Vitrification of Simulated Fernald K-65 Silo Waste at Low Temperature

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Publication Date: January 31, 1999

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PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

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EXECUTIVE SUMMARY

Vitrification is the technology that has been chosen to solidify ~18,000 tons of geologic mill tailings at the Fernald Environmental Management Project (FEMP) in Fernald, Ohio. The geologic mill tailings are residues from the processing of pitchlende ore during 1949-1958. These waste residues are contained in silos in Operable Unit 4 (OU4) at the FEMP facility. Operable Unit 4 is one of five operable units at the FEMP. Operating Unit 4 consists of four concrete storage silos and their contents. Silos 1 and 2 contain K-65 mill tailing residues and a bentonite cap, Silo 3 contains non-radioactive metal oxides, and Silo 4 is empty.

The K-65 residues contain radium, uranium, uranium daughter products, and heavy metals such as lead and barium. The K-65 waste leaches lead at greater than 100 times the allowable Environmental Protection Agency (EPA) Resource, Conservation, and Recovery Act (RCRA) concentration limits when tested by the Toxic Characteristic Leaching Procedure (TCLP). Vitrification was chosen by FEMP as the preferred technology for the Silos 1, 2, 3 wastes because the final waste form met the following criteria:

- controls radon emanation
- eliminates the potential for hazardous or radioactive constituents to migrate to the aquifer below FEMP
- controls the spread of radioactive particulates
- reduces leachability of metals and radiological constituents
- reduces volume of final wasteform for disposal
- silo waste composition is favorable to vitrification
- will meet current and proposed RCRA TCLP leaching criteria

Glasses that melt at 1350°C were developed by Pacific Northwest National Laboratory (PNNL) and glasses that melt between 1150-1350°C were developed by the Vitreous State Laboratory (VSL) for the K-65 silo wastes. Both crucible studies and pilot scale vitrification studies were conducted by PNNL and VSL. Subsequently, a Vitrification Pilot Plant (VPP) was constructed at FEMP capable of operating at temperatures up to 1450°C. The VPP began operation on June 19, 1996. The VPP was used to test surrogate FEMP wastes at melt temperatures between 1130°C and 1350°C. The VPP failed on December 26, 1996 while processing surrogate waste. After the failure of the FEMP VPP, vitrification technology and glass chemistry were reevaluated.

This report documents the glass formulation development for K-65 waste completed at SRTC in April, 1993 in conjunction with Associated Technical Consultants (ATC) of Toledo, Ohio. The glass developed for the FEMP was formulated in a lithia substituted soda-lime-lithia-silica (SLLS) glass per the Savannah River Technology Center (SRTC) patented Lithia Additive Melting Process (LAMP™)* to avoid problematic phase separation known to occur in the borosilicate glass system ($MO-B_2O_3-SiO_2$), where (MO = CaO, MgO, BaO, and PbO). Lime, MgO, BaO and PbO are all constituents of the FEMP wastes and thus subject to phase separation when vitrified in borosilicate glass. Phase separation is known to compromise waste glass stability.

The SRTC soda-lithia-lime-silica (SLLS) glass melted at 1050°C. Similar SLLS glass formulations have recently been demonstrated at the Oak Ridge Reservation (ORR) in a full scale melter with mixed (radioactive and hazardous) wastes. The low melting temperatures achieved with the SLLS glass minimize volatilization of hazardous species such as arsenic, lead, and selenium during vitrification. An 84% K-65 waste loading was demonstrated. The

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* Patent pending

SLLS glass is as durable as the high melting PNNL SLS glass and is more durable than the borosilicate glasses previously developed by VSL for the K-65 wastes.

The SRTC SLLS glass passes the Environmental Protection Agency (EPA) Toxic Characteristic Leach Procedure (TCLP) for all the hazardous constituents of concern under the pre August, 1998 final Phase IV regulations and at the delisting limits calculated in this study. Further optimization of glass formulations in the SLLS glass forming system using the LAMP™ technology should be based on new waste analyses for Silo 1 and 2 wastes provided by FEMP in February, 1998. Slight modification of the SLLS glass at lower PbO content, e.g. lower waste loadings, and/or higher Al_2O_3 compositions, e.g. higher concentrations of BentoGrout™ would provide glass formulations which meet the more stringent 1998 EPA TCLP limits so that the glass waste form would not need to be delisted.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

LIST OF ACRONYMS

ATC:	Associated Technical Consultants, Toledo, OH
BDAT:	Best Demonstrated Available Technology
DOE:	United States Department of Energy
EPA:	United States Environmental Protection Agency
FEMP:	Fernald Environmental Management Program
HWVP:	Hanford Waste Vitrification Project
LDR:	Land Disposal Restrictions
LAMP:	Lithia Additive Melting Process (patent pending)
NTS:	Nevada Test Site
ORR:	Oak Ridge Reservation
OU4	FEMP Operating Unit 4
PNNL:	Pacific Northwest National Laboratory
RCRA:	Resource Conservation and Recovery Act
REDOX:	Reduction/Oxidation
SBS:	Soda-Boro-Silicate glass
SLS:	Soda-Lime-Silica glass
SLLS	Soda-Lithia-Lime-Silica glass
SRS:	Savannah River Site
SRTC:	Savannah River Technology Center
TCLP:	EPA Toxicity Characteristic Leaching Procedure
TRU:	Transuranic waste
TVS:	Transportable Vitrification System
UTS:	Universal Treatment Standards for Land Disposal Restrictions
VPP:	Vitrification Pilot Plant (at Fernald, Oh.)
VSL:	Vitreous State Laboratory, Catholic University of America, Washington, DC
WSRC:	Westinghouse Savannah River Company, Aiken, SC

