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Full-Scale Cold Crucible Test on Vitrification of Savannah River Site SB4 HLW Surrogate

A.P. Kobelev, S.V. Stefanovsky, V.V. Lebedev, M.A. Polkanov, V.V. Gorbunov, A.G. Ptashkin, O.A. Knyazev
SIA Radon, Moscow, Russia

J.C. Marra
Savannah River National Laboratory, Aiken, SC, USA

K.D. Gerdes
US DOE Office of Environmental Management, Washington, DC, USA

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Abstract

The full-scale cold crucible test on vitrification of sludge batch 4 (SB4) Savannah River Site HLW surrogate using a 418 mm inner diameter stainless steel crucible was carried-out for 66 hrs. Commercially available Frit 503-R4 (8 wt.% Li_2O, 16 wt.% B_2O_3, 76 wt.% SiO_2) was used as a glass forming additive at a calcine to frit ratio of 1:1 (50 wt.% calcine, 50 wt.% frit). Two portions of slurry prepared from frit and mixture of chemicals simulating waste in amount of ~750 kg and from frit and waste surrogate prepared by the SRT-MST-2007-00070 procedure in amount of ~1,300 kg with water content of ~27 and ~50 wt.%, respectively, was processed and ~875 kg of the vitrified product in total (~415 + 460 kg) was obtained. Average parameters were as follows: vibration power - 121.6 to 134.1 kW, feed rate (capacity) - 25.1 to 39.8 kg/hr, glass pour rate (productivity) - 14.0 kg/hr specific energy expenses for feed processing - 4.8 to 3.4 kW×hr/kg, specific energy expenses for glass production (melting ratio) - 8.7 to 9.6 kW×hr/kg, specific glass productivity - 2453 kg/(m^2×d). The product was composed of major vitreous and minor spinel structure phases. No nepheline phase was found. Average degree of crystallinity was estimated to be ~12 vol.%. Cesium was found to be the most volatile component (up to ~60 wt.% of total). Lithium, sodium and boron are less volatile. Other major feed constituents (Al, Si, Mg, Fe, Mn) were not volatile but their carry-over with gas-vapor flow occurred.

Introduction

The successful vitrification demonstration of sludge batch 2 (SB2) waste surrogate using a cold crucible inductive melting (CCIM) process [1-8] allowed transfer to the next step of SRNL – Daymos Ltd./Radon collaborative work on CCIM vitrification of SB4 waste surrogate. As followed from SRNL research work on selection of frit and glass composition and Radon lab-scale melting tests in alumina crucibles in a resistive furnace, a frit 503-R4 with chemical composition (wt.%): B_2O_3 – 16, Li_2O – 8, SiO_2 – 76, was an appropriate glass forming additive to vitrify the SB4 waste. Tests in small (50 mL) alumina crucibles showed that the waste/frit mixtures at waste loading of 40 to 60 wt.% yielded almost homogeneous glass. Traces of spinel type crystalline phase were observed only at the highest waste loading of 60 wt.% in glass.

Larger melt volume in the cold crucible may cause enhanced crystallization of glass yielding higher degree of crystallinity of the vitrified waste product [1,5]. In initial testing, two tests with U-bearing SB4 waste surrogate in a lab-scale (56 mm inner diameter) cold crucible were performed. According to the Contract**,** two 216 mm diameter CCIM tests with U-free SB4 waste surrogate in a bench-scale (236 mm inner diameter) cold crucible and one test in a large-scale (418 mm inner diameter) cold crucible were conducted to optimize process parameters to achieve maximized waste loading (60 wt % target) and throughput for the SB4 composition. Specific emphasis will be placed on minimizing cesium, halide and sulfate volatility from the melt.

Equipment and feed preparation

The test was conducted at the Radon full-scale liquid waste vitrification plant (Fig. 1) consisting of waste concentration system, feed preparation system, vitrification unit, off-gas system, energization, and auxiliary equipment. The melter - cold crucible is energized from a VCI-1.60/1.76 high frequency generator with vibration power of 160 kW and operation frequency of 1.76 MHz.

