particles could pass through it. Using an ultrasonic cleaner, the crushed material was cleaned in deionized water and ethanol. It was then dried, weighed, and 2 g of the powdered waste form was added to a 60-ml Teflon container filled with 20 mL of deionized water. The ratio of the surface area of the waste form to solution volume was taken as 2000/m. The Teflon container and its contents were placed for 7 days in an oven preheated to 90°C. After the tests were terminated, aliquots of the leachate were filtered through a 0.45- μm filter and analyzed by ICP/AES. The PCT was performed in duplicate.

Results are reported as normalized elemental-mass losses according to the following equation:

$$NL_i = \frac{C_i}{f_i \frac{S}{V}} \quad (2)$$

where

- NL_i = the normalized mass loss based on element i (g/m^2),
- C_i = the analyzed concentration of element i in leachate (g/m^3),
- f_i = mass fraction of element i in the vitreous ceramics (unitless), and
- S/V = the ratio of glass surface area to solution volume ($1/\text{m}$), which is 2000/m here.

Nominal compositions are used for the normalized mass loss calculations.

2.4 Solution and Solid Analyses

2.4.1 Solution Analysis

Cations and radionuclides were analyzed with inductively coupled plasma - atomic emission spectrometry (ICP-AES) with an accuracy of $\pm 10\%$ for major elements and $\pm 50\%$ for radionuclides and minor elements. Anions were analyzed with ion chromatography or selective ion electrodes with an accuracy of about 50%. The pH was measured with a combination electrode with an accuracy of ± 0.1 pH unit.

2.4.2 Fusion Method for Composition Analysis

The composition of selected waste forms after melting was determined through the fusion method. The portions of the solid sample were ground and fused in molten potassium hydroxide or sodium peroxide. Each flux solution was solidified, then dissolved in HCl solution. The resulting solutions were then analyzed by ICP to identify the elements contained in the sample. Two different flux agents were used so that all major metal ions could be analyzed.

2.4.3 Solids Analyses

X-Ray Diffraction Analysis

X-ray powder diffraction (XRD) analysis was performed using a Philips vertical goniometer incorporating a variable divergence slit, a fixed (0.2°) receiving slit, and a graphite diffracted beam monochromator. The X-ray source was a fixed-anode, long fine-focus Cu tube operated at 40 kV, 45 mA (1800 W). The 2-theta range studied was $5-75^\circ$, step-scanned at $0.05^\circ/1.5\text{sec}$.

Specimens for XRD analysis were mounted in front-loading, cavity-type holders. Cavity dimensions were approximately 12mm diameter x 0.5 mm dp. (holding approximately 0.05 cm^3 or 0.13g of sample material). The sample holders were fabricated from off-axis single-crystal quartz (Gem Dugout, State College, PA) and produced low X-ray background.

Optical Microscopy Analysis

The samples were first cut into thin sections using a diamond saw. Some of the samples were observed directly without cutting. An Olympus PMG 3 optical microscope was used to examine the morphology and abundance of crystals.

Scanning Electron Microscopy

Scanning electron microscopy was done using a Hitachi S-450 with a Tracor-Northern TN-2000 energy-dispersive X-ray analysis system (EDS) which uses a solid state Si(Li) spectrometer for qualitative chemical analysis of major elements.

3.0 Results and Discussion

Three solid mixed waste streams were studied in this task. They are metal-R, Savannah River heterogeneous debris Pu^{238} waste, and Nom-60 waste. The Metal-R waste is a waste surrogate consisting of INEL soil, lime, and metals (approximately 50% by mass) and the metals in this surrogate are almost completely carbon steel. The chemical composition of metal-R waste is shown in Table I. The heterogeneous debris Pu^{238} waste exists at the Savannah River site and its composition is similar to the MWTP 5400 composition according to discussions with Dennis Bickford of Westinghouse Savannah River Company. This waste consists of activated carbon, wood, PVC, Neoprene, steels, glass beads, concretes, alumina crucibles, diatomaceous earth, RCRA metals and RARA organics such as Naphthalene, 1,2-dichlorobenzene, chlorobenzene, plutonium and cesium. The chemical composition of the Pu^{238} waste is also shown in Table I, where cerium was used as a surrogate for plutonium. The Nom-60 waste is a waste simulant consisting of the nominal INEL waste blended with 60% INEL soil composition. The nominal INEL waste is a weighted average blend of the waste materials expected from the INEL TSA at the Radioactive Waste Management Complex (RWMC). This waste consists of materials such as metals, solid combustibles, silicated organics, nitrate salts, and metal hydroxide sludge. The chemical composition of the Nom-60 waste is listed in Table I. The detailed formulations for all the waste forms produced in this task are tabulated in Appendix A. The waste formulation efforts were assisted by statistical glass property models developed in this laboratory and the predicted viscosity and PCT durability are shown in Appendix B.

3.1 Metal-R Waste

3.1.1 Melting and Waste Form Characterization

A total of four melts, Metal-A, Metal-B, Metal-C, and Metal D, were performed on this waste and the composition of each melt was shown in Table II. The melting conditions and the waste loadings of these melts are listed in Table III. Metal-A with waste loading of 34% was made in air and was quenched as a glass. Metal-C with the same chemical composition as Metal-A was melted with 1% carbon and was quenched as a glass. The effects of reducing melting conditions on glass processing and product properties can be assessed by comparing the behavior of Metal-A and Metal-C. Metal-B with waste loading of 100% was melted in air and was heat treated at 1000°C for 17 hours to produce a devitrified product, a vitreous ceramic waste form. Metal-D with the same chemical composition as Metal-B was melted with 1% carbon and was heat treated in the same way as that for Metal-B to become a vitreous ceramic waste form. The effects of reducing melting conditions on waste form processing and devitrified product

Table I Waste stream compositions used to formulate the vitreous waste forms

Oxide	Nom-60 Waste Stream	Metals-R Waste Stream	MWTP 5400 Waste Stream
Ag ₂ O	0.000	0.584	
Al ₂ O ₃	8.728	5.212	18.575
As ₂ O ₃	0.001	0.006	
BaO	0.065		
CaO	21.618	6.657	3.567
CdO	0.000	0.009	0.238
CeO ₂	0.428		0.296
Cl	2.566	0.016	0.498
Cr ₂ O ₃	1.188	0.042	0.121
Cs ₂ O	0.001	0.177	0.355
CuO	0.276	0.003	
Fe ₂ O ₃	20.207	58.801	24.154
HfO ₂	0.000		
Hg	0.000		
K ₂ O	1.846		0.229
MgO	1.752	1.081	0.475
MnO	0.105	0.034	0.107
MoO ₃	0.017		
Na ₂ O	1.471	1.221	2.630
NiO	0.488	0.010	0.133
P ₂ O ₅	0.106	0.133	0.340
PbO	0.075	0.002	0.078
S	0.070		
SeO ₂	0.000		
SiO ₂	38.124	25.703	48.204
TiO ₂	0.387	0.302	
ZnO	0.010		
ZrO ₂	0.471	0.006	
Total	100.000	100.000	100.000

Table II Target vitreous waste form compositions formulated from each waste stream.

Oxide	Nom-60-A	Nom-60-B	Nom-60-C*	Nom-60-D	Nom-60-E*	Metal-A	Metal-C*	Metal-B	Metal-D*	Lab-1Ga	Lab-1Gb*	Lab-1VCa	Lab-1VCb*	Lab-.83a	Lab-.83b*
Ag ₂ O	-	-	-	0.000	0.000	0.199	0.199	0.584	0.584						
Al ₂ O ₃	8.728	8.728	8.728	7.803	7.803	6.000	6.000	5.212	5.212	18.575	18.575	18.575	18.575	15.396	15.396
As ₂ O ₃	-	-	-	0.001	0.001	0.002	0.002	0.006	0.006						
B ₂ O ₃						5.917	5.917	0.000	0.000						
BaO	0.065	0.065	0.065	0.058	0.058										
CaO	21.618	21.618	21.618	19.326	19.326	2.264	2.264	6.657	6.657	3.567	3.567	3.567	3.567	2.956	2.956
CdO	-	-	-	0.000	0.000	0.003	0.003	0.009	0.009	0.238	0.238	0.238	0.238	0.197	0.197
CeO ₂	0.428	0.428	0.428	0.383	0.383					0.296	0.296	0.296	0.296	0.245	0.245
Cl	2.566	2.566	2.566	2.294	2.294	0.006	0.006	0.016	0.016	0.498	0.498	0.498	0.498	0.412	0.412
Cr ₂ O ₃	1.188	1.188	1.188	1.062	1.062	0.014	0.014	0.042	0.042	0.121	0.121	0.121	0.121	0.100	0.100
Cs ₂ O	-	-	-	0.001	0.001	0.060	0.060	0.177	0.177	0.355	0.355	0.355	0.355	0.294	0.294
CuO	0.276	0.276	0.276	0.247	0.247	0.001	0.001	0.003	0.003						
Fe ₂ O ₃	20.207	20.207	20.207	18.064	18.064	20.000	20.000	58.801	58.801	24.154	24.154	24.154	24.154	20.020	20.020
HfO ₂	-	-	-	0.000	0.000										
K ₂ O	1.846	1.846	1.846	1.650	1.650					0.229	0.229	0.229	0.229	0.190	0.190
MgO	1.752	1.752	1.752	1.566	1.566	0.368	0.368	1.081	1.081	0.475	0.475	0.475	0.475	0.393	0.393
MnO	0.105	0.105	0.105	0.094	0.094	0.012	0.012	0.034	0.034	0.107	0.107	0.107	0.107	0.089	0.089
MoO ₃	0.017	0.017	0.017	0.016	0.016										
Na ₂ O	1.471	1.471	1.471	6.000	6.000	15.000	15.000	1.221	1.221	2.630	2.630	2.630	2.630	15.000	15.000
NiO	0.488	0.488	0.488	0.436	0.436	0.004	0.004	0.010	0.010	0.133	0.133	0.133	0.133	0.110	0.110
P ₂ O ₅	0.106	0.106	0.106	0.095	0.095	0.045	0.045	0.133	0.133						
PbO	0.075	0.075	0.075	0.067	0.067	0.001	0.001	0.002	0.002	0.340	0.340	0.340	0.340	0.282	0.282
S	0.070	0.070	0.070	0.062	0.062					0.078	0.078	0.078	0.078	0.065	0.065
SeO ₂	-	-	-	0.000	0.000										
SiO ₂	38.124	38.124	38.124	40.000	40.000	50.000	50.000	25.703	25.703	48.204	48.204	48.204	48.204	44.244	44.244
TiO ₂	0.387	0.387	0.387	0.346	0.346	0.103	0.103	0.302	0.302						
ZnO	0.010	0.010	0.010	0.009	0.009										
ZrO ₂	0.471	0.471	0.471	0.421	0.421	0.002	0.002	0.006	0.006						
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.99	99.99

- Denote oxides present at less than 0.001 wt% in waste that were not included in waste form.

*1wt% carbon powder addition to the ground glass to promote reducing atmosphere.

properties can be assessed by comparing the behavior of Metal-B and Metal-D. The detailed melting and characterization of each waste form are described in more details below.

Metal-B:

Metal-B was melted at 1450°C in Pt. under oxidizing conditions then heat treated in the same crucible overnight at 1000°C for 17 hours. The viscosity at 1450°C was approximately 5 Pa.s by comparing the pouring behavior of Metal-B versus the viscosity standard liquids. The heat treated vitreous ceramic was quenched in a room temperature water bath for aid in the removal of the sample.

The sample appears to be fully crystalline and is black with a metallic shine. When ground to a 100 mesh powder the color remained black. Figure 1 is an optical photo of the fracture surface taken under reflected light at 400 X magnification. Diamond shaped crystals (or mirrored triangles appear to be embedded in a continuous matrix. The average crystal size is in the range of twenty to thirty microns. Qualitative x-ray diffraction was performed on powders ground to finer than 200 mesh. The results report the sample to be 75 vol % crystalline with 80% spinel type, 10% hematite, and 10% Augite type crystals. An environmental SEM and qualitative EDS were used to analyze the structure. Figure 2 is a SEM micrograph taken at 700 X. Figure 3 shows the EDS spectrums of the observed phases. The spectrum (a) of Figure 3 is a microprobe of the central diamond shaped crystal. The large Fe peak identifies the crystal as the spinel type. The glassy matrix shown in the spectrum (b) of Figure 3 produces a large silicon peak with peaks of aluminum, calcium, and iron at a quarter of the intensity. The spectrum (c) of Figure 3 represents the needle shaped crystals with an Agate type structure. Figure 4 shows a large triangular spinel type crystal with attached agate type crystals.