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INTRODUCTION

Vitrification is the technology that has been chosen to solidify ~18,000 tons of geological mill tailings at the Fernald Environmental Management Project (FEMP) at Fernald, Ohio. After stabilization, the waste glass will be transported to the Nevada Test Site (NTS) for final geological disposal. The geologic mill tailings are residues from the processing of pitchlende ores during 1949-1958. These residues are contained in silos in Operable Unit 4 (OU4) at the FEMP facility. Operable Unit 4, which is one of five operable units at the FEMP, consists of four concrete storage silos and their contents. Silos 1 and 2 contain K-65 mill tailing residues, Silo 3 contains non-radioactive metal oxides, and Silo 4 is empty.

The K-65 residues contain radium (Ra^{226}), uranium ($U^{234,235,236,238}$), uranium daughter products (Th^{230}), and heavy metals such as lead and barium. Of the 14,262 tons (12.9 million kilograms) of K-65 residues in Silos 1 and 2, the content is about 4 kg, the $U^{234,235,236,238}$ isotopes are about 11,000 kgs, and the Th^{230} is about 2 kgs [1]. The gamma radiation from the residue is sufficient to result in an average of about 200 mr/h outside the silo dome. Prior to 1991, the radon concentration of the silo headspace was between 50-60 million pCi/L. For this reason ~1,185 tons of bentonite was added in the headspace in 1991 which lowered the radon concentration in the head space to 200,000-300,000 pCi/L. The radon levels in the headspace have been gradually increasing since 1991 as the radon diffuses through the bentonite.

The K-65 waste leaches lead at greater than 100 times [1] the allowable Environmental Protection Agency (EPA) Resource, Concentration, and Recovery Act (RCRA) concentration limits [2] when tested by the Toxic Characteristic Leaching Procedure (TCLP). This causes the K-65 Silo 1 and 2 wastes to be RCRA characteristically hazardous. Since the Silo 1 and 2 wastes are both characteristically hazardous and radioactive, the K-65 residues are considered mixed wastes.

The Silo 3 wastes are a dry powdery mixture of metal oxides with a much lower concentration of radionuclides (thorium, uranium and Pb^{210}) than the Silo 1 and 2 wastes. There are approximately 4 tons of Silo 3 wastes for disposal. The Silo 3 wastes leach arsenic, chromium, cadmium, and selenium at concentrations exceeding characteristically hazardous RCRA limits. Since the Silo 3 wastes are characteristically hazardous and radioactive, they are also considered to be mixed wastes.

Vitrification was chosen as the preferred technology for the Silo 1, 2, and 3 wastes because the final waste form met the following criteria [1,3]:

- controls radon emanation: the radon released from the storage/disposal facility must not exceed the EPA limit of 20 pCi/M^2/s
- eliminates the potential for hazardous or radioactive constituents to migrate to the aquifer below FEMP
- controls the spread of radioactive particulates
- reduces leachability of metals and radiological constituents
- reduces volume of waste for disposal
- controls the gamma radiation from the residue
- silo waste composition is favorable to vitrification
- pass TCLP for RCRA hazardous constituents

Glasses that melt at 1350°C were developed by Pacific Northwest National Laboratory (PNNL) [1,3] and glasses that melted between 1150-1350°C were developed by the Vitreous State Laboratory (VSL) for the K-65 silo wastes. The PNNL formulated glasses primarily in the soda-lime-silica (SLS) system since the waste is high in BaO and BaO readily substitutes for CaO (lime) in SLS glass. The VSL formulated glasses primarily in the borosilicate glass forming system. The PNNL and the VSL conducted both crucible and pilot scale vitrification studies.

Radon losses from the PNNL glasses were monitored during vitrification. Although the glasses were melted at 1350°C, the radon emissions were monitored as a function of heat up temperature and time [3]. Significant radon losses were encountered in the 500-900°C temperature range. Radon losses after vitrification at 1350°C were computed to be 84-100%. The authors state that although radium (Ra^{226}) will be contained in the glass once it is atomistically bonded and limit future Rn^{222} release, essentially all of the radon (Rn^{222}) present in the waste will be released during high temperature vitrification. Waste loadings for the PNNL glass formulations ranged from ~70 to 90 wt% with volume reductions reported to vary between 50 and 68% [1,3].

After pilot scale testing by PNNL and VSL, a Vitrification Pilot Plant (VPP) was constructed at FEMP to operate at temperatures up to 1450°C. The VPP began operation at FEMP on June 19, 1996. During this initial startup testing 12.5 tons of benign startup "frit" were produced. Silo 1, 2, and 3 surrogate melter tests were completed in September 1996 and produced 11 tons of glass. Another campaign using Silo 1 and 2 surrogate with bentonite was subsequently performed. All of the VPP surrogate testing was completed at melt temperatures between 1130°C and 1350°C. A final transition to lower temperature operation at ~1150°C was planned when the VPP failed on December 26, 1996.

After the failure of the FEMP VPP, vitrification technology and glass chemistry was reassessed. This report documents SRTC glass formulation and development for K-65 waste that was completed in April, 1993 but, here-to-fore undocumented.