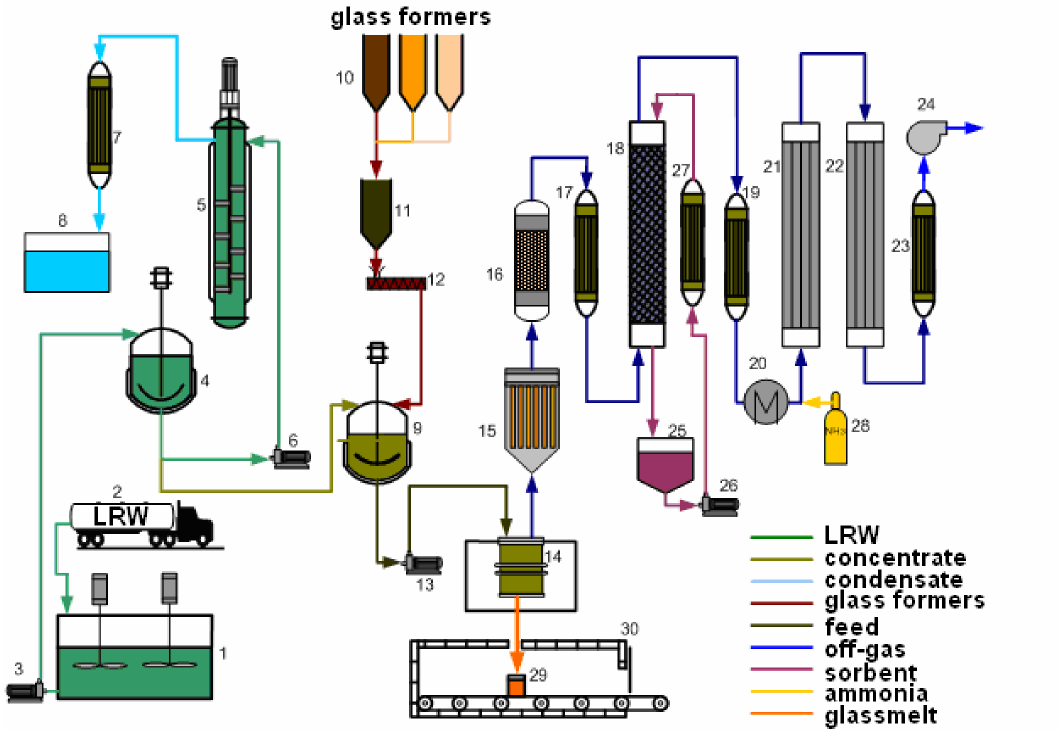

Figure 1. Schematic flowsheet of the Radon LILW vitrification plant (US Patent 6058741).

1 - storage tank, 2 – LRW transportation vehicle, 3, 6, 13, 26 - pumps, 4 – concentrate collector, 5 – rotary film evaporator, 6 - collecting tanks, 7, 17, 19, 23, 27 – heat-exchangers, 8 – condensate collector, 9 - mixer, 10 – glass formers bins, 11 – hopper, 12 – screw feeder, 13 - annealing tunnel furnace, 14 – cold crucible, 15 – coarse filter, 16 – glass fiber filter, 18 - absorbing columns, 20 - heater, 21 - catalytic reactor for NO$_x$ reduction, 22 – catalytic reactor for NH$_3$ oxidation, 24 – ventilator, 25 – vessel with sorbent, 28 – cylinder with ammonia, 29 – canister, 30 – annealing furnace.

Liquid radioactive waste (LRW) from an interim storage tank (1) is pumped into a concentrate tank (4) and concentrated in a rotary film evaporator (5) to a solids concentration of 1000 to 1100 kg/m^3. Vapor phase is cooled in a heat-exchanger (7) and collected in a tank (8).

The waste concentrate is fed into a mixer (9) where it is intermixed with glass-formers fed from bins (10) and hopper (11) using a screw (12). The slurry with a water content of ~25 wt.% is fed using a peristaltic pump (13) into the cold crucible melter (14). Molten glass is poured into canisters (29) and annealed in a tunnel furnace (30) to relax mechanical stress. Canisters with annealed glass are sent to a repository. Off-gas from the melter is purified from entrainment in coarse filter (15) and from aerosols and fine solids in a glass fiber filter (16), cooled in a heat-exchanger (17), and passes an absorption column (18). Gas containing traces of NO_x passes a heater (20) and is purified from residual NO_x followed by NO_x reduction with NH_3 and oxidation of residual ammonia using catalytic reactors (21) and (22), respectively. The purified gas after cooling to room temperature in a heat-exchanger (23) is discharged to the atmosphere.

In our test we excluded the first step of the process (LRW concentration) preparing the feed immediately in the mixer (9). We also used a simplified off-gas purification scheme where off-gas after purified on filters (15) and (16) was cooled in a heat-exchanger (17) and condensate was collected in a separate tank, whereas off-gas was discharged to the atmosphere.

The cold crucible design was the same as in our previous tests and described in detail elsewhere [1,5,9]. The 418 mm inner diameter and 860 mm height cold crucible was manufactured from the 12 mm outer diameter stainless steel pipes and equipped with a cover having feeding and off-gas ports, observation window, and a hole for the screw used to open the pouring unit (Fig. 2).