Metal-D:

Metal-D was melted under reducing conditions at 1450°C in an alumina crucible and heat treated in the same crucible overnight at 1000°C for 17 hours. A first attempt to melt Metal-D was unsuccessful do to severe corrosion of a silica crucible and the second attempt to melt the melt in a high purity alumina crucible was successful. The viscosity of the melt was lower than Metal-B. The top and bottom sections of the sample have a similar appearance of a rough metallic black fracture surface. The top section appears to have a slightly smaller particle size than the bottom. Several metal beads assumed to be silver are present in the bottom section of the melt due to the reducing melting condition. The central region of the sample consists of shiny black layered crystals that are not densely packed. When ground to a 100 mesh powder

Table III Laboratory Melting Conditions During Second Hour*

Glass ID	Waste Loading (Wt%)	Temperature (°C)	Cruc.	Atmosphere	Heat Treatment	Viscosity (Pa-s) Predicted**	Viscosity (Pa-s) Observed
Nom-60-A	100	1425	Pt	Air	Quenched	5	8
Nom-60-B	100	1425	Pt	Air	16hrs.@ 1000°C	5	8
Nom-60-C	100	1425	Si	Reducing	Quenched		5
Nom-60-D	90	1300	Pt	Air	Quenched	2	6
Nom-60-E	90	1300	Si	Reducing	Quenched		5
Metal-A	34	1280	Pt	Air	Quenched	7	12
Metal-C	34	1280	Si	Reducing	Quenched		10
Metal-B	100	1450	Pt	Air	17hrs.@ 1000°C	1	5
Metal-D	100	1450	Al	Reducing	17hrs.@ 1000°C		
Lab-1Ga	100	1450	Pt	Air	Quenched	22	28
Lab-1Gb	100	1450	Al	Reducing	Quenched		25
Lab-1VCa	100	1450	Pt	Air	16hrs.@ 1000°C	22	28
Lab-1VCb	100	1450	Al	Reducing	16hrs.@ 1000°C		
Lab-.83a	83	1375	Pt	Air	Quenched	7	15
Lab-.83b	83	1375	Si	Reducing	Quenched		12

*All first melts were performed at the above temperature in Pt crucibles in air for 1 hour then quenched and ground to a powder prior to the second hour of melting under the specified conditions.

**Predictions obtained from Arrhenius Equation and CVS Model for LLW Low Temperature Study May 5, 1995.

the color remained black. Figure 5 is an optical photo of the central region of the melt at 100 X magnification under reflected light. The layered triangular crystals range in size from 200 - 600 microns. The materials in the central region are 100% crystalline and are not imbedded in a matrix. Qualitative x-ray diffraction was performed on powders ground to finer than 200 mesh. The results describe the sample as 100% crystalline made up of four different phases. The major phases are spinel type and Augite type at 55% and 25% respectively. Fayalite and Albite type crystals make up the remaining composition. SEM and EDS analysis were used to determine the distribution of the crystal types. Figure 6 is a SEM micrograph of the central region of the crucible taken at 300 X magnification. The three-dimensional crystals with triangular faces are spinel type according to the spectrum labeled (b) of Figure 6. Figure 7 is a SEM micrograph taken at 1500 X magnification and the EDS spectra presented in Figure 8 suggest that the diamond shaped crystal in the center be spinel type embedded in an Albite type crystalline matrix. Figure 9 is taken at 1500 X magnification. The spectrum in Fig. 10(a) presents the composition of the feather shaped crystals to be Agate type, and Fig. 10(b) suggests the flat crystal in the lower right-hand corner to be spinel type.

Metal-A:

Metal-A was melted in Pt. at 1280 C under normal furnace atmospheric conditions then quenched on a room temperature stainless steel plate. The viscosity was estimated to be 12 Pa.s. The bulk of the glass sample is black and appears to be homogeneous with no bubbles or agglomerates. A tan powder is produced upon grinding of the sample. The twenty four-hour qualitative x-ray diffraction analysis was performed on powder finer than 200 mesh. The sample was reported to be fully amorphous.

Metal-C:

Metal-C was melted in a high silica crucible under reducing conditions at 1280 C then quenched on a room temperature stainless steel plate. The viscosity was estimated to be 10 Pa.s. The bulk of the glass sample is black and appears to be homogeneous with no bubbles or agglomerates. The top of the quenched glass has brownish parallel to the flow of glass. A black powder is produced upon grinding of the sample. The reflective setting was used on the optical microscope to photograph the cross section of the sample. At 100 X magnification the cross section is uniform with thousands of white specs of less than a micron in diameter. On the top surface of the sample the same type of crystals can be seen within the streaks. Figure 11 shows the streaking magnified 500 X. The crystals do not appear in the dark streaks. Twenty-four hour

qualitative x-ray diffraction analysis was performed on powder finer than 200 mesh. The sample was reported to contain 1 vol % crystals determined to be silver metal.

Composition and Redox Analysis:

The results of the ICP analysis of the Na and K fused samples are presented in Appendix C-1. No notable deviation between the analyzed and target compositions are observed. The results of the redox analysis are presented in Table IV in a ratio of FeII/(Total Fe). The ratio for Metal-A is 0.035 compared to a ratio of 0.473 for Metal-C. If translated to percentages, the percent of Fe II present under reducing conditions is about 47% apposed to 3.5% in the sample melted under oxidizing conditions. The percentage of Fe II in Metal-B is 14% compared to 30% Fe II in Metal-D. The decreased ratio of Fe II in the vitreous ceramics may be linked to the diffusion of oxygen over 17 hours of heat treatment at 1000 C.

3.1.2 Waste Form Chemical Durabilities

The chemical durability of the waste form was evaluated using TCLP and PCT tests. The results of TCLP were shown in Table V. The normalized major element release from 7 day PCT tests are shown in Table VI and the complete PCT results are shown in Appendix D. The PCT results show that:

- (1) The devitrified Metal-B with 100% waste loading is 44% more durable (according to sodium release) than the glass melt of Metal-A. It is known that spinel crystals are very durable and the formation of crystalline spinel in the melt alters the chemical composition of the residual glass. Recalculation of composition in the residue glass after removing 53% of the spinel crystals shows that the residual glass composition is enriched in SiO_2 (55%) and Al_2O_3 (11.6%), but low in alkalis (2.6% Na_2O). It is now easy to understand why the durability of Metal-B is better than Metal-A by comparing the residual glass composition of Metal-B with the glass composition of Metal-A. Metal-A glass has 10 times higher in Na_2O (20%) while having a similar amount of silica and alumina as the residual glass of Metal-B;
- (2) The reducing melting conditions decreased the chemical durability (in terms of sodium release) of glass by 400% (Metal-A versus Metal-C) and of vitreous ceramics by 720% (Metal-B versus Metal-D). The reducing condition in glass converted 44% more ferric iron into ferrous iron and ferric iron can serve as a network former to strength glass structure while ferrous iron cannot. The more sever redox effect on the durability of vitreous ceramic is probably because the reducing condition promoted the formation of 45% silica and alumina consuming crystals such as Albite-type ($\text{Na}(\text{Si}_3\text{Al})\text{O}_8$), Fayalite

Table IV Results from iron II and total iron analysis

	Target Fe wt%	Analyzed Fe wt%	Analyzed FeII wt%	FeII/Fe Ratio
Lab-1Ga	16.894	16.991	4.594	0.270
Lab-1Gb	16.894	17.278	10.038	0.581
Lab-1VCa	16.894	16.337	4.741	0.290
Lab-1VCb	16.894	16.590	6.219	0.375
Lab-.83a	14.003	14.170	0.984	0.069
Lab-.83b	14.003	13.942	6.009	0.431
Metal-A	13.988	14.117	0.497	0.035
Metal-C	13.988	14.101	6.663	0.473
Metal-B	41.125	38.711	5.416	0.140
Metal-D	41.125	40.187	11.904	0.296
Nom-60-A	14.133	14.493	1.813	0.125
Nom-60-B	14.133	11.334	0.802	0.071
Nom-60-C	14.133	12.743	6.492	0.509
Nom-60-D	12.635	12.873	0.221	0.017
Nom-60-E	12.635	12.898	8.342	0.647

Table V ICP Analysis Results from TCLP Testing (Mg/L.)

	Ag	As	Ba	Cd	Cr	Ni	Pb	Se
N-60-A	-	-	<0.01	-	<0.06	<0.06	<0.03	-
N-60-B	-	-	0.29	-	<0.06	<0.06	0.69	-
N-60-C	-	-	<0.01	-	<0.06	<0.06	<0.03	-
N-60-D	<0.02	-	<0.01	<0.02	<0.06	<0.06	<0.03	-
N-60-E	0.03	-	0.03	<0.02	<0.06	0.12	<0.03	-
Metal-A	<0.02	-		<0.02	<0.06	<0.06	<0.03	
Metal-C	<0.02	-		<0.02	<0.06	<0.06	<0.03	
Metal-B	<0.02	-		<0.02	<0.06	<0.06	<0.03	
Metal-D	<0.02*	-		<0.02	<0.06	<0.06	<0.03	
L-1Ga				<0.02	<0.06	0.06	<0.03	
L-1Gb				<0.02	<0.06	0.65	<0.03	
L-1VCa				<0.02	<0.06	<0.06	<0.03	
L-1VCb				<0.02	<0.06	<0.06	<0.03	
L-.83a				<0.02	<0.06	<0.06	<0.03	
L-.83b				<0.02	<0.06	<0.06	<0.03	
EPA Limit (TCLP)	5.00	5.00	100.00	1.00	5.00		5.00	1.00
Land Disposal Limit	0.072			0.066	5.200	0.320	0.510	

* Silver metal droplets seperated during melting were not present in the test sample in a representative proportion.

- Elements are present in the waste composition with out inclusion in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Table VI Normalized elemental release from ICP analysis of PCT leachate.

Normalized Elemental Release 7 Day PCT Test						
Element	Metal-A	Metal-C*	Metal-B	Metal-D*		
Si	0.164	0.357	0.084	0.109		
B	0.266	0.829				
Na	0.341	1.377	0.237	1.714		
pH	10.310	11.285	9.305	10.305		
Element	Lab-1Ga	Lab-1Gb*	Lab-1VCa	Lab-1VCb*	Lab-.83a	Lab-.83b*
Si	0.039	0.039	0.040	0.060	0.134	0.222
Na	0.088	0.179	0.090	0.360	0.297	0.996
pH	8.155	8.805	8.255	9.650	10.840	11.505
Normalized Elemental Release 8 Day PCT Test						
Element	Nom-60-A	Nom-60-B	Nom-60-C*	Nom-60-D	Nom-60-E*	
Si	0.043	0.044	0.042	0.043	0.020	
Na	0.129	0.392	0.097	0.123	0.117	
pH	9.665	9.920	10.010	10.345	10.345	

*Denotes Reducing atmosphere.

(Fe_2SiO_4), and Augite ($\text{Ca}[\text{Fe,Mg}]\text{Si}_2\text{O}_6$). This results in the depletion of network formers in the residual glass matrix that determines the overall durability of the waste form.

- (3) The reducing condition increased the leachate pH in both glasses and vitreous ceramics by one pH unit within 7-day of PCT tests. Vitreous ceramics had a leach pH of one unit lower than glasses in both oxidized and reduced conditions.

The TCLP results in Table V showed that all the melts pass the EPA land disposal requirements. The comparison of TCLP results versus PCT results may also suggest that the TCLP results are not very sensitive to waste form composition change and processing variations. TCLP may not be a suitable test for waste form optimization, but is a test related to the acceptance of the waste forms developed.

3.1.3 Conclusions

In treating Metal-R wastes, the waste form durability can be reduced by 400 to 700%, the melt viscosity can also be lowered by more than 20%, and the tendency of the waste form to devitrify can be increased when it is produced in a reducing environment similar to that of a DC plasma process. The devitrified waste form is actually more durable than the quenched glass, suggesting the waste form produced from treating Metal-R wastes can be poured into a large container without concerns of devitrification and slow cooling. The final products always pass TCLP tests.

It is recommended to treating Metal-R waste at 100% waste loading in the DC plasma process and pour them into a large container to reduce the overall waste volume. Moderate increases in oxygen concentration in the processing atmosphere during plasma processes are recommended for the sake of the long-term durability of the product, but it is not required to pass TCLP tests.