BACKGROUND

Waste Composition

In 1991 Pacific Northwest National Laboratory analyzed the composition of the K-65 residues from Fernald on a dry wt% calcine (oxide) basis. The as analyzed K-65 residue had a 29 wt% moisture content and contained rock and debris (e.g. metal springs, cloth, and leather gloves) [1]. The waste analyses determined by PNNL for a waste samples taken in 1989 and 1990 (designated as PNNL-89 and PNNL-90) are compared to analyses performed by Controls for Environmental Pollution, Inc. (CEP) on a sample taken in 1990 (designated CEP-90) and to analyses performed by National Lead of Ohio (NLO) on a sample taken prior to 1989 (designated as NLO) in Table I. In 1993, PNNL published an average composition for the K-65 residues in Silos 1 and 2, an average composition of the Silo 3 waste, and an average composition of the BentoGrout™ added to the tank headspace. For comparison, all of these compositions are given in Table I. Note that the PNNL analyses are normalized and contain significant contributions of unspecified components designated as "other." The "other" component is reported as containing between 1-3 wt% sulfate in the K-65 residues and up to 18 wt% sulfate in the Silo 3 material [3] on a dry solids basis.

Analyses performed by FEMP in 1993 indicate that the sulfate in Silo 1 varies between 0.04 and 0.35 wt% on a wet solids basis while the sulfate in Silo 2 varies between 0.26-1.93 wt% on a wet basis [4]. The total organic carbon (TOC) for Silo 1 wastes varies from 0.52 to 3.48 wt% on a wet basis while the TOC for Silo 2 varies from 0.01 to 2.44 wt% on a wet solids basis [4].

In February, 1998 FEMP developed revised surrogate waste compositions for Silo 1 and 2 wastes with BentoGrout™[5]. These compositions are similar but not identical to the average compositions reported by PNNL in 1993 (Table I).

Glass Formulations

Due to the high concentration of silica (SiO_2), lime (CaO), baria (BaO) and PbO in the K-65 Fernald wastes, PNNL chose the use of soda-lime-silica (SLS) glass for solidification. PNNL only formulated glasses in the borosilicate system (Table II) for Silo 3 wastes which contained no PbO. For the K-65 wastes, the initial PNNL formulations required only one simple additive, Na_2O as Na_2CO_3, rather than formulation of a complex glass making frit. This was because BaO in the K-65 waste could readily substitute for CaO in SLS glass.

The SLS glass forming system is a well known system used to make window glass and it has been extensively studied by glass chemists [6-10]. Soda-silica glass is known to be extremely tolerant of high Zn [9] and other heavy metals. SLS glasses have also been shown to be tolerant of high Pb [10] content whereas the borosilicate based glasses tend to phase separate [11, 12]. SLS glass formulations have been used for In-Situ Vitrification (ISV) to solidify contaminated soils [13, 14]. SLS glasses were also successfully used to treat reactive Na metal formed and contaminated during efforts to develop sodium-cooled fast breeder nuclear reactors [15].

Table I.

Summary of Normalized Chemical Analyses of K-65 Residues (Silos 1 and 2), Silo 3 and BentoGrout™ [1,3,5]

Oxide	Normalized Oxide Wt %									
	1990 CEP-90 [1]	1990 PNNL-90 [1]	1991 NLO [1]	1989 PNNL-89 [1]	1993 Average K-65 Silos 1 & 2 [3]	1998 K65 Silo 1 Surrogate* with 9 wt% Bento-Grout™ [5]	1998 K65 Silo 2 Surrogate* with 9 wt% Bento-Grout™ [5]	1993 FEMP Silo 3 [3]	1993 Bento-Grout™ [3]	1998 Bento-Grout™ [5]
Al_2O_3	6.94	3.81	2.71	3.67	3.7	4.77	5.77	8.3	15.8	16.27
BaO	8.12	2.90	0.13	6.36	6.3	6.32	3.48	0.1	---	---
CaO	1.32	2.85	8.91	3.33	1.5	0.18	3.73	7.3	1.8	1.85
Fe_2O_3	9.67	5.37	2.81	4.43	4.8	3.56	6.69	12.6	3.6	3.71
K_2O	---	---	---	---	0.9	0.81	1.50	2.8	0.8	0.82
MgO	2.41	3.83	2.30	1.51	1.7	2.18	2.11	15.8	6.3	6.49
Na_2O	1.59	1.19	1.55	1.89	1.7	1.41	1.34	9.3	3.4	3.50
P_2O_5	---	5.08	---	0.76	0.8	0.58	0.68	14.5	1.4	---
PbO	10.27	9.97	9.00	10.45	12.3	13.81	6.92	0.3	---	---
SiO_2	56.62	61.28	71.43	63.46	62.9	62.08	62.38	22.0	65.4	67.35
SO_4	---	---	---	---	---	4.30	5.41	---	---	---
Other	3.06	3.72	1.16	4.14	3.5	---	---	7.2	1.5	---
SUM	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.0	100.0	100.00

*2-3 grams kerosene added to the surrogate to simulate the Total Organic Carbon

PNNL developed four series of glass compositions for various combinations of actual FEMP wastes [1,3]. Series A glass was a SLS formulation containing K-65 waste alone (Table II). Series B glass was an SLS formulation containing equal amounts of K-65 and BentoGrout™. Series C glass was a borosilicate formulation containing Silo 3 waste. Series D glass was an SLS formulation containing equal amounts of K-65 and Silo 3 wastes. Carbon was added to PNNL-A, B, and C melts to reduce the sulfate (SO_4) to SO_3 or SO_2 vapor [3]. However, the final glass redox was not measured and several of the glasses exhibited the formation of metallic nodules (PNNL-A) indicating that too much reduction had occurred in the glass causing sulfides and metallic species to form. For the Series D glasses even significant quantities of carbon (9g of Carbon/100g of glass) did not mitigate sulfate formation on the melt surface. Additional tests that were performed by PNNL are discussed in the text of reference 3 and state that when glass PNNL-D (9g of Carbon/100g glass) was reformulated to be similar to PNNL-C (no carbon) then no sulfate layer formed.