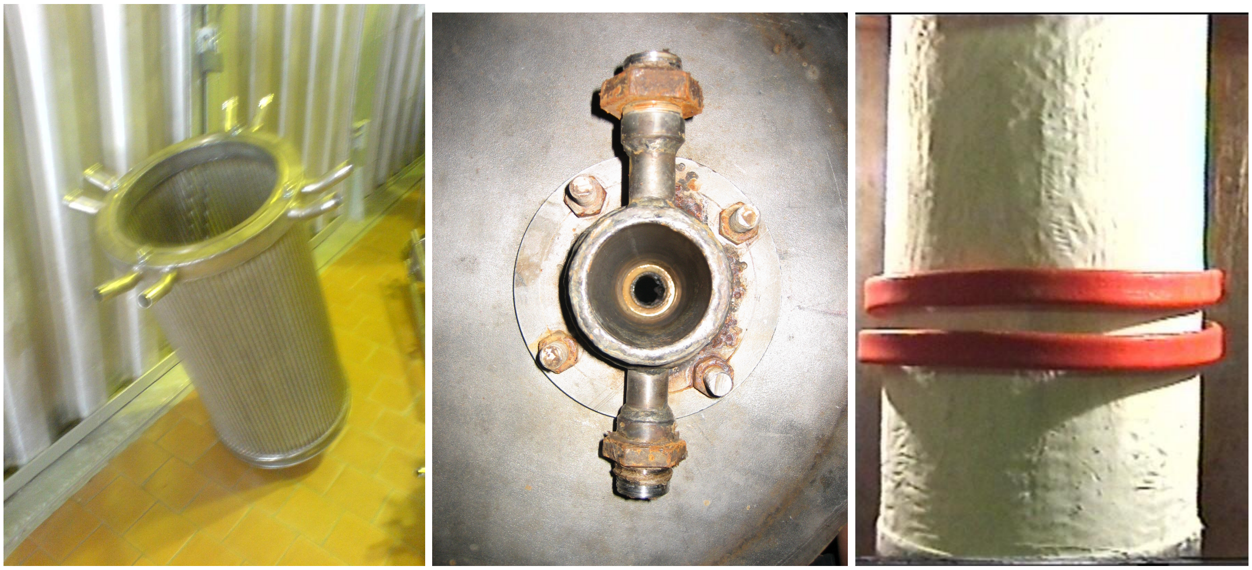

Figure 2. View of the cold crucible (left), pouring unit installed in the cold crucible (middle) and cold cruicble coated with a protective putty (right).

Two types of feed with target waste loading in glass of 50 wt.% were prepared. The first portion of the feed to produce a starting melt was prepared from chemicals: Al_2O_3, H_3BO_3, $CaCO_3$, $Ce(NO_3)_3 \cdot 6H_2O$, Cr_2O_3, Cs_2CO_3, Fe_2O_3, Li_2CO_3, MgO, MnO_2, Na_2CO_3, NiO, $PbSO_4$, Na_2SO_4, SiO_2. Water content in the feed was ~27 wt.% i.e. similar to conventional Radon LRW vitrification process.

The second portion of the feed was prepared from waste surrogate composed of chemicals (see above) and Frit 503-R4. In total 750 kg of feed with a water content of ~27 wt.% was prepared.

The third portion of the feed in amount of about 1,300 kg was prepared from waste surrogate obtained in accordance with a procedure SRT-MST-2007-00070 "Sludge and Glass Compositions for Cold Crucible Induction Melter (CCIM) Testing – Sludge Batch 4" [10] including 5 phases:

- Phase 1: Precipitation of MnO_2 [by the $KMnO_4$-$Mn(NO_2)$ reaction] followed by the precipitation of Fe and Ni as hydroxides.
- Phase 2: Analytical confirmation that the Mn, Fe and Ni meet the targeted specification.
- Phase 3: Washing the slurry prepared in Phase 1 with alkaline "inhibited wash water" until the soluble solids concentration in the aqueous fraction of the slurry is below 0.20 wt%. Inhibited wash water is water where NaOH and $NaNO_2$ (equimolar quantities) have been added to obtain a pH of 10.5±0.25.
- Phase 4: Addition of the remaining waste components and final adjustment of the total solids concentration.
- Phase 5: Analytical confirmation that the final slurry batch meets specifications described in Table I.

Table I. Expected Concentration of Anion/Cation Content of Sludge Feed Simulants (Final Product)

Cation	wt% total solids	wt % calcined solids	Cation	wt% total solids	wt % calcined solids	Anion	Concentra-tion, g/L
Al^{3+}	10.381	14.830	Mg^{2+}	1.287	1.839	SO_4^{2-}	1.386
Ba^{2+}	0.048	0.069	Mn^{2+}	3.447	4.924	PO_4^{3-}	0.284
Ca^{2+}	1.521	2.172	Na^+	10.679	15.256	CO_3^{2-}	7.449
Ce^{3+}	0.141	0.201	Ni^{2+}	1.004	1.434	SO_4^{2-}	1.319
Cr^{3+}	0.104	0.149	Pb^{2+}	0.273	0.390	NO_3^-	5.595
Cu^{2+}	0.031	0.045	Si	0.975	1.393	Cl^-	3.550
Fe^{3+}	15.602	22.289	Zn^{2+}	0.031	0.044	F^-	0.045
K^+	0.043	0.062	Zr^{4+}	0.052	0.074	I^-	0.085
						NO_2^-	13.762
						OH^-	5.158
Specific Gravity, g/mL			1.14	Total Organic Carbon (TOC) [wet]			<0.05
Total Solids, wt. %			20	Total Solids, g			273.6
Soluble Solids, wt. %			4.72%	Estimated Calcine Factor			0.7