3.2 Savannah River Heterogeneous Debris Pu^{238} Waste

3.2.1 Formulation & Melting

The initial task consisted of transforming the raw waste materials of known weight percentages into their respective chemical formulas in moles. The hydrogen and oxygen present as water and organic compounds are ignored based on the assumption that they will be completely oxidized into gases and volatilized during processing. The chemical formula for mild steel was obtained by averaging the composition of 1010 and 1020 steels. The formula for

the glass beads are assumed to be common soda lime silicate window glass. The formula for the cured and crushed concrete is derived from the composition of Type I Portland Cement, sand, gravel, and water mixed in a weight ratio of 1:2:3:0.5 respectively. The sand is assumed to be silica, and the gravel composition is based on a phase diagram for igneous plutonic rocks. The chosen composition is 60% plagioclase, 20% quartz, and 20% alkali feldspar. From the moles of each element the mass of each was calculated based on the weight percent of the raw waste component. The mass of each metal oxide was then calculated along with carbon, chlorine, and sulfur.

The mass fraction of each oxide plus carbon, chlorine, and sulfur represents the laboratory simulation of the waste stream including cerium oxides as a surrogate for plutonium oxide. In preparation for crucible melting, chlorine was changed to 0.5 wt % and the carbon was eliminated in the formulation of the 100% waste compositions (Lab-1). In an attempt to formulate a glass with maximum waste loading and increased processability the amount of Fe_2O_3 was held at 20% while Na_2O was increased 15%. This resulted in a waste form with 83% waste loading (Lab-.83). The steps used in these calculations are outlined in Appendix A.

The CVS Model shown in Appendix B provides predictions helpful during formulation and melting. A viscosity of 22 Pa.s at 1450°C based on the Arrhenius equation was predicted for Lab-1. The Lab-1 composition contains 48% silica, 18% alumina, and 3% alkali. The high silica in the waste stream makes the production of a glass and vitreous ceramic waste form possible with 100% waste loading. To eliminate composition variable and facilitate the comparison of the melting behavior, durability, crystallization behavior and morphology, several melts were made at each waste loading.

The target compositions of the six melts: Lab-1Ga, Lab-1Gb, Lab-1VCa, Lab-1VCb, Lab-.83a, and Lab-.83b are listed in Table II. The sample ID for each waste is designed as: (VC) represents a heat treated sample of vitreous ceramic; (G) represents a quenched glass sample; (b) represents a reducing melting condition achieved by melting the waste form powders with 1 wt% of carbon powders; (a) represents normal oxidizing melting conditions present in an air environment.

After the first hour of melting to produce three 500g melts, Lab-1G, Lab-1VC, and Lab-.83, each melt was ground and the powder was halved to produce the oxidized waste form suffixed with (a) and reduced waste form suffixed with (b). Table III lists the melting conditions, heat treatment, and viscosities of each waste form during the second hour of melting. The melting temperature for the first hour and second hour are the same. 1 wt% carbon powder was

mixed with the 250 g powder samples for the production of Lab-1Gb, Lab-1VCb, and Lab-.83b. A high silica crucible and lid were used for Lab-.83b at a melting temperature of 1375°C. Alumina crucibles had to be used for the second melt of Lab-1Gb and Lab-1VCb due to the reducing conditions and the corrosive nature of FeO at 1450°C (which penetrated a high silica crucible). The oxidizing samples were melted in Pt. crucibles with lids in air. Lab-1Ga and Lab-1VCa were melted at 1450°C. Lab-.83a was melted at 1375°C. The glass samples were quenched on a stainless steel plate, and the vitreous ceramic samples were heat treated for sixteen hours at 1000°C. Powder preparation began with the selection of fractured samples representative of the entire melt.

3.2.2 Characterization of Waste Form

Lab-1Ga:

Lab-1Ga was melted in a Pt crucible at 1450°C then quenched on a room temperature stainless steel plate. The viscosity was estimated to be 28 Pa.s. The bulk of the glass sample is black and appears to be uniform with no bubbles or agglomerates. A black powder is produced upon grinding of the sample. The top surface of the glass is reddish brown with a predominant streak of dull bluish grey crystals parallel to the direction of glass flow. The reflective setting was used on the optical microscope to photograph the cross section of the sample. At 500 X magnification the sample is uniform, but there is an apparent crystalline texture. Twenty-four hour qualitative x-ray diffraction analysis was performed on powder finer than 200 mesh. The sample was reported to be 45 volume % crystalline. The crystals have a nickel rich spinel type structure.

Lab-1Gb:

Lab-1Gb was melted at 1450°C in an alumina crucible then quenched on a room temperature stainless steel plate. Lab-1Gb had a skim over the melt that showed signs of carbon powder and a reddish mineral that had possibly precipitated from the crucible atmosphere. The melt possessed a short working viscosity range and there were several agglomerates of carbon visible. The viscosity of the melt was observed to be 25 Pa.s. The variation of 3 Pa.s is not truly resolvable with this singular observation, but the apparent lower viscosity of the reducing melt was notable. The bulk of the glass is black and appears to be uniform apart from a few large open bubbles on the surface of the quenched sample. Powders ground to 100 meshes still appear black. The top surface of the sample is a dull grey black with a rough texture. The bottom of the sample is glossy black with streaks of graphite powder. With the reflective setting on the optical

microscope the cross section of the sample appears to be completely amorphous with instances of wake hackle caused by inclusions of 50 microns in diameter. Twenty-four hour qualitative x-ray diffraction analysis was performed on the quenched glass sample ground to powder finer than 200 mesh. The sample was reported to be less than 5 vol. % crystalline and these crystal was a nickel rich spinel type.

Lab-1VCa:

Lab-1VCa was melted at 1450°C in Pt. then heat treated in the same crucible overnight at 1000°C for 16 hours. The vitreous ceramic had a uniformly dense appearance. The top surface was silvery grey metallic and the bulk of the sample was smooth black. When ground to a 100 mesh powder the color remained black. Perpendicular rows of diamond shaped crystals in what appears to be a continuous matrix observed with the optical microscope under reflected light. The average crystal size is in the range of one to two microns. Qualitative x-ray diffraction was performed on powders ground to finer than 200 mesh. The results report the sample to be 40 vol % crystalline with a spinel type structure. An environmental SEM and qualitative EDS were used to analyze the crystals. Figure 12 shows that the diamond shaped crystals appear to be four diamonds attached to a fifth central diamond's corners. It also appears to be similar to the spinel type crystal pictured in Figure 7 of Metal-D. Figure 13(a) suggests the diamond shaped crystals to be spinel type. Spectrum 13(b) shows the glassy matrix represented by a large silicon peak with peaks of aluminum, calcium, and iron at a quarter of the intensity.

Lab-1VCb:

Lab-1VCb was melted under reducing conditions at 1450°C in an alumina crucible then heat treated in the same crucible for sixteen hours at 1000 C. The crucible and sample were slit in half to reveal that part of an alumina lid had fallen into the melt. The majority of the vitreous ceramic appeared to be uniformly dense and crystalline with the same characteristics as Lab-1VCa. Along the top surface and contact points with the alumina crucible the texture of the fractured sample appeared rough with a dull black texture compared to the alternate glossy black surface away from the alumina. When ground to a 100 mesh powder the color remained black. The crystal shape and pattern in the smooth areas of the sample appear to be identical to those observed in Lab-1VCa. The qualitative x-ray diffraction was performed on powders ground to finer than 200 mesh selected from the rough and smooth sections of the sample. The sample was reported to be 40 vol % crystalline composed of 28% spinel type and 12% albite type $\text{Na}(\text{Si}_3\text{Al})\text{O}_8$. Figure 14 is a SEM micrograph taken at 1000 X magnification showing a region very similar to the one seen in Lab-1VCa. Spectrums (a) and (b) in Figure 15 show the peaks

obtained from the diamond shaped crystals and the glassy matrix. The qualitative EDS analysis suggests the diamond shaped crystals to be spinel type and the silica and alumina peaks were from the glass beneath the crystals. The glassy matrix of Figure 15(b) has a large silicon peak, an aluminum peak of quarter of the intensity, and small calcium and iron peaks. The uniform crystal patterns are not predominant in the rough section of the sample near the crucible wall. The glassy areas give similar patterns with a dominant silicon peak. Many areas in the rough section produce high aluminum peaks, but a specific crystal shape was not observed.

Lab-.83a:

Lab-.83a was melted in Pt. at 1375°C then quenched on a room temperature stainless steel plate. The viscosity was estimated to be 15 Pa.s. The glass has a relatively good working range. The bulk of the glass sample is black and appears to be uniform with no bubbles or agglomerates. When ground to a 100 mesh powder it remains black. The top surface of the glass has a bluish silver glazing with minimal streaking. A few specks of metal can be seen. The reflective setting was used on the optical microscope to photograph the cross section of the sample. At 500 X magnification the sample appears amorphous. The twenty-four-hour XRD analysis reported the sample to be 10 vol % crystalline of a spinel type structure.

Lab-.83b:

Lab-.83b was melted in a high silica crucible at 1375°C then quenched on a room temperature stainless steel plate. The viscosity was estimated to be 12 Pa.s. The bulk of the glass sample is black and appears to be uniform with no bubbles or agglomerates. When ground to a 100 mesh powder it still remains black. The top surface of the glass has a streaking pattern of blue, red, green, and metallic silvers. The streaking is not patterned after the glass flow. At 500 X magnification the sample appears amorphous with many small inclusions of approximately one micron apparent on the fractured surface. The twenty four-hour XRD analysis reported the sample to be 5 vol % crystalline of a spinel type structure.

Analyses of Glass Compositions and Redox States:

The results of the ICP analysis of the Na and K fused samples are presented in Appendix C-3. No notable deviation between the analyzed and target compositions are observed. The results of the redox analysis are presented in Table III in a ratio of FeII/(Total Fe). The ratio for Lab-1Ga is 0.27 compared to a ratio of 0.58 for Lab-1Gb. If translated to percentages, the percent of Fe II present under reducing conditions is about 60% apposed to 30% in the sample

melted under oxidizing conditions. The percentage of Fe II in Lab-1VCa is also 30%, but Lab-1VCb possesses only 40% Fe II. The decreased amount of Fe II may be linked to the diffusion of oxygen over 16 hours of heat treatment at 1000°C. A more drastic percentage change can be seen between Lab-.83a, with 7% Fe II, and Lab-.83b with 40% Fe II. The larger percentage of Fe II in Lab-1Ga & 1VCa compared to that of Lab-.83a may be due to the higher melting temperature.

3.2.3 Waste Form Chemical Durabilities

The complete PCT results for each sample are presented in Appendix D. The pH of the leachate and the normalized releases of Si and Na are presented in Table VI. Lab-1Ga has lower releases than Lab-1Gb for all detected elements except Ca. The most drastic change is apparent in Na. The reduced glass had a release of 0.18 g/m² compared to 0.09 g/m² for the oxidizing melt. The pH changed from 8.16 to 8.81 under reducing conditions. The increased elemental release and increased pH for the reduced melts is an obvious trend. Lab-1VCa has almost identical releases as those of Lab-1Ga for the detected elements and the pH is comparable at 8.26. Lab-1VCb has the highest releases and pH of the 100% waste samples. The Na release is 0.36 g/m² and the pH is 9.65. This is the same trend as seen in the waste forms for Metal-R waste in that the reducing condition deteriorates waste form durability more in vitreous ceramics than in glass. The PCT leachates from the Lab-.83 compositions had a yellowish tint signifying low durability. The release of Na from L-.83b is 1.00 g/m² with a pH of 11.5. Lab-.83a has a Na release of 0.30 g/m² and a pH of 10.8. It is also notable that Lab-.83b has detectable releases of the majority of the elements, including Cd, Cr, and Ni. The reduced waste loading in Lab-.83 melts also substantially reduced their chemical durability in comparison with glasses (Lab-1 series) of 100% waste loading.

The Toxicity Characteristic Leaching Procedure was performed on each sample to ensure the acceptability of the waste form based on the EPA standards for regulated waste storage and land disposal limits. The results of the ICP analysis of the leachates for hazardous elements are presented in Table V. All of the waste forms except Lab-1Gb passed the land disposal limits for all the hazardous elements. In the case of Lab-1Gb, nickel was detected at a level of 0.65 mg/L, which passes the regulated disposal limits for nickel, but failed the land disposal limit.

3.2.4 Conclusions

In treating the heterogeneous debris Pu²³⁸ wastes, the reducing conditions during DC plasma process can decrease chemical durability 330% for glass and 400% for vitreous ceramics,

lower viscosity of the melts, and decrease the tendency for glass to devitrify. The devitrified product has similar durability as the glass because the formation of spinel crystals does not affect glassy matrix durability. However, the reduced melting condition deteriorated waste form durability more in the case of vitreous ceramics than in the case of glass. Increasing waste loading in the case of Pu^{238} heterogeneous wastes actually increased waste form durability under oxidizing condition and also increased the tolerance of waste form to reducing conditions. The lower waste loading failed the TCLP test under reducing conditions.