All of the PNNL glasses melted at elevated temperatures between 1289° and 1595°C (Table II). Glasses PNNL-A, C and D contained unidentified white crystalline inclusions while PNNL-C (a borosilicate glass) and PNNL-D (a high sulfate containing glass) were reported to be potentially phase separated.

Table II.

Summary of Estimated Chemical Compositions and Melt Conditions of Glasses Developed By Pacific Northwest National Laboratory with Actual FEMP Wastes [1,3]

Oxide	Normalized Oxide Wt%				
	PNNL-K-65 [1]	PNNL-A [3]	PNNL-B [3]	PNNL-C [3]	PNNL-D [3]
Al_2O_3	6.46	3.2	8.7	20.0	3.1
B_2O_3	---	---	---	5.0	---
BaO	7.57	5.4	2.7	---	3.0
CaO	1.23	1.3	1.4	4.7	1.9
Cr_2O_3	0.49	---	---	---	---
Fe_2O_3	9.01	4.1	3.7	8.1	4.4
K_2O	---	0.8	0.7	1.8	0.9
Li_2O	---	---	---	0.5	0.1
MgO	2.24	1.5	3.6	10.2	3.4
Na_2O	13.49	15.1	15.1	6.0	14.8
NiO	0.64	---	---	---	---
P_2O_5	---	0.7	1.0	9.4	2.8
PbO	9.57	10.6	5.2	0.2	5.9
SiO_2	52.75	54.3	56.3	30.0	56.9
Other	---	3.0	1.6	4.1	2.8
SUM	100.0	100.0	100.0	100.0	100.0
Glass Characteristics					
Redox	Not Measured	Reducing	Reducing	Not Measured	Very Reduced
Waste/Additive	K-65 + Na_2O	K-65 + Na_2O + 2g Carbon	K-65 + Bent + Na_2O + 2g Carbon	Silo 3 + Al_2O_3 + B_2O_3 + SiO_2	K-65 + Silo 3 + Na_2O + SiO_2 + 9g Carbon
Waste Loading (Wt%)	88	89	89	74	74
Density	3.1	Not Determied	Not Determied	Not Determied	Not Determied
Vol Red (%).	61	55	55	68	50
Melt Temp (°C)	ng	1383	1595	1289	1413
Glass System	SLS	SLS	SLS	Borosilicate	SLS
Second Phase (Crystalline or Phase Separation)	Not Determined	Metal Nodules + Crystalline Inclusions	Crystalline Inclusions	Possible phase separation	Sulfate layer + Crystalline Inclusions + possible phase separation

The PNNL continued glass formulation work in the SLS glass forming system using up to ~5 wt% B_2O_3[1]. Many of the crucible studies were performed with simulated waste and did not contain carbon or sulfate. Most of the PNNL SLS glasses melted at temperatures between 1350°C to greater than 1450°C and had waste loadings of 80-84 wt%. Subsequently, the PNNL performed minimelter tests with actual K-65 residues which were mixed with waste from the Hanford Waste Vitrification Program (HWVP-12) and contained high boron (14 wt%) and lithia (5 wt%). This glass melted at 1150°C but the K-65 waste loading was limited to <50 wt%.

The Vitreous State Laboratory (VSL) formulated K-65 waste glasses in the sodium-borosilicate (SBS) system at various boron concentrations (5.88-12.8 wt%) but with less lithia (1.5-2 wt%) than used in the current study. During minimelter tests, urea was added to control foaming of sulfate. These SBS glasses had waste loadings varying between 60-74 wt% and melted at temperatures between 1000-1050°C.

Glass Durability

The EPA declared vitrification as the Best Demonstrated Available Technology (BDAT) for high level radioactive waste in 1990 [16] and produced a Handbook of Vitrification Technologies for Treatment of Hazardous and Radioactive Waste in 1992 [17]. Glass in general performs well during EPA Toxic Characteristic Leach Procedure testing and in the more severe leaching tests developed for high level waste compliance [18-21], because the waste species are chemically bonded in the glass structure on an atomic scale [22-27].

If a waste fails the TCLP test at greater than the Characteristically Hazardous Limits listed in Table III, the waste has to be treated to meet the Universal Treatment Standards (UTS) The UTS were promulgated by the EPA on September 19, 1994 [28] for listed mixed wastes having to meet the Land Disposal Restrictions (LDR). In August, 1995, the EPA proposed a new rule (Phase IV of the Land Disposal Restrictions) which would require Characteristically Hazardous Waste to be treated to the same UTS limits as listed wastes [29]. The Phase IV Rule was finalized on May 26, 1998, and took affect August 26, 1998 [30]. This lowered the acceptable Pb TCLP concentration for K-65 wastes from the 5 ppm used in the PNNL and VSL studies to 0.75 ppm.