The recommendation for treating the heterogeneous debris Pu^{238} waste is that processing 100% waste directly without needing any other additives. To achieve the desired chemical durability, it may be necessary either to provide a more oxidizing atmosphere (that may be difficult with the DC plasma process) or to pour the final product into small size gems to reduce devitrification, which is achievable in the DC plasma process.

3.3 Nom-60 Waste

3.3.1 Formulation & Melting

Five glasses based on Nom-60 wastes were formulated and melted and the chemical composition of each melt is shown in Table II. Nom-60-A with 100% waste loading was melted at 1425°C and the viscosity was estimated to be about 8 Pa.S. This melt was quenched onto a stainless steel plate in air. Nom-60-B has the same chemical composition and the same melting viscosity, but was heat treated at 1000°C for 16 hours to form a vitreous ceramic waste form. Nom-60-C has the same chemical composition as that of Nom-60-A, but the second hour melt was mixed with 1wt% carbon powders to achieve reducing melting condition. The viscosity was estimated to be about 5 Pa.S and the melt was quenched onto a stainless steel plate. Nom-60-D and Nom-60-E have 90% waste loading and were melted at 1300°C . Nom-60-E was mixed with 1% carbon powders during the second hour of melting and both Nom-60-D and Nom-60-E were quenched onto a stainless steel plate. The detailed melting conditions for these melts are also listed in Table III.

3.3.2 Characterization of Waste Form

The optical micrograph in Figure 16(a) shows the slightly crystalline top surface of Nom-60-A. This is consistent with the XRD results that indicate approximately 25 volume% of crystallinity in the Nom-60-A sample. These crystals consist of 75% magnetite, FeFe_2O_4 , and 25% clinopyroxene, $\text{Ca}(\text{Ti},\text{Mg},\text{Al})(\text{Si},\text{Al})_2\text{O}_6$.

The XRD results for N-60-B indicate 100% crystallinity in this vitreous ceramic sample. The identified crystals are 50% of Augite-type ($\text{Ca}(\text{Fe,Mg})\text{Si}_2\text{O}_6$), 30% spinel-type, 15% wallastonite (CaSiO_3), and Anorthite-type ($((\text{Ca,Na})(\text{Al,Si})_2\text{Si}_2\text{O}_8)$). The optical micrograph in Figure 16(b) shows the fully crystallized crystalline surface. Figure 17 is a SEM micrograph of Nom-60-B showing the triangular shaped spinel type crystals and the more abundant needle shaped augite type crystals. Their corresponding EDS spectra of these crystals are shown in Figure 18. Figure 19 also shows a large Si-Ca-Fe crystals within the more abundant Augite-type and spinel type of crystals.

The Nom-60-C sample is a quenched glass sample melted under reducing conditions and Figure 16(c) shows the amorphous surface of the sample surface. There is only about 5% crystallinity was detected in the Nom-60-C sample and they are 95% magnetite and 5% clinopyroxene. This means that a reducing melting condition can effectively reduce the tendency of devitrification.

Nom-60-D and Nom-60-E have 90% waste loading and were both quenched onto a stainless steel plate after melting. The normal melting in the air (Nom-60-D) shows much more crystallinity (Figure 20a) than the Nom-60-E (Figure 20b). The square shaped augite crystals are shown clearly in the SEM micrograph of Figure 21 and the compositions of the Augite and the residual glassy phase are shown in Figure 22 of Nom-60-D. A well-formed triangular spinel phase in Nom-60-D is shown in Figure 23 with its EDS composition. The spinel crystals are much more abundant than Augite type crystals in Nom-60-D. Nom-60-E is essentially amorphous.

Analyses of Glass Compositions and Redox States:

The results of the ICP analysis of the Na and K fused samples are presented in Appendix C-3. No notable deviation between the analyzed and target Nom-60 compositions are observed. The results of the redox analysis are presented in Table III in a ratio of $\text{FeII}/(\text{Total Fe})$. The ratio for Nom-60-A is 0.13 compared to a ratio of 0.51 for Lab-1Gb. If translated to percentages, the percent of Fe II present under reducing conditions is about 51% apposed to 13% in the sample melted under oxidizing conditions. The percentage of Fe II in Nom-60D is 2%, but Nom-60-E possesses 65% Fe II.

3.3.3 Waste Form Chemical Durabilities

The chemical durability of these glasses was evaluated using TCLP and PCT tests. The TCLP results are presented in Table V and PCT results are presented in Table VI. The TCLP results show that the heat treated sample, Nom-60-B, failed TCLP tests for lead release according to the Land Disposal Limit. The results also show that the reduced Norm-60-E has slightly higher releases of Ag, Ba, and Ni than those of the same composition melted under oxidizing condition of Nom-60-C. Such redox effects were not observed between the redox pair of Nom-60-A and Nom-60-C when waste loading is high.

PCT results are interesting and show that:

- (1) The 100% waste loading group glasses have similar normalized sodium releases as the 90% waste loading glasses (except of the heat treated glasses);
- (2) The redox effects on glass durability are minimal in comparison of Nom-A and Nom-60-C or between Nom-60-D and Nom-60-E;
- (3) The heat treatment did decrease the glass durability by 300%, i.e., the heat treated Nom-60-B has a sodium release rate three times as high as the quenched Nom-60-A.

3.3.4 Conclusions

Waste loading for treating Nom-60 waste can be as high as 100% and it can be conveniently melted under DC plasma condition with a viscosity of less than 8 Pa.s at 1425°C. The reducing melting condition, such as that in DC plasma melting, has shown insignificant effects on waste form durability in terms of both TCLP and PCT results. The reducing condition, on the other hand, can enhance melting rates by decreasing viscosity about 20%. The reducing condition is also expected to decrease the volatilities of some volatile elements such as Tc. On the other hand, the data suggest that devitrification of the waste form have had much large effects on glass durability: it increased sodium release by 300% and made the waste form fail TCLP for lead release. This is an important issue facing every high temperature process.

The recommendation for treating Nom-60 wastes with DC plasma is 1) treating the waste directly at 100% waste loading; 2) avoid waste form devitrification by pouring the melted waste form into small gems to quench the melt and minimize devitrification.

4.0 Comparison of Waste Forms Among the Three Wastes

A comparison of the waste form behavior among several attributes gives us the insight on the relationship between waste form performance and their compositions and processing conditions. Table VII is a brief summary of the waste forms developed for the three wastes.

Table VII. Waste Form Comparison

Effects	Metal-R	Pu ²³⁸ Debris	Nom-60
Reducing Melting	<ul style="list-style-type: none">- Reduced durability over 400-700%- Reduced viscosity- Reduced the tendency to devitrify	<ul style="list-style-type: none">- Reduce durability over 300-400%- Reduced viscosity- Reduced the tendency to devitrify	<ul style="list-style-type: none">- <u>No significant effect on durability</u>- Reduced viscosity- Reduced the tendency to devitrify
Devitrification	Increased durability by about 40%	No significant effects	Decreased durability by 200%
Increasing Waste Loading	Slightly decrease durability	Increased durability by 240%	No significant effects

The data in Table VII indicate very different responses for each processing effect. The reducing melting condition can decrease the waste form durability from 300 to 700% for wastes of Metal-R and Pu²³⁸ debris, but has essentially no effects on waste form durability developed for Nom-R waste. The devitrification of the waste forms increased durability for Metal-R waste, exhibited no effect for Pu²³⁸ debris, and substantially decreased durability for Nom-R waste. The increased waste loading also affects waste form behavior differently from decreasing durability for Metal-R waste, increasing durability by 240% for Pu²³⁸ debris, to no effects for Nom-60 waste.

However, these different responses to the processing and composition variations are not random and are understandable from the glass chemistry point of view. The following is a brief summary of such an understanding of the each effect:

Redox effects:

For Metal-R waste, the oxidized (or normal melting) waste form (such as Metal-B) developed 90% spinel type of crystals and 10% Augite type of crystals. The formation of spinel crystals deplete elements such as Fe, Ni, Cr, and Mn from the melt, resulting in an enrichment of silica and alumina in the residual glass. Spinel type crystals are themselves very stable and durable. Therefore, the formation of spinel usually increases the durability of waste form. On the other hand, the formation of Augite type of crystals, $\text{Ca(Fe,Mg)Si}_2\text{O}_6$, usually decreases waste form durability because of the extraction of silica from glassy matrix into the crystals. When the waste form with the same composition was melted under reducing conditions as in the case of Metal-D. The beneficial crystals of spinel type were decreased to 55% while the silica- and alumina-consuming crystals of Augite ($\text{Ca(Fe,Mg)Si}_2\text{O}_6$), Faayalite (Fe_2SiO_4), and albite ($\text{Na(Si}_3\text{Al)O}_8$) - types increased to 45%. This is why we observed substantial decreasing in durability for Metal-R wastes melted under reducing conditions.

For Pu^{238} debris waste, only the beneficial spinel crystals are formed in both oxidized waste form, Lab-1Ga, and the reduced waste form, Lab-1Gb. The durability is, therefore, proportional to the amount of spinel to be formed in the waste form. The oxidized waste form, Lab-1Ga, formed 45% spinel type crystals while the reduced waste form, Lab-1Gb, formed only 5% spinel crystals. This is why the reduced waste form decreased the durability by 300-400% because of the 9 times less spinel in the reduced waste form.

For Nom-60 waste, the oxidized waste form, Nom-60-A, had about 19% spinel and 6% clinopyroxene, $\text{Ca(Ti,Mg,Al)(Si,Al)}_2\text{O}_6$, which consumed silica and alumina. The reduced waste form, Nom-60-C, had about 4% spinel and 1% clinopyroxene. The ratio of the beneficial crystal of spinel to the harmful crystal of clinopyroxene is similar in both oxidized and reduced waste forms. This may explain why the reducing melting conditions has no significant effects on waste form durability.

Devitrification Effects:

For Metal-R waste, the devitrified Metal-B has 90% beneficial spinel crystals and only 10% harmful crystals of Augite type. The benefit from forming spinel outweighed the harmful effects of formation of Augite. The devitrification of Metal-B improved its chemical durability. The glass waste form (non-devitrified) Metal-A is completely amorphous. We observed an increase in waste form durability when the waste form was heat treated to enhance the devitrification.

For Pu²³⁸ debris waste, both the heat treated Lab-1VCa and quenched Lab-1Ga have the same amount of 40% spinel formation and, therefore, they have similar chemical durability.

For Nom-60 waste, the heat treated Nom-60-B formed 70% harmful crystals of Augite-type ($\text{Ca}(\text{Fe},\text{Mg})\text{Si}_2\text{O}_6$), Wallastonite (CaSiO_3), and Anorthite-type ($((\text{Ca},\text{Na})(\text{Al},\text{Si})_2\text{Si}_2\text{O}_8)$), which consume silica and alumina, and 30% spinel type crystals. The quenched Nom-60-A, had about 19% spinel and 6% clinopyroxene, $\text{Ca}(\text{Ti},\text{Mg},\text{Al})(\text{Si},\text{Al})_2\text{O}_6$. Therefore, the heat treated Nom-60-B produced a poor durability glassy matrix after the extraction of silica and alumina from the glassy phase during the formation of the large amount of Augite-type, Wallastonite, and Anorthite-type crystals. The quenched sample had more beneficial crystals of spinel than the harmful clinopyroxene. This is why devitrification of Nom-60 waste forms resulted in a 200% decrease in durability.

Waste Loading Effects:

For Metal-R waste, the 100% waste loading Metal-D (heat treated) is about 20% less durable than the 34% waste loading Metal-C (quenched). Metal-D had 45% silica- and alumina-consuming crystals of Augite ($\text{Ca}(\text{Fe},\text{Mg})\text{Si}_2\text{O}_6$), Faayalite (Fe_2SiO_4), and albite ($\text{Na}(\text{Si}_3\text{Al})\text{O}_8$) - types and 55% spinel crystals. The formation of these crystals resulted in the deteriorated glass durability. The 34% waste loading Metal-A is essentially amorphous and its glassy matrix is not deteriorated by crystal formation as in the case of the 100% waste loading Metal-D.

For Pu²³⁸ debris waste, the 100% waste loading Lab-1Ga formed about 40% beneficial spinel type of crystals while the 83% waste loading Lab-83a had only about 10% such crystals. This is why we observed 240% increase in durability when the waste loading increased from 83 to 100%.

For Nom-R waste, similar chemical durability was observed for the 100% waste loading Nom-60-A and 90% waste loading Nom-60-D. It is because both glass maintained similar ratio of spinel type crystals to silica-consuming crystals such as Augite and clinopyroxene.