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[1] This report distinguishes between borosilicate and soda-lime-silica (SLS) based on the ASTM C-162 definition of borosilicate glass containing >5 wt% B_2O_3.

Table III.

Environmental Protection Agency Concentration Limits for Hazardous Wastes

Element	Characteristically Hazardous TCLP Limits [2] (ppm)	Universal Treatment Standard Limits [30] (ppm)
Ag	5.0	0.14
As	5.0	5.0
Ba	100.0	21
Be	---	1.22
Cd	1.0	0.11
Cr	5.0	0.60
Hg	0.2	0.025
Ni	-	11.0
Pb	5.0	0.75
Sb	---	1.15
Se	1.0	5.7*
Tl	---	0.20
V**	---	1.6
Zn**	---	4.3
F	---	400
CN	---	30

* Se must be treated to the characteristic limit (1.0 mg/L) to be non-hazardous, although it may be land disposed as a hazardous waste if <5.7 mg/L TCLP

** V and Zn are not underlying hazardous constituents

Some of the SLS glasses developed for the FEMP wastes by PNNL passed the TCLP testing below the 1998 UTS limits (Table IV). In particular, the SLS glass with K-65 waste (PNNL-B) released 0.38-0.47 ppm Pb for an average K-65 waste (Silo 1 plus Silo 2) while PNNL-D, a mixture of Silo 1, 2, and 3 wastes released 0.50-0.58 ppm Pb. Both of these glasses melted at ~1400°C. The borosilicate glass developed for the Silo 3 waste alone released only 0.017-0.019 Pb and melted at 1300°C. However, Silo 3 waste only contains 0.3 wt% PbO while the Silo 1 and 2 (K-65) wastes contain between 9.00-10.45 wt% PbO. These glasses contained lower PbO content and higher Al_2O_3 content (more BentoGrout™) than the remaining PNNL glass formulations. All of the SLS glasses used for the In-Situ Vitrification (ISV) of contaminated soils [14] and some of the SLS slags used to stabilize Pb [31] also passed the EPA Toxicity Characteristic Leaching Procedure (TCLP) test at the promulgated May, 1998 UTS limits.

Table IV.

Comparative EPA TCLP Testing for Unstabilized K-65 Residues and for SLS Glasses Formulated by PNNL

RCRA Metal	EPA Limit (mg/L)	Raw K-65 Residue [1]	PNNL-A K-65 Waste Glass [3] (mg/L)	PNNL-B K-65 Waste + Bentonite Glass [3] (mg/L)	PNNL-C Silo 3 Waste Glass [3] (mg/L)	PNNL-D[3] K-65 Waste + Silo 3 Waste Glass (mg/L)
Ag	5.0	<0.1	<0.01	<0.01	<0.01	<0.01
As	5.0	<1.0	0.004-0.005	0.003-0.03	0.47-0.79	0.038-0.067
Ba	100.0	0.76	0.69-0.87	0.43-0.56	0.028-0.074	1.6-2.0
Cd	1.0	0.1	<0.005	<0.005	0.007-0.012	<0.005
Cr	5.0	<0.2	<0.01	<0.01	<0.01	<0.01
Hg	0.2	<0.03	<0.0002	<0.0002	<0.0002	<0.0002
Pb	**5.0**	**630**	**0.81-1.2**	**0.38-0.47**	**0.017-0.019**	**0.50-0.58**
Se	1.0	<0.1	<0.002	<0.002	<0.002	<0.002

EXPERIMENTAL

Rationale for Soda-Lithia-Lime-Silica (SLLS) Glass Formulation

The K-65 wastes contain 9-12.3 wt% PbO, 0.13-8.12 wt% BaO, 1.3-8.9 wt% CaO, and 1.5 to 3.8 wt% MgO (Table I). The $PbO-SiO_2-B_2O_3$, $BaO-SiO_2-B_2O_3$, $CaO-SiO_2-B_2O_3$, and $MgO-SiO_2-B_2O_3$ glass forming systems all contain large regions of phase separation [12]. A composite ternary phase diagram is shown in Figure 1, e.g. the $MO-SiO_2-B_2O_3$ system where MO = PbO, BaO, CaO and MgO. At concentrations greater than 40 wt% SiO_2, the phase separation is metastable in the $PbO-SiO_2-B_2O_3$ system [32]. The metastable portion of the immiscibility dome dips beneath the liquidus surface on the high-SiO_2 side of the boundary between the fields of SiO_2 and $PbO \bullet 2B_2O_3$. This causes the separation of an SiO_2 rich glass from a much denser $PbO \bullet 2B_2O_3$ rich glass [32]. Therefore, formulation of borosilicate waste glasses for the K-65 wastes may readily produce phase separated glasses with the PbO being stabilized in a borate rich phase. The borate rich phase would release more Pb than a homogeneous glass during TCLP testing.

The low melting borosilicate glasses developed by the VSL and those subsequently developed by FEMP had total MO (where MO = PbO, BaO, CaO and MgO) concentrations within the known regions of phase separation in the $PbO-SiO_2-B_2O_3$ and $BaO-SiO_2-B_2O_3$, $CaO-SiO_2-B_2O_3$, and $MgO-SiO_2-B_2O_3$ systems (see Figure 1). Due to the potential for stable and/or metastable phase separation in the $MO-SiO_2-B_2O_3$ glass forming systems, the current study concentrated on methods to lower the melt temperature of SLS glasses. In the SLS glass forming system PbO, MgO and BaO can all substitute for CaO in the glass structure without separation of a second phase.