5.0 References

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Figure 1 Optical micrograph of a cross-section of Metal-B showing diamond shaped spinel type of crystals.

Figure 2. SEM micrograph of a cross-section of Metal-B showing diamond shaped spinel type and needle shaped Augite type crystals in a glassy matrix.

Figure 3. EDS spectra of Metal-B. (A) Spinel type crystals, (b) Glassy matrix, and © Augite type crystals.

(a)

(b)

Figure 4. (a) SEM micrograph of a cross-section of Metal-B showing a large triangular shaped spinel type crystals.
(b) EDS spectrum of the spinel-type crystals in Metal-B.

Figure 5 Optical micrograph of a cross-section of Metal-D showing the triangular shaped spinel type of crystals.

(a)

(b)

Figure 6. (a) SEM micrograph of Metal-D showing large spinel type crystals in the center part of the sample..
(b) EDS spectrum of the spinel-type crystals showing in (a).

Figure 7. SEM micrograph of Metal-D showing diamond shaped spinel type crystals imbedded in an Augite type of crystal matrix.

Figure 8. EDS spectra of Metal-D. (a) Spinel type crystals, and (b) Augite type of crystals.

Figure 9. SEM micrograph of the lower portion of the cross-section of Metal-D showing spinel type crystals imbeded in an Augite type of crystal matrix.

Figure 10. EDS spectra of Metal-D. (a) Augite type of crystals, and (b) Spinel type crystals.

Figure 11 Optical micrograph of the top surface of Metal-C at 500X.

Figure 12. SEM Micrograph of Lab-1VCa showing the crystals aligned in a cross type pattern in a glassy matrix.

Figure 13. EDS spectrum of Lab-1VCa. (a) Spinel type crystals, (b) Glassy matrix.

Figure 14. SEM Micrograph of Lab-1VCb showing the diamond shaped spinel crystals in a glassy matrix.

Figure 15. EDS spectrum of Lab-1VCb. (a) Spinel type crystals, (b) Glassy matrix.

(a)

(b)

(c)

Figure 16. Optical micrographs of the top surfaces of (a) Nom-60-A, (b) Nom-60-B, (c) Nom-60-C.

Figure 17. SEM Micrograph of N-60-B showing triangular shaped spinel type crystals and needle shaped augite type crystals.

Figure 18. EDS spectra of N0m-60-B. (a) Spinel type crystals, (b) Augite type crystals.

Figure 19. (a) SEM Micrograph of Nom-60-B showing the large Si-Ca-Fe phase, (b). EDS spectra of the Si-Ca-Fe phase in Nom-60-B.

(a)

(b)

Figure 20. Optical micrographs of the top surfaces of (a) Nom-60-D, and (b) Nom-60-E.

Figure 21. SEM Micrograph of N-60-D showing square shaped Augite type crystals.

Figure 22 EDS spectrum of N-60-D (a) Augite type crystals, (b) Glassy matrix

Figure 23. (Upper): SEM Micrograph of Nom-60-D showing a triangular shaped spinel type crystal.
(Bottom): EDS spectrum of the triangular crystal in Nom-60-D as a spinel type crystal.

Appendix A: Waste Form Formulation

MWTP 5400: Heterogeneous Debris (Surrogate Formulation)

COMPONENTS	Wt. %	Compound Weight	M.W.	Moles	Elem.	Mass (g)	Factor	Oxide	Mass (g)
BULK:	97.50								
Activated Carbon	5.00	12.0	12.0	1.0	C	5.000	1.000		5.000
Water	14.50	18.0					1.000		0.000
Wood (C ₆ H ₉ O ₄)	10.00	145.0	12.0	6.0	C	4.966	1.000		4.966
PVC	10.00	62.5	12.0	2.0	C	3.840	1.000		3.840
(C ₂ H ₃ Cl)			35.5	1.0	Cl	5.680	1.000		5.680
Neoprene	10.00	88.5	12.0	4.0	C	5.424	1.000		5.424
(C ₄ H ₅ Cl)			35.5	1.0	Cl	4.011	1.000		4.011
Mild Steel	10.00	56.1	55.8	1.000	Fe	9.940	0.699	Fe ₂ O ₃	14.212
1010 & 1020			12.0	0.007	C	0.015	1.000		0.015
			54.9	0.005	Mn	0.049	0.774	MnO	0.063
Glass Beads	10.00	10.0	28.1	0.120	Si	3.366	0.467	SiO ₂	7.202
SLS (window)			23.0	0.045	Na	1.046	0.742	Na ₂ O	1.410
			40.1	0.018	Ca	0.715	0.715	CaO	1.000
			24.3	0.006	Mg	0.145	0.603	MgO	0.240
			27.0	0.001	Al	0.032	0.529	Al ₂ O ₃	0.060
			32.1	0.001	S	0.036	0.416	SO ₃	0.087
CONCRETE	8.00	600.0	40.1	1.476	Ca	0.789	0.715	CaO	1.104
(cured, crushed)			28.1	7.013	Si	2.627	0.467	SiO ₂	5.621
			27.0	1.322	Al	0.476	0.529	Al ₂ O ₃	0.899
RATIO			55.8	0.033	Fe	0.024	0.699	Fe ₂ O ₃	0.035
1: Type I Portland			23.0	0.343	Na	0.105	0.742	Na ₂ O	0.142
2: Sand			39.1	0.216	K	0.112	0.830	K ₂ O	0.135
3: Gravel			24.3	0.074	Mg	0.024	0.603	MgO	0.040
0.5: Water			32.1	0.023	S	0.010	0.416	SO ₃	0.024
Alumina Crucibles	10.00	102.00	27.00	2.00	Al	5.294	0.529	Al ₂ O ₃	10.003
Diatomaceous Earth	10.00	60.10	28.10	1.00	Si	4.676	0.467	SiO ₂	10.003
RCRA METALS:	1.00								
CrCl ₃ *6H ₂ O	0.25	266.5	52.0	1.0	Cr	0.049	0.684	Cr ₂ O ₃	0.071
			35.5	3.0	Cl	0.100	1.000		0.100
NiCl ₂ *6H ₂ O	0.25	237.7	58.7	1.0	Ni	0.062	0.786	NiO	0.079
			35.5	2.0	Cl	0.075	1.000		0.075
PbCl ₂	0.25	278.2	207.2	1.0	Pb	0.186	0.928	PbO	0.201
			35.5	2.0	Cl	0.064	1.000		0.064
CdCl ₂ *2.5H ₂ O	0.25	228.4	112.4	1.0	Cd	0.123	0.875	CdO	0.141
			35.5	2.0	Cl	0.078	1.000		0.078
RCRA ORGANICS:	1.00								
Napthalene (C ₁₀ H ₈)	0.40	128.0	12.0	10.0	C	0.375	1.000		0.375
1,2-Dichloro-benzene	0.30	502.0	12.0	6.0	C	0.043	1.000		0.043
(C ₆ H ₄ Cl ₂)			35.5	12.0	Cl	0.255	1.000		0.255
Chlorobenzene	0.30	112.5	12.0	6.0	C	0.192	1.000		0.192
(C ₆ H ₅ Cl)			35.5	1.0	Cl	0.095	1.000		0.095
SURROGATES:	0.50								
CeCl ₃	0.25	246.6	140.1	1.0	Ce	0.142	0.814	CeO ₂	0.174
			35.5	3	Cl	0.108	1.000		0.108
CsCl	0.25	168.4	132.9	1.0	Cs	0.197	0.943	Cs ₂ O	0.209
			35.5	1.0	Cl	0.053	1.000		0.053
TOTAL:	100.00								

Appendix A: Waste Form Formulation

Concrete Composition

	Ratio (g)	Composition	Mass	M.W.	Moles	Oxide	Moles
Portland Cement Type I	100.00	3CaO·SiO ₂	46.0	228.4	0.20	CaO	1.153
		2CaO·SiO ₂	28.0	172.3	0.16	SiO ₂	0.364
		3CaO·Al ₂ O ₃	11.3	270.3	0.04	Al ₂ O ₃	0.058
		4CaO·Al ₂ O ₃	8.0	486.0	0.02	Fe ₂ O ₃	0.016
		•Fe ₂ O ₃					
		CaSO ₄	3.2	136.2	0.02	MgO	0.074
		MgO	3.0	40.3	0.07	SO ₃	0.023
		CaO	0.5	56.1	0.01		
Sand	200.00	SiO ₂	200	60.1	3.33	SiO ₂	3.328
Gravel	300.00	SiO ₂	60	60.1	1.00	CaO	0.32
		2NaAlSi ₃ O ₈	90	524.6	0.17	SiO ₂	3.321
		CaAl ₂ Si ₂ O ₈	90	278.3	0.32	Al ₂ O ₃	0.603
		2KAlSi ₃ O ₈	60	556.8	0.11	Na ₂ O	0.172
						K ₂ O	0.108
Concrete	600.00	Mass (g)	Oxide	Moles	M.W.	Mass	Wt. %
			CaO	1.476	56.1	82.819	13.803
			SiO ₂	7.013	60.1	421.465	70.244
			Al ₂ O ₃	0.661	102.0	67.420	11.237
			Fe ₂ O ₃	0.016	159.6	2.627	0.438
			Na ₂ O	0.172	62.0	10.637	1.773
			K ₂ O	0.108	94.2	10.151	1.692

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Appendix A: Waste Form Formulations

MWTP 5400: Waste Form (Surrogate Formulation)

Oxide	Mass	Mass Frac.	Lab-1 M Frac	Lab-1Ga Wt%	Lab-1Gb* Wt%	Lab-1VCa Wt%	Lab-1VCb* Wt%	x .83	Additions	Lab-.83Ga Wt%	Lab-.83Gb* Wt%
SiO2	28.439	0.3191	0.4820	48.2041	48.2041	48.2041	48.2041	39.9537	4.2900	44.244	44.244
Al2O3	10.959	0.1230	0.1858	18.5753	18.5753	18.5753	18.5753	15.3961		15.396	15.396
Fe2O3	14.250	0.1599	0.2415	24.1543	24.1543	24.1543	24.1543	20.0202		20.020	20.020
Na2O	1.552	0.0174	0.0263	2.6303	2.6303	2.6303	2.6303	2.1801	12.8200	15.000	15.000
K2O	0.135	0.0015	0.0023	0.2294	0.2294	0.2294	0.2294	0.1901		0.190	0.190
MgO	0.280	0.0031	0.0047	0.4746	0.4746	0.4746	0.4746	0.3934		0.393	0.393
CaO	2.104	0.0236	0.0357	3.5667	3.5667	3.5667	3.5667	2.9562		2.956	2.956
MnO	0.063	0.0007	0.0011	0.1070	0.1070	0.1070	0.1070	0.0887		0.089	0.089
NiO	0.079	0.0009	0.0013	0.1332	0.1332	0.1332	0.1332	0.1104		0.110	0.110
PbO	0.201	0.0023	0.0034	0.3400	0.3400	0.3400	0.3400	0.2818		0.282	0.282
CdO	0.141	0.0016	0.0024	0.2382	0.2382	0.2382	0.2382	0.1974		0.197	0.197
Cr2O3	0.071	0.0008	0.0012	0.1208	0.1208	0.1208	0.1208	0.1002		0.100	0.100
CeO2	0.174	0.0020	0.0030	0.2957	0.2957	0.2957	0.2957	0.2451		0.245	0.245
Cs2O	0.209	0.0023	0.0035	0.3546	0.3546	0.3546	0.3546	0.2939		0.294	0.294
S	0.046	0.0005	0.0008	0.0782	0.0782	0.0782	0.0782	0.0648		0.065	0.065
Cl	10.563	0.1185	0.0050	0.4975	0.4975	0.4975	0.4975	0.4124		0.412	0.412
C	19.854	0.2228	-	-	-	-	-	-	-	-	-
Total	89.12	1.00	1.00	100.00	100.00	100.00	100.00	82.88	17.11	99.99	99.99

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix A: Waste Form Formulation

Metals -R Waste Form Formulation

Elem.	Elem. Wt%	Oxide	Waste Mass	Metal-B wt%	Metal-D* wt%	Additives	Waste	Metal-A wt. %	Metal-C* wt. %
Ag	6.6E-01	Ag ₂ O	0.711	0.584	0.584		0.199	0.199	0.199
Al	3.4E+00	Al ₂ O ₃	6.347	5.212	5.212	4.227	1.773	6.000	6.000
As	5.3E-03	As ₂ O ₃	0.007	0.006	0.006		0.002	0.002	0.002
B	0.0E+00	B ₂ O ₃				5.917	0.000	5.917	5.917
Ca	5.8E+00	CaO	8.106	6.657	6.657		2.264	2.264	2.264
Cd	1.0E-02	CdO	0.011	0.009	0.009		0.003	0.003	0.003
Cl	2.0E-02	Cl	0.020	0.016	0.016		0.006	0.006	0.006
Cr	3.5E-02	Cr ₂ O ₃	0.051	0.042	0.042		0.014	0.014	0.014
Cs	2.0E-01	Cs ₂ O	0.215	0.177	0.177		0.060	0.060	0.060
Cu	2.6E-03	CuO	0.003	0.003	0.003		0.001	0.001	0.001
Fe	5.0E+01	Fe ₂ O ₃	71.598	58.801	58.801	2.940	20.000	20.000	20.000
Mg	7.9E-01	MgO	1.317	1.081	1.081		0.368	0.368	0.368
Mn	3.3E-02	MnO	0.042	0.034	0.034		0.012	0.012	0.012
Na	1.1E+00	Na ₂ O	1.487	1.221	1.221	14.585	0.415	15.000	15.000
Ni	1.0E-02	NiO	0.013	0.010	0.010		0.004	0.004	0.004
O	2.4E+01	O							
P	7.1E-02	P ₂ O ₅	0.162	0.133	0.133		0.045	0.045	0.045
Pb	1.8E-03	PbO	0.002	0.002	0.002		0.001	0.001	0.001
Si	1.5E+01	SiO ₂	31.296	25.703	25.703	41.258	8.742	50.000	50.000
Ti	2.2E-01	TiO ₂	0.368	0.302	0.302		0.103	0.103	0.103
Zr	5.3E-03	ZrO ₂	0.007	0.006	0.006		0.002	0.002	0.002
Total	101.02	Total	121.76	100.00	100.00	65.99	34.01	100.00	100.00

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix A: Waste Form Formulation

Nominal-60 Waste Form Formulation

Elem.	Elem. Wt%	Oxide	Waste Mass	Nom-60-A wt%	Nom-60-B wt%	Nom-60-C* wt%	Additives	Waste	Nom-60-D wt%	Nom-60-E* wt%
Ag	8.9E-05	Ag ₂ O	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Al	4.0E+00	Al ₂ O ₃	7.533	8.728	8.728	8.728		8.728	7.803	7.803
As	4.1E-04	As ₂ O ₃	0.001	0.001	0.001	0.001		0.001	0.001	0.001
Ba	5.0E-02	BaO	0.056	0.065	0.065	0.065		0.065	0.058	0.058
C	7.1E+00	C	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Ca	1.3E+01	CaO	18.658	21.618	21.618	21.618		21.618	19.326	19.326
Cd	1.4E-04	CdO	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Ce	3.0E-01	CeO ₂	0.369	0.428	0.428	0.428		0.428	0.382	0.382
Cl	2.2E+00	Cl	2.214	2.566	2.566	2.566		2.566	2.294	2.294
Cr	7.0E-01	Cr ₂ O ₃	1.025	1.188	1.188	1.188		1.188	1.062	1.062
Cs	5.5E-04	Cs ₂ O	0.001	0.001	0.001	0.001		0.001	0.001	0.001
Cu	1.9E-01	CuO	0.238	0.276	0.276	0.276		0.276	0.247	0.247
Fe	1.2E+01	Fe ₂ O ₃	17.440	20.207	20.207	20.207		20.207	18.064	18.064
H	1.7E+00	H	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Hf	3.1E-04	HfO ₂	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Hg	6.9E-06	Hg	0.000	0.000	0.000	0.000		0.000	0.000	0.000
K	1.3E+00	K ₂ O	1.593	1.846	1.846	1.846		1.846	1.650	1.650
Mg	9.1E-01	MgO	1.512	1.752	1.752	1.752		1.752	1.566	1.566
Mn	7.0E-02	MnO	0.091	0.105	0.105	0.105		0.105	0.094	0.094
Mo	1.0E-02	MoO ₃	0.015	0.017	0.017	0.017		0.017	0.016	0.016
N	2.3E-01	N	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Na	9.4E-01	Na ₂ O	1.270	1.471	1.471	1.471	5.240	6.711	6.000	6.000
Ni	3.3E-01	NiO	0.421	0.488	0.488	0.488		0.488	0.436	0.436
O	3.8E+01	O	0.000	0.000	0.000	0.000		0.000	0.000	0.000
P	4.0E-02	P ₂ O ₅	0.092	0.106	0.106	0.106		0.106	0.095	0.095
Pb	6.0E-02	PbO	0.065	0.075	0.075	0.075		0.075	0.067	0.067
S	3.0E-02	S	0.060	0.070	0.070	0.070		0.070	0.062	0.062
Se	8.9E-05	SeO ₂	0.000	0.000	0.000	0.000		0.000	0.000	0.000
Si	1.5E+01	SiO ₂	32.903	38.124	38.124	38.124	6.620	44.744	40.000	40.000
Ti	2.0E-01	TiO ₂	0.334	0.387	0.387	0.387		0.387	0.346	0.346
Zn	6.6E-03	ZnO	0.008	0.010	0.010	0.010		0.010	0.009	0.009
Zr	3.0E-01	ZrO ₂	0.406	0.471	0.471	0.471		0.471	0.421	0.421
Total	100.00	Total	86.31	100.00	100.00	100.00	11.86	111.86	100.00	100.00

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995
LLW Low Temp. Study

Metal-A,C		PCT Normalized Release (g/m ² /7day)	
Oxide	Mass Frac.	Si	0.145
SiO ₂	0.5000	B	0.322
B ₂ O ₃	0.0592	Na	0.334
Na ₂ O	0.1500	Li	0.285
K ₂ O		pH	10.36
Li ₂ O		MCC - 1 Normalized Release (g/m ² /28day)	
CaO	0.0226	Si	9.948
MgO	0.0037	B	11.083
Fe ₂ O ₃	0.2000	Na	10.864
Al ₂ O ₃	0.0600	Li	11.075
ZrO ₂	0.0000	pH	9.55
P ₂ O ₅	0.0005	Liquidus Temperature	
Bi ₂ O ₃		Spinel	1137 °C
TiO ₂	0.0010	Clino.	929 °C
Others	0.0030	Zircon	846 °C
SUM	1.0000	Glass Density	
Temp (C)	1280	Tg	501 °C
TR = TG/TL 0.5489		Viscosity	
		Arrhenius	
		A	-9.977
		B	18435.518
		1280	6.6 Pa.s
		Fulcher	
		A	-8.970
		B	11620.571
		To	165.808
		1280	4.3 Pa.s
		Electrical Conductivity	
		A	8.581
		B	-8143.399
		1280	28.15 S/m

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995

LLW Low Temp. Study

Metal-B,D	Mass Frac.	PCT Normalized Release (g/m ² /7day)
Oxide	0.2570	Si 0.012
SiO2		B 0.010
B2O3	0.0122	Na 0.009
Na2O		Li 0.008
K2O		pH 9.16
Li2O	0.0666	
CaO	0.0108	MCC - 1 Normalized Release (g/m ² /28day)
MgO	0.5880	Si 2.412
Fe2O3	0.0521	B 2.163
Al2O3	0.0001	Na 2.372
ZrO2	0.0013	Li 2.752
P2O5		pH 9.45
Bi2O3	0.0030	
TiO2	0.0088	Liquidus Temperature
Others	1.0000	Spinel 1810 °C
SUM		Clino. 1336 °C
	1450	Zircon 1208 °C
Temp (C)		
		Glass Densit 2.52 g/cc
		Tg 493 °C
	0.3676	Viscosity
TR = TG/TL		Arrhenius
		A -8.886
		B 15581.536
		1450 1.2 Pa.s
		Fulcher
		A -10.333
		B 12437.228
		To 144.585
		1450 0.4 Pa.s
		Electrical Conductivity
		A 9.666
		B -10853.227
		1450 29.00 S/m

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995

LLW Low Temp. Study

Lab-1	
Oxide	Mass Frac.
SiO2	0.4815
B2O3	0.0000
Na2O	0.0263
K2O	0.0023
Li2O	
CaO	0.0356
MgO	0.0047
Fe2O3	0.2413
Al2O3	0.1855
ZrO2	
P2O5	
Bi2O3	
TiO2	
Others	0.0228
SUM	1.0000
Temp (C)	1450

$$TR = TG/TL \quad 0.4783$$

PCT Normalized Release (g/m²/7day)

Si	0.004
B	0.001
Na	0.001
Li	0.001
pH	8.56

MCC - 1 Normalized Release (g/m²/28day)

Si	0.724
B	0.349
Na	0.385
Li	0.408
pH	7.97

Liquidus Temperature

Spinel	1428 °C
Clino.	1161 °C
Zircon	987 °C

Glass Density 2.37 g/cc

Tg 540 °C

Viscosity

Arrhenius

A	-9.204
B	21168.601
1450	21.8 Pa.s

Fulcher

A	-7.942
B	13500.529
To	139.766
1450	10.6 Pa.s

Electrical Conductivity

A	8.775
B	-10043.990
1450	19.03 S/m

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995

LLW Low Temp. Study

Lab-83

Oxide	Mass Frac.
SiO2	0.4420
B2O3	
Na2O	0.1500
K2O	0.0019
Li2O	
CaO	0.0295
MgO	0.0039
Fe2O3	0.2000
Al2O3	0.1538
ZrO2	
P2O5	
Bi2O3	
TiO2	
Others	0.0189
SUM	1.0000

Temp (C) 1375

TR = TG/TL 0.5096

PCT Normalized Release (g/m²/7day)

Si	0.034
B	0.018
Na	0.023
Li	0.023
pH	10.48

MCC - 1 Normalized Release (g/m²/28day)

Si	2.461
B	1.826
Na	1.838
Li	1.983
pH	9.20

Liquidus Temperature

Spinel	1226 °C
Clino.	1008 °C
Zircon	860 °C

Glass Density 2.44 g/cc
Tg 491 °C

Viscosity
Arrhenius

A	-9.336
B	18614.237
1375	7.1 Pa.s

Fulcher

A	-6.778
B	10352.937
To	173.692
1375	6.3 Pa.s

Electrical Conductivity

A	8.397
B	-7857.597
1375	37.71 S/m

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995

LLW Low Temp. Study

Nom-60-A,B,C	
Oxide	Mass Frac.
SiO ₂	0.3812
B ₂ O ₃	0.0000
Na ₂ O	0.0147
K ₂ O	0.0185
Li ₂ O	
CaO	0.2162
MgO	0.0175
Fe ₂ O ₃	0.2021
Al ₂ O ₃	0.0873
ZrO ₂	0.0047
P ₂ O ₅	0.0011
Bi ₂ O ₃	
TiO ₂	0.0039
Others	0.0529
SUM	1.0000
Temp (C)	1425

$$TR = TG/TL \quad 0.4882$$

PCT Normalized Release (g/m²/7day)

Si	0.010
B	0.003
Na	0.010
Li	0.008
pH	10.49

MCC - 1 Normalized Release (g/m²/28day)

Si	1.753
B	1.740
Na	1.770
Li	2.317
pH	9.31

Liquidus Temperature

Spinel	1397 °C
Clino.	1211 °C
Zircon	978 °C

Glass Density 2.66 g/cc

T_g 542 °C

Viscosity

Arrhenius

A	-12.514
B	21150.279
1425	0.9 Pa.s

Fulcher

A	-5.780
B	6584.670
To	328.541
1425	1.3 Pa.s

Electrical Conductivity

A	10.245
B	-12103.211
1425	22.60 S/m

Appendix B: CVS Model Predictions

Glass Property Estimation, based on CVS Model with slight additions of other coefficients, May 5, 1995

LLW Low Temp. Study

Nom-60-D,E	
Oxide	Mass Frac.
SiO2	0.4000
B2O3	
Na2O	0.0600
K2O	0.0165
Li2O	
CaO	0.1933
MgO	0.0157
Fe2O3	0.1806
Al2O3	0.0780
ZrO2	0.0042
P2O5	0.0010
Bi2O3	
TiO2	0.0035
Others	0.0473
SUM	1.0000
Temp (C)	1300

$$TR = TG/TL \quad 0.5066$$

PCT Normalized Release (g/m²/7day)

Si	0.023
B	0.010
Na	0.032
Li	0.022
pH	10.97

MCC - 1 Normalized Release (g/m²/28day)