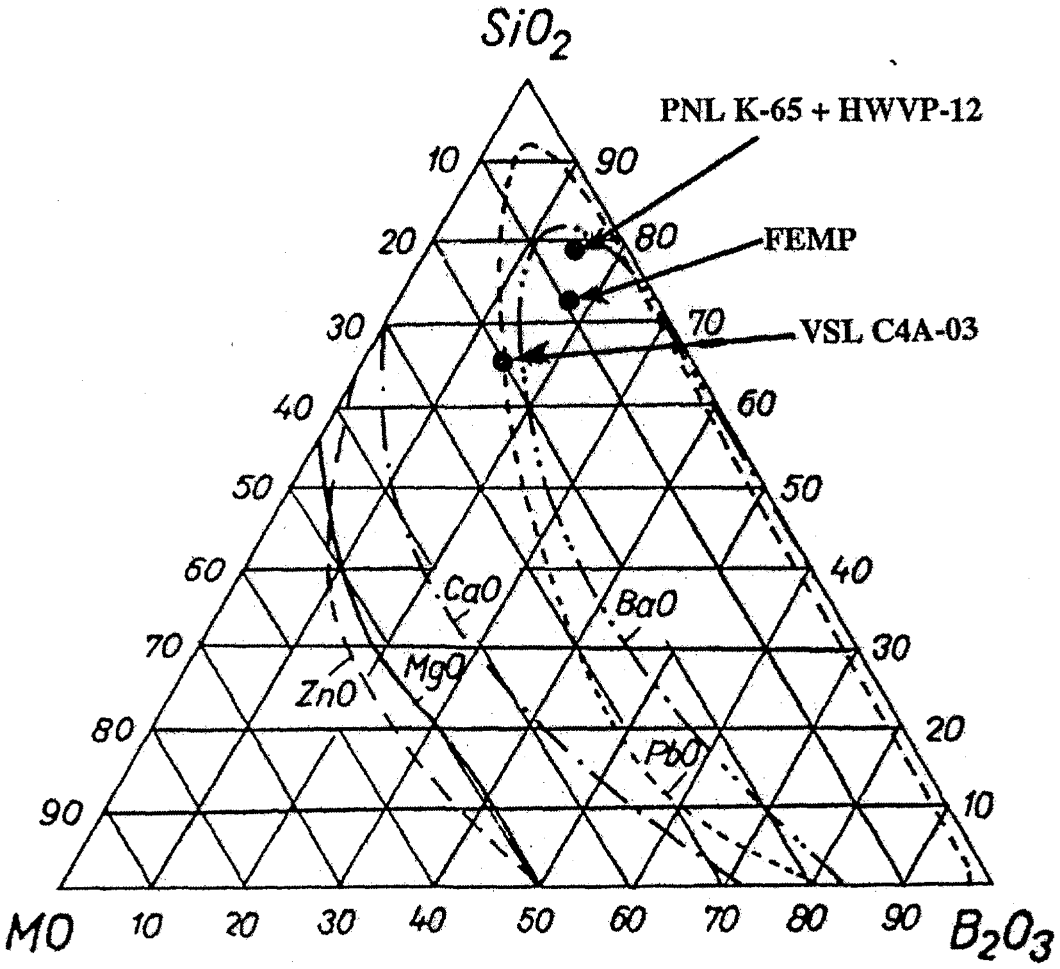

Figure 1. The Phase Diagram for the Systems (CaO, BaO, PbO, MgO)-SiO_2-B_2O_3 [12].

Glass Composition

The process product models [26] developed by SRTC for the operation of the Defense Waste Processing Facility (DWPF) were used to formulate an SLS glass for the K-65 waste composition given in Table I. The calculations were optimized to allow for calculation of an optimum glass formulation based on various concentrations of K-65 waste with Silo 3 waste and BentoGrout™. The K-65 waste composition used for the calculations are those given in Table I in this report from Reference 3 and are similar to the average of the 1998 Silo 1 + 2 wastes mixed with 9 wt% BentoGrout™.

One SLS glass was formulated by SRTC with an 85% waste loading. Lithia (Li_2O) was partially substituted for the Na_2O so that about 7 wt% Li_2O and 7 wt% Na_2O were the only two additives. This surrogate waste glass composition was melted by Ray Richards of Associated Technical Consultants (ATC) at 1050°C and sent to SRTC for density determination, composition analysis, and leach testing using the EPA TCLP. The target and analyzed composition of this glass is given in Table V. The final waste loading in the as fabricated glass was 84%.

Glass Durability

The TCLP results for the SRTC glass formulation are given in Table VI and compared with published TCLP results of the raw K-65 waste and the glasses formulated by PNNL. Although the SRTC formulation melts 300°C lower than the PNNL formulation it is of equivalent durability and waste loading to the high temperature K-65 glass and more durable than the low melting VSL borosilicate glass. The PNNL, VSL, and SRTC glasses all pass the TCLP at the pre 1998 UTS concentration limits (Table VI). All of the glass formulations, therefore, need to be modified to compositions resembling PNNL-B or PNNL-D(Tables II and VI) by either lowering the PbO content (e.g. lowering the waste loading) and/or increasing the Al_2O_3 or BentoGrout™ component of the waste form.

Table V.

SRTC Soda-Lithia-Lime-Silica (SLLS) Glass

GLASS COMPONENT	OXIDE WT% Target	OXIDE WT% As Analyzed
Al_2O_3	3.14	4.32
BaO	5.35	4.95
CaO	1.27	1.44
Cr_2O_3	---	0.14
Fe_2O_3	4.08	4.51
K_2O	0.76	0.73
Li_2O	8.50	7.92
MgO	1.44	1.63
Na_2O	10.88	9.85
NiO	-	0.09
P_2O_5	0.68	0.96
PbO	10.45	10.30
SiO_2	53.44	53.10
Other		
SUM	99.99	100.03
GLASS CHARACTERISTICS		
Waste Loading	85	84
MELTED (°C)	1050	1050
Homogeneous (visual)	N/A	YES

Table VI.

Comparative EPA TCLP Testing for Unstabilized K-65 Residues and for K-65 Waste Glasses Formulated by PNNL, VSL, and SRTC

RCRA Metal	1998 EPA Final Phase IV UTS Limits [30] (mg/L)	Raw K-65 Residue [1] Silo's 1 & 2 (mg/L)	Ranges Measured for Silo 3 Residues [34]	PNNL-A SLS K-65 Waste Glass [3] (mg/L)	VSL Borosilicate K-65 Waste Glass [3] (mg/L)	SRTC SLLS K-65 Waste Glass Replicate A (mg/L)	SRTC SLLS K-65 Waste Glass Replicate B (mg/L)
Ag	0.11	<0.1	ND-0.032	<0.01	Not Determined	0.026	0.020
As	5.0	<1.0	ND-41.5	0.004-0.005	Not Determined	<0.002	<0.002
Ba	21	0.76	0.02-0.156	0.69-0.87	Not Determined	2.62	2.73
Cd	0.20	0.1	0.108-6.32	<0.005	Not Determined	<0.010	<0.010
Cr	0.60	<0.2	0.336-11.9	<0.01	Not Determined	<0.04	<0.04
Hg	0.025	<0.03	ND-0.003	<0.0002	Not Determined	<0.0096	<0.0096
Ni	11.0	3.2	Not Determined	Not Determined	Not Determined	0.062	<0.050
Pb	**0.75**	630	ND-1.01	**0.81-1.2**	**1.9-2.76**	**1.272**	**1.619**
Se	5.7	<0.1	0.92-11.7	<0.002	Not Determined	0.158	0.297

ND = Not detected

Impact of the 1998 EPA Regulations

Prior to August, 1998, the FEMP wastes would have had to be treated to meet the EPA Characteristically Hazardous Limits given in Table III. However in August, 1995, the EPA proposed a new rule (Phase IV of the Land Disposal Restrictions) which would require Characteristically Hazardous Waste to be treated to the same Universal Treatment Standards (UTS) as those for listed mixed wastes [29]. The Phase IV Rule was finalized on May 26, 1998, and compliance was required for any wastes treated after August 26, 1998 [30]. Any mixed wastes not stabilized by that date will be required to be treated to the UTS limits given in Table III, rather than to the Characteristically Hazardous Limits. This lowers the acceptable Pb TCLP concentration for K-65 waste forms from the previous 5 mg/L to 0.75 mg/L. None of the glasses formulated by PNNL, VSL or SRTC are adequate to meet the 1998 final Phase IV Land Disposal Restrictions (LDR) given in Table III, VI and VII [30].

The final rule requires that a treated waste form be tested against the UTS standards not only for the constituents which fail the TCLP characteristically hazardous limit, but for all "hazardous constituents which are present in the waste, i.e., underlying hazardous constituents" [29, 30]. The Fernald Silo 1 and 2 wastes exhibit TCLP leaching concentrations for lead which exceed the Toxicity Limit. Therefore, under the final regulations the final waste form will have to meet the UTS for lead as given in Table III and VI. Similarly, the Fernald Silo 3 wastes exhibit TCLP leaching concentrations for As, Cd, Cr, and Se which exceed the Toxicity Limit [34] and Pb exceeds the UTS limits and is, therefore, an underlying constituent (Table VI). The final waste form for the Silo 3 waste will have to meet the UTS standards for all of these constituents.

Alternatively, a delisting petition could be prepared per 40 CFR 260.22 "Petitions to Exclude a Waste Produced at a Particular Facility," for the K-65 treated waste glass. The potential delisting limits required for the K-65 waste glass can be calculated as follows:

- assume 1.8 million gallons waste x 50% waste loading x 10 yr treatment = 180,000 gallons of glass disposed/yr.

This allows a Dilution/Attenuation Factor (DAF) of 100 and generates the Delisting Limits given in Table VII. If a 5 year treatment time is used, then the DAF is reduced to 80. If the waste form is delisted it will have to meet the delisting limits for all the constituents given in Table VII, including fluoride and cyanide. Delisting of the K-65 waste glass product is attractive because it raises the Pb TCLP release limit from 0.75 ppm to 1.5 ppm. This would necessitate less reformulation of the Fernald waste glass product than the alternative of meeting the UTS limits.

Glass Physical Properties and Materials Compatibility

The density of the glass as measured by ASTM C693 procedure is 2.86 g/cm^3. Testing at ATC indicated that the SRTC SLLS glass formulation was totally compatible with Inconel® 690 electrodes commonly used in waste glass melters.