Si	2.705
B	2.905
Na	2.880
Li	3.672
pH	9.59

Liquidus Temperature

Spinel	1307
Clino.	1141 °C
Zircon	922 °C
	°C

Glass Density

Tg	2.64
	528 g/cc
	°C

Viscosity

Arrhenius

A	-12.294
B	20539.825
1300	2.1

Fulcher Pa.s

A	-5.699
B	6417.534
To	318.156
1300	2.3

Pa.s

Electrical Conductivity

A	9.923
B	-11096.477
1300	17.63

S/m

Appendix C: ICP Analysis of Na and K Fused Samples

Metals-R Waste Form Chemical Analysis

Oxide	Metal-B wt%		Metal-D* wt%		Metal-A wt. %		Metal-C* wt. %	
	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed
Ag ₂ O	0.584	0.526	0.584	0.327	0.199	0.182	0.199	0.058
Al ₂ O ₃	5.212	5.125	5.212	6.148	6.000	5.636	6.000	5.552
As ₂ O ₃	0.006	-	0.006	-	0.002	-	0.002	-
B ₂ O ₃					5.917	5.475	5.917	5.412
CaO	6.657	6.688	6.657	6.718	2.264	2.190	2.264	2.199
CdO	0.009	0.014	0.009	0.014	0.003	0.007	0.003	
Cl	0.016	-	0.016	-	0.006	-	0.006	-
Cr ₂ O ₃	0.042	0.061	0.042	0.060	0.014		0.014	
Cs ₂ O	0.177	-	0.177	-	0.060	-	0.060	-
CuO	0.003	0.011	0.003		0.001	0.008	0.001	
Fe ₂ O ₃	58.801	53.515	58.801	55.054	20.000	18.784	20.000	18.342
MgO	1.081	1.016	1.081	0.943	0.368	0.359	0.368	0.370
MnO	0.034	-	0.034	-	0.012	0.027	0.012	-
Na ₂ O	1.221	1.310	1.221	1.263	15.000	13.889	15.000	13.990
NiO	0.010	0.044	0.010	0.046	0.004	0.022	0.004	
P ₂ O ₅	0.133	0.223	0.133	0.269	0.045		0.045	
PbO	0.002		0.002		0.001		0.001	
SiO ₂	25.703	25.358	25.703	25.427	50.000	45.597	50.000	45.321
TiO ₂	0.302	0.303	0.302	0.283	0.103	0.103	0.103	0.100
ZrO ₂	0.006	0.033	0.006	0.022	0.002	0.077	0.002	0.050
Total	100.00	94.23	100.00	96.57	100.00	92.36	100.00	91.39

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix C: ICP Analysis of Na and K Fused Samples

Nominal-60 Waste Form Chemical Analysis

Oxide	Nom-60-A wt%		Nom-60-B wt%		Nom-60-C* wt%		Nom-60-D wt.%		Nom-60-E* wt.%	
	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed
Ag ₂ O							0.000		0.000	
Al ₂ O ₃	8.728	8.523	8.728	7.753	8.728	9.081	7.803	7.829	7.803	7.420
As ₂ O ₃		-		-		-	0.001	-	0.001	0.072
BaO	0.065	0.074	0.065	0.071	0.065	0.074	0.058	0.070	0.058	
CaO	21.618	21.260	21.618	20.368	21.618	20.604	19.326	19.966	19.326	20.489
CdO			0.000		0.000		0.000		0.000	
CeO ₂	0.428	0.402	0.428	0.365	0.428	0.408	0.382	0.360	0.382	0.342
Cl	2.566	-	2.566		2.566		2.294		2.294	
Cr ₂ O ₃	1.188	1.235	1.188	1.530	1.188	1.030	1.062	1.040	1.062	1.098
Cs ₂ O		-	0.001		0.001		0.001		0.001	
CuO	0.276	0.143	0.276	0.171	0.276	0.133	0.247	0.202	0.247	0.191
Fe ₂ O ₃	20.207	19.811	20.207	22.566	20.207	19.290	18.064	19.144	18.064	19.337
HfO ₂			0.000		0.000		0.000		0.000	
K ₂ O	1.846	1.539	1.846	0.688	1.846	0.780	1.650	1.972	1.650	1.417
MgO	1.752	1.518	1.752	1.667	1.752	1.456	1.566	1.477	1.566	1.517
MnO	0.105	-	0.105	0.098	0.105	0.152	0.094	0.110	0.094	0.114
MoO ₃	0.017	0.030	0.017	0.025	0.017	0.028	0.016	0.030	0.016	0.029
Na ₂ O	1.471	1.762	1.471	1.723	1.471	1.874	6.000	6.377	6.000	5.162
NiO	0.488	0.512	0.488	0.637	0.488	0.437	0.436	0.485	0.436	0.453
P ₂ O ₅	0.106		0.106	0.223	0.106	0.206	0.095	0.020	0.095	0.198
PbO	0.075		0.075		0.075		0.067	0.150	0.067	0.132
S	0.070		0.070		0.070		0.062	0.010	0.062	
SeO ₂		-					0.000		0.000	
SiO ₂	38.124	37.125	38.124	39.312	38.124	42.663	40.000	39.971	40.000	42.889
TiO ₂	0.387	0.396	0.387	0.360	0.387	0.449	0.346	0.388	0.346	0.401
ZnO	0.010		0.010		0.010		0.009		0.009	
ZrO ₂	0.471	0.490	0.471	0.438	0.471	0.488	0.421	0.449	0.421	0.372
Total	100.00	94.82	100.00	98.00	100.00	99.15	100.00	100.05	100.00	101.63

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix C: ICP Analysis of Na and K Fused Samples

MWTP 5400 Waste Form Chemical Analysis

Oxide	Lab-1Ga		Lab-1Gb*		Lab-1VCa		Lab-1VCb*		Lab-.83a		Lab-.83b*	
	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed	Formulated	Analyzed
Al ₂ O ₃	18.575	17.641	18.575	17.878	18.575	17.325	18.575	17.973	15.396	14.394	15.396	14.500
CaO	3.567	3.512	3.567	3.481	3.567	3.492	3.567	3.420	2.956	2.839	2.956	2.922
CdO	0.238	0.155	0.238	0.101	0.238	0.160	0.238	0.102	0.197	0.180	0.197	0.104
CeO ₂	0.296	0.294	0.296	0.252	0.296	0.277	0.296	0.241	0.245	2.460	0.245	0.198
Cl	0.498	-	0.498	-	0.498	-	0.498	-	0.412	-	0.412	-
Cr ₂ O ₃	0.121	0.129	0.121	0.129	0.121	0.140	0.121	0.128	0.100	0.107	0.100	0.106
Cs ₂ O	0.355	-	0.355	-	0.355	-	0.355	-	0.294	-	0.294	-
Fe ₂ O ₃	24.154	23.060	24.154	23.323	24.154	22.702	24.154	22.310	20.020	18.983	20.020	18.838
K ₂ O	0.229	0.809	0.229	0.627	0.229	0.709	0.229	0.621	0.190	0.631	0.190	0.986
MgO	0.475	0.454	0.475	0.455	0.475	0.449	0.475	0.444	0.393	0.372	0.393	0.390
MnO	0.107	-	0.107	-	0.107	-	0.107	-	0.089	-	0.089	-
Na ₂ O	2.630	2.530	2.630	2.609	2.630	2.417	2.630	2.551	15.000	13.690	15.000	13.462
NiO	0.133	0.149	0.133	0.148	0.133	0.147	0.133	0.136	0.110	0.122	0.110	0.131
PbO	0.340	0.322	0.340	0.320	0.340	0.292	0.340	0.271	0.282	0.268	0.282	0.258
S	0.078	-	0.078	-	0.078	-	0.078	-	0.065	-	0.065	-
SiO ₂	48.204	45.774	48.204	45.421	48.204	44.626	48.204	45.081	44.244	41.068	44.244	43.334
Total	100.00	94.83	100.00	94.74	100.00	92.74	100.00	93.28	99.99	95.11	99.99	95.23

* 1Wt% carbon powder addition to ground glass to promote reducing conditions.

Appendix D: ICP Results for PCT Test Elemental Release

7 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	M-A1		M-A2		Average g/m ²	M-C1		M-C2		Average g/m ²
			μg/ml	g/m ²	μg/ml	g/m ²		μg/ml	g/m ²	μg/ml	g/m ²	
Na2O	Na	15.000	75.63	0.34	76.15	0.34	0.341	301.44	1.35	311.45	1.40	1.377
B2O3	B	5.917	9.79	0.27	9.80	0.27	0.266	29.39	0.80	31.51	0.86	0.829
SiO2	Si	50.000	77.10	0.16	76.13	0.16	0.164	151.70	0.32	182.11	0.39	0.357
Al2O3	Al	6.000	7.67	0.12	7.54	0.12	0.120	13.94	0.22	14.32	0.23	0.222
CaO	Ca	2.264	0.27	0.01	0.28	0.01	0.008	2.13	0.07	2.20	0.07	0.067
P2O5	P	0.045	0.12	0.30	0.19	0.48	0.393	0.37	0.95	0.35	0.90	0.922
Cl	Cl	0.006	-	-	-	-	-	-	-	-	-	-
Fe2O3	Fe	20.000	2.88	0.01	2.64	0.01	0.010	38.94	0.14	40.92	0.15	0.143
MgO	Mg	0.368	0.10	0.02	0.09	0.02	0.021	1.14	0.26	1.15	0.26	0.258
TiO2	Ti	0.103	0.00	0.00	0.00	0.00	0.000	0.09	0.09	0.09	0.09	0.088
MnO	Mn	0.012	0.00	0.00	0.00	0.00	0.000	0.08	0.43	0.08	0.43	0.430
ZrO2	Zr	0.002		0.00		0.00	0.000		0.00	0.01	0.33	0.165
Cs2O	Cs	0.060	-	-	-	-	-	-	-	-	-	-
CuO	Cu	0.001	0.01	0.68		0.00	0.338	0.04	2.84	0.04	2.84	2.838
As2O3	As	0.002	-	-	-	-	-	-	-	-	-	-
Ag2O	Ag	0.199	0.02	0.01	0.02	0.01	0.005	0.03	0.01	0.04	0.01	0.009
CdO	Cd	0.003		0.00		0.00	0.000	0.01	0.25	0.02	0.27	0.260
Cr2O3	Cr	0.014		0.00		0.00	0.000	0.02	0.10	0.02	0.10	0.102
NiO	Ni	0.004		0.00		0.00	0.000	0.05	0.89	0.05	0.89	0.895
PbO	Pb	0.001		0.00		0.00	0.000		0.00		0.00	0.000
pH				10.33		10.29	10.310		11.29		11.28	11.285

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

7 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	M-B1		M-B2		Average g/m ²	M-D1		M-D2		Average g/m ²
			μg/ml	g/m ²	μg/ml	g/m ²		μg/ml	g/m ²	μg/ml	g/m ²	
Na2O	Na	1.221	4.30	0.24	4.28	0.24	0.237	31.29	1.73	30.81	1.70	1.714
SiO2	Si	25.703	20.14	0.08	20.09	0.08	0.084	26.61	0.11	25.93	0.11	0.109
Al2O3	Al	5.212	3.19	0.06	3.18	0.06	0.058	9.60	0.17	9.22	0.17	0.171
CaO	Ca	6.657	5.48	0.06	5.51	0.06	0.058	1.18	0.01	1.21	0.01	0.013
P2O5	P	0.133		0.00		0.00	0.000	0.26	0.22	0.25	0.22	0.220
Cl	Cl	0.016	-	-	-	-	-	-	-	-	-	-
Fe2O3	Fe	58.801	0.04	0.00	0.03	0.00	0.000	0.31	0.00	0.27	0.00	0.000
MgO	Mg	1.081	0.11	0.01	0.11	0.01	0.008		0.00		0.00	0.000
TiO2	Ti	0.302	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
MnO	Mn	0.034	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
ZrO2	Zr	0.006		0.00		0.00	0.000		0.00		0.00	0.000
Cs2O	Cs	0.177	-	-	-	-	-	-	-	-	-	-
CuO	Cu	0.003		0.00		0.00	0.000		0.00		0.00	0.000
As2O3	As	0.006	-	-	-	-	-	-	-	-	-	-
Ag2O	Ag	0.584	0.01	0.00	0.01	0.00	0.001	0.01	0.00	0.01	0.00	0.001
CdO	Cd	0.009		0.00		0.00	0.000		0.00		0.00	0.000
Cr2O3	Cr	0.042		0.00		0.00	0.000		0.00		0.00	0.000
NiO	Ni	0.010		0.00		0.00	0.000		0.00		0.00	0.000
PbO	Pb	0.002		0.00		0.00	0.000		0.00		0.00	0.000
pH				9.28		9.33	9.305		10.35		10.26	10.305

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

8 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	Nom-60-A1		Nom-60-A2		Average g/m ²	Nom-60-B1		Nom-60-B2		Average g/m ²	Nom-60-C1		Nom-60-C2		Average g/m ²
			µg/ml	g/m ²	µg/ml	g/m ²		µg/ml	g/m ²	µg/ml	g/m ²		µg/ml	g/m ²	µg/ml	g/m ²	
Na ₂ O	Na	1.471	2.82	0.13	2.80	0.13	0.129	8.52	0.39	8.57	0.39	0.392	2.12	0.10	2.11	0.10	0.097
SiO ₂	Si	38.124	15.29	0.04	15.34	0.04	0.043	15.89	0.04	15.66	0.04	0.044	15.09	0.04	15.13	0.04	0.042
Al ₂ O ₃	Al	8.728	3.49	0.04	3.50	0.04	0.038	3.37	0.04	3.30	0.04	0.036	2.44	0.03	2.45	0.03	0.026
CaO	Ca	21.618	12.79	0.04	12.72	0.04	0.041	10.07	0.03	9.79	0.03	0.032	12.03	0.04	12.04	0.04	0.039
P ₂ O ₅	P	0.106		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
K ₂ O	K	1.846	2.84	0.09	2.6	0.08	0.089	5.33	0.17	5.29	0.17	0.173	2.33	0.08	2.52	0.08	0.079
Cl	Cl	2.566	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
S	S	0.070	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
HfO ₂	Hf	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CeO ₂	Ce	0.428	0.05	0.01		0.00	0.004		0.00		0.00	0.000		0.00		0.00	0.000
MoO ₃	Mo	0.017	0.02	0.09	0.02	0.09	0.086	0.57	2.45	0.57	2.45	2.454	0.11	0.47	0.11	0.47	0.474
ZnO	Zn	0.010		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
Fe ₂ O ₃	Fe	20.207	0.03	0.00	0.03	0.00	0.000	0.02	0.00	0.02	0.00	0.000	0.03	0.00	0.03	0.00	0.000
MgO	Mg	1.752		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
TiO ₂	Ti	0.387	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
MnO	Mn	0.105	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
ZrO ₂	Zr	0.471		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
Ce ₂ O	Ce	0.001	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CuO	Cu	0.276		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
Ag ₂ O	Ag	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
As ₂ O ₃	As		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BaO	Ba	0.065	0.02	0.02	0.02	0.02	0.017	0.09	0.08	0.08	0.07	0.073	0.01	0.01	0.01	0.01	0.009
CdO	Cd	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cr ₂ O ₃	Cr	1.188		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
NiO	Ni	0.488		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
PbO	Pb	0.075		0.00		0.00	0.000		0.00		0.00	0.000		0.00		0.00	0.000
SeO ₂	Se	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
pH				9.90		9.43	9.665		9.99		9.85	9.920		10.00		10.02	10.010

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

8 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	Nom-60-D1		Nom-60-D2		Average	Nom-60-E1		Nom-60-E2		Average
			$\mu\text{g/ml}$	g/m^2	$\mu\text{g/ml}$	g/m^2	g/m^2	$\mu\text{g/ml}$	g/m^2	$\mu\text{g/ml}$	g/m^2	g/m^2
Na ₂ O	Na	6.000	11.11	0.12	10.78	0.12	0.123	10.58	0.12	10.31	0.12	0.117
SiO ₂	Si	40.000	16.25	0.04	15.59	0.04	0.043	14.55	0.04	14.47	0.04	0.039
Al ₂ O ₃	Al	7.803	3.16	0.04	3.09	0.04	0.038	2.40	0.03	2.39	0.03	0.029
CaO	Ca	19.326	10.91	0.04	10.57	0.04	0.039	11.57	0.04	11.52	0.04	0.042
P ₂ O ₅	P	0.095		0.00		0.00	0.000		0.00		0.00	0.000
K ₂ O	K	1.650	2.60	0.09	2.43	0.09	0.092	2.63	0.10	2.58	0.09	0.095
Cl	Cl	2.294	-	-	-	-	-	-	-	-	-	-
S	S	0.062	-	-	-	-	-	-	-	-	-	-
HfO ₂	Hf	0.000	-	-	-	-	-	-	-	-	-	-
CeO ₂	Ce	0.383		0.00		0.00	0.000		0.00		0.00	0.000
MoO ₃	Mo	0.016	0.02	0.10	0.02	0.10	0.096	0.01	0.05	0.01	0.05	0.048
ZnO	Zn	0.009		0.00		0.00	0.000		0.00		0.00	0.000
Fe ₂ O ₃	Fe	18.064	0.03	0.00	0.03	0.00	0.000	0.03	0.00	0.04	0.00	0.000
MgO	Mg	1.566		0.00		0.00	0.000		0.00		0.00	0.000
TiO ₂	Ti	0.346	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
MnO	Mn	0.094	0.00	0.00	0.00	0.00	0.000	0.00	0.00	0.00	0.00	0.000
ZrO ₂	Zr	0.421		0.00		0.00	0.000		0.00		0.00	0.000
Cs ₂ O	Cs	0.001	-	-	-	-	-	-	-	-	-	-
CuO	Cu	0.247		0.00		0.00	0.000	0.01	0.00		0.00	0.001
Ag ₂ O	Ag	0.000		0.00		0.00	0.000		0.00		0.00	0.000
As ₂ O ₃	As	0.001	-	-	-	-	-	-	-	-	-	-
BaO	Ba	0.058	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01	0.01	0.010
CdO	Cd	0.000		0.00		0.00	0.000		0.00		0.00	0.000
Cr ₂ O ₃	Cr	1.062		0.00		0.00	0.000		0.00		0.00	0.000
NiO	Ni	0.436		0.00		0.00	0.000		0.00		0.00	0.000
PbO	Pb	0.067		0.00		0.00	0.000		0.00		0.00	0.000
SeO ₂	Se	0.000	-	-	-	-	-	-	-	-	-	-
pH				10.34		10.35	10.345		10.34		10.35	10.345

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

7 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	L-1Ga1		L-1Ga2		Average g/m ²	L-1Gb1		L-1Gb2		Average g/m ²
			µg/ml	g/m ²	µg/ml	g/m ²		µg/ml	g/m ²	µg/ml	g/m ²	
Na2O	Na	2.6303	3.42	0.09	3.44	0.09	0.088	6.71	0.17	7.30	0.19	0.179
SiO2	Si	48.2041	17.76	0.04	17.74	0.04	0.039	17.50	0.04	17.94	0.04	0.039
Al2O3	Al	18.5753	5.32	0.03	5.31	0.03	0.027	6.25	0.03	6.49	0.03	0.032
CaO	Ca	3.5667	1.87	0.04	1.88	0.04	0.037	1.17	0.02	1.14	0.02	0.023
K2O	K	0.2294		0.00		0.00	0.000		0.00		0.00	0.000
Cl	Cl	0.4975	-	-	-	-	-	-	-	-	-	-
S	S	0.0782	-	-	-	-	-	-	-	-	-	-
CeO2	Ce	0.2957		0.00		0.00	0.000		0.00		0.00	0.000
Fe2O3	Fe	24.1543		0.00	0.01	0.00	0.000	0.03	0.00	0.02	0.00	0.000
MgO	Mg	0.4746	0.14	0.02	0.15	0.03	0.025	0.42	0.07	0.39	0.07	0.071
MnO	Mn	0.1070	0.01	0.01	0.01	0.01	0.006	0.00	0.00	0.00	0.00	0.000
Cs2O	Cs	0.3546	-	-	-	-	-	-	-	-	-	-
CdO	Cd	0.2382		0.00		0.00	0.000		0.00		0.00	0.000
Cr2O3	Cr	0.1208		0.00		0.00	0.000		0.00		0.00	0.000
NiO	Ni	0.1332		0.00		0.00	0.000		0.00		0.00	0.000
PbO	Pb	0.3400		0.00		0.00	0.000		0.00		0.00	0.000
pH				8.15		8.16	8.155		8.76		8.85	8.805

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

7 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	L-1VCa1		L-1VCa2		Average	L-1VCb1		L-1VCb2		Average
			$\mu\text{g/ml}$	g/m^2	$\mu\text{g/ml}$	g/m^2	g/m^2	$\mu\text{g/ml}$	g/m^2	$\mu\text{g/ml}$	g/m^2	g/m^2
Na ₂ O	Na	2.6303	3.48	0.09	3.53	0.09	0.090	13.94	0.36	14.18	0.36	0.360
SiO ₂	Si	48.2041	17.60	0.04	18.09	0.04	0.040	26.79	0.06	26.89	0.06	0.060
Al ₂ O ₃	Al	18.5753	5.09	0.03	5.30	0.03	0.026	6.93	0.04	6.95	0.04	0.035
CaO	Ca	3.5667	1.81	0.04	1.84	0.04	0.036	0.68	0.01	0.71	0.01	0.014
K ₂ O	K	0.2294		0.00		0.00	0.000		0.00		0.00	0.000
Cl	Cl	0.4975	-	-	-	-	-	-	-	-	-	-
S	S	0.0782	-	-	-	-	-	-	-	-	-	-
CeO ₂	Ce	0.2957		0.00		0.00	0.000		0.00		0.00	0.000
Fe ₂ O ₃	Fe	24.1543		0.00		0.00	0.000	0.16	0.00	0.14	0.00	0.000
MgO	Mg	0.4746	0.10	0.02	0.10	0.02	0.017	0.06	0.01	0.07	0.01	0.011
MnO	Mn	0.1070	0.01	0.01	0.01	0.01	0.006	0.00	0.00	0.00	0.00	0.000
Cs ₂ O	Cs	0.3546	-	-	-	-	-	-	-	-	-	-
CdO	Cd	0.2382		0.00		0.00	0.000		0.00		0.00	0.000
Cr ₂ O ₃	Cr	0.1208		0.00		0.00	0.000		0.00		0.00	0.000
NiO	Ni	0.1332		0.00		0.00	0.000		0.00		0.00	0.000
PbO	Pb	0.3400		0.00		0.00	0.000		0.00		0.00	0.000
pH				8.25		8.26	8.255		9.64		9.66	9.650

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

Appendix D: ICP Results for PCT Test Elemental Release

7 Day PCT Test Normalized Elemental Release

Oxide	Element	Oxide Wt%	L-83a1		L-83a2		Average g/m ²	L-83b1		L-83b2		Average g/m ²
			µg/ml	g/m ²	µg/ml	g/m ²		µg/ml	g/m ²	µg/ml	g/m ²	
Na2O	Na	15.00	66.83	0.30	65.22	0.29	0.297	220.81	0.99	222.67	1.00	0.996
SiO2	Si	44.24	55.49	0.13	55.24	0.13	0.134	92.18	0.22	91.25	0.22	0.222
Al2O3	Al	15.40	23.28	0.14	23.20	0.14	0.143	37.32	0.23	37.01	0.23	0.228
CaO	Ca	2.96	0.57	0.01	0.56	0.01	0.013	1.05	0.02	0.96	0.02	0.024
K2O	K	0.19	-	0.00	-	0.00	0.000	-	0.00	-	0.00	0.000
Cl	Cl	0.41	-	-	-	-	-	-	-	-	-	-
S	S	0.06	-	-	-	-	-	-	-	-	-	-
CeO2	Ce	0.25	0.07	0.02	0.08	0.02	0.019	0.11	0.03	0.10	0.03	0.026
Fe2O3	Fe	20.02	3.91	0.01	3.85	0.01	0.014	7.41	0.03	6.72	0.02	0.025
MgO	Mg	0.39	0.09	0.02	0.11	0.02	0.021	0.17	0.04	0.15	0.03	0.034
MnO	Mn	0.09	0.01	0.01	0.01	0.01	0.007	0.03	0.02	0.03	0.02	0.022
Cs2O	Cs	0.29	-	-	-	-	-	-	-	-	-	-
CdO	Cd	0.20	0.04	0.01	0.04	0.01	0.012	0.04	0.01	0.04	0.01	0.012
Cr2O3	Cr	0.10	-	0.00	-	0.00	0.000	0.04	0.03	0.04	0.03	0.029
NiO	Ni	0.11	-	0.00	-	0.00	0.000	0.03	0.02	0.03	0.02	0.017
PbO	Pb	0.28	-	0.00	-	0.00	0.000	-	0.00	-	0.00	0.000
pH				10.84		10.84	10.840		11.49		11.52	11.505

*blanks denote release below detection limits of ICP for this element.

- Elements present in the waste composition that are not included in the waste form.

- ICP results were not obtained for these elements present in the waste form.

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