Glass Attributes

The glass attributes of the SRTC SLLS glass are compared to the glasses formulated for FEMP by PNNL and VSL in Table VIII.

Table VII.

Final 1998 EPA TCLP Land Disposal Restrictions, Phase IV [30].

Element	1998 Final Phase IV UTS Limits TCLP, mg/L[30]	Delisting Limit TCLP, mg/L 10 Year Treatment DAF =100	Delisting Limit TCLP, mg/L 5 Year Treatment DAF =80
Ag	0.14	20	16
As	5.0	5.0	4.0
Ba	21	200	160
Be	1.22	0.40	0.32
Cd	0.11	0.50	0.40
Cr	0.60	10	18.0
Hg	0.025	0.20	0.16
Ni	11	10	8.0
Pb	0.75	1.5	1.2
Sb	1.15	0.60	0.48
Se	5.7*	5.0	4.0
Tl	0.20	0.20	0.16
V**	1.6	20	16
Zn**	4.3	---	---
F	400	400	320
CN	---	590 total mg/kg	590 total mg/kg

* Se must be treated to the characteristic limit (1.0 mg/L) to be non-hazardous, although it may be land disposed if <5.7mg/L TCLP

** V and Zn are not underlying hazardous constituents

Table VIII.

Comparison of Attributes of Glasses Formulated for FEMP K-65 Silo Wastes.

GLASS ATTRIBUTES IN REFERENCES SURVEYED AND GIVEN IN THIS STUDY	SRTC Formulation	PNNL Formulations	PNNL Formulations	VSL Formulations
Glass Forming System	Soda-Lithia-Lime-Silica	Soda-Lime-Silica and Borosilicate	Borosilicate	Borosilicate
Phase Separation	No Potential	No Potential	Observed	Potential
Melt Temperature (°C)	1050	1289-1595	1150	1000-1050
Waste Loading Demonstrated (%)	84	80-84	<50	60-74
Volume Reduction (%)	~60	55-68	Not Determined	Not Determined
Density (g/cm^3)	2.86	3.1	Not Determined	Not Determined
Pb TCLP Concentrations (mg/L)	1.3-1.6	0.81-1.2	Not Determined	1.9-2.76
Inconel®690 Compatible	Demonstrated	Not Determined	Not Determined	Not Determined

CONCLUSIONS

Glasses for the FEMP K-65 silo wastes should be formulated in the Soda-Lime-Silica (SLS) system to avoid problematic phase separation known to occur in borosilicate glass forming systems, e.g. the MO-B_2O_3-SiO_2 glass forming systems where MO = CaO, MgO, BaO, and PbO. The K-65 wastes contain all of these MO oxides which makes phase separation likely. Phase separation is known to compromise glass durability.

Glasses for the FEMP K-65 silo wastes should be formulated to melt at the lowest temperatures reasonably achievable to minimize volatilization of radioactive radon and hazardous species such as arsenic, and selenium. A SLS glass can be formulated to melt as low as 1050°C while still maintaining a high waste loading, e.g. 84 wt%, by the addition of only two glass forming additives, Li_2O and Na_2O using the SRTC Lithia Additive Stabilization Process (LAMP).*

The SRTC SLLS glass passes the Environmental Protection Agency (EPA) Toxic Characteristic Leach Procedure (TCLP) for all the hazardous constituents of concern under the pre 1998 EPA Land Disposal Restrictions and at the delisting limits calculated in this report. The SLLS glass is as durable as the high melting SLS glasses formulated by Pacific Northwest Laboratory (PNNL) and is more durable than the borosilicate glasses formulated by the Vitreous State Laboratory (VSL) for the K-65 wastes. Slight modification of the SLLS glass at lower PbO content, e.g. lower waste loadings, and/or higher Al_2O_3 compositions, e.g. higher concentrations of BentoGrout™ would provide glass formulations which meet the more stringent 1998 EPA TCLP limits so that the glass waste form would not need to be delisted.

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* Patent pending.

RECOMMENDATIONS

Further optimization of glass formulations, redox issues, and sulfate control in the Soda-Lithia-Lime-Silica (SLLS) system needs to be pursued. The soda-lithia-lime-silica (SLLS) glass reported in this study should be retested with a more complete K-65 waste composition which includes the reference amounts of K-65 waste sulfate and organics. Additional crucible tests should be performed in the presence of the reported extremes for sulfate and TOC in the Silo 1 and 2 wastes. Other Li_2O containing glasses of similar composition should be optimized to lower the release of Pb during EPA TCLP testing to comply with the new EPA proposed Phase IV Rule for Universal Treatment Standards (UTS) which require that the Pb release be <0.75 ppm instead of <5.0 ppm. Current formulations using the SRTC LAMP™ technology already pass the TCLP at the delisting limits calculated in this study. Slight modification of the SLLS glass at lower PbO content, e.g. lower waste loadings, and/or higher Al_2O_3 compositions, e.g. higher concentrations of BentoGrout™ would provide glass formulations which meet the more stringent 1998 EPA TCLP limits so that the glass waste form would not need to be delisted. After glass optimization a series of minimelter tests should be completed on both surrogate and real waste.

SRTC successes in late 1997 with co-vitrification of two highly variable mixed wastes at the Oak Ridge Reservation (ORR) indicate that high sulfate and high organic containing (including wood chips) wastes can successfully be vitrified. These vitrification successes were completed in a field scale Transportable Vitrification System (TVS) at the ORR [33].

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