A feature of the sludge prepared according to procedure [10] is high homogeneity and formation of magnetite-type spinel and nepheline phases at low temperatures (Fig. 3).

The waste surrogate was intermixed with Frit 503-R4 (produced by Ferro Corp., USA) having a chemical composition (wt.%): 8 Li_2O, 16 B_2O_3, 76 SiO_2 and the particle size distribution: ≤2.0 wt.% retained on a U.S. Standard 80 mesh sieve, ≤10 wt.% passing through a U.S. Standard 200 mesh sieve. Final water content in the feed was ~50 wt.%. In total 1,300 kg of feed was prepared. In order to evaluate Cs volatilization, $CsNO_3$ was added to the feed in amount to achieve 0.64 wt.% Cs_2O in the final glass.

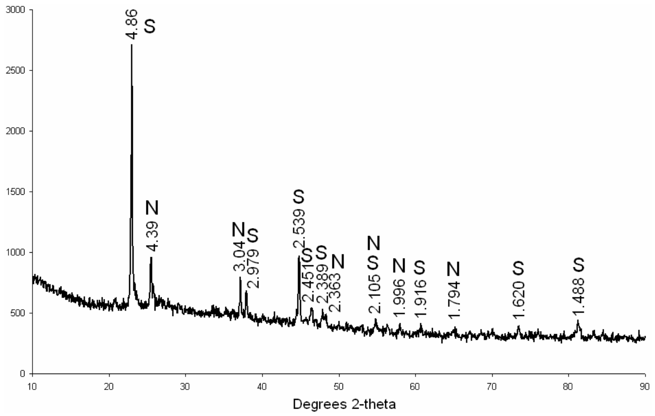

Figure 3. XRD Pattern of the Sludge Prepared by SRT-MST-2007-00070 Procedure and Air-Dried at 115 °C.

N – nepheline, S – spinel.

ICCM Process Description

Major steps of the run were as follows:

1) Starting melt production from chemicals;
2) Vitrification of waste surrogate with Frit 503-R4 at water content of ~27 wt.%;
3) Vitrification of waste surrogate prepared using SRT-MST-2007-00070 procedure [10] with Frit 503-R4 at water content of ~50 wt.%;
4) Process stopping and melter cooling.

In step 1, to start-up the CCIM process, the cold crucible was charged with the glass cullet produced in our previous test at the bench-scale unit (~50 wt.% waste loading) and about 6.5 kg of dry batch applied at the Radon vitrification plant. The starting melt was initiated through step-by-step increases of vibration power from 29.5 to 149 kW for 2 hrs. 25 min. Molten glass was poured into canister #1.

In step 2, a mixture of chemicals with water content of ~27 wt.% was fed for 29 hrs. 40 min. Slurry was fed into the crucible in amount of 46 to 90 kg followed by holding the melt to complete melting of feed components and melt homogenization. Then the pour gate was open and portion of the melt was poured into a ~12 L canister. During this step the melt was poured into canisters #2 to #15 and ~415 kg of glass was produced (Table 2).

Table 2. Major ICCM Process Variables under Steady-State Conditions

Process Variables	Step 2	Step 3
Test Duration, hr:min	29:40	32:50
Average Vibration Power, kW	121.6	134.1
Weight of Processed Feed, kg	745.8	1307.9
Weight of Produced Glass, kg	414.3	458.6
Average Feed Rate (Capacity), kg/hr	25.1	39.8
Average Glass Pour Rate (Productivity), kg/hr	14.0	14.0
Specific Energy Expenses for Feed Processing, kW×hr/kg	4.8	3.4
Specific Energy Expenses for Glass Production (Melting Ratio), kW×hr/kg	8.7	9.6
Specific Glass Productivity, kg/(m^2×d)	2453	2453

There was variation in input power during feeding and holding the melt to homogenize the glass (Fig. 4). Average value of input power at the step 2 was found to be ~122 kW (Table 2).

Major process variables given in Table 2 demonstrate high efficiency of the cold crucible during the melting process: average feed rate (capacity), glass pour rate (productivity) and specific glass pour rate achieved ~25 kg/hr, ~14 kg/hr, and ~2450 kg/(m^2×d) even with the variable process conditions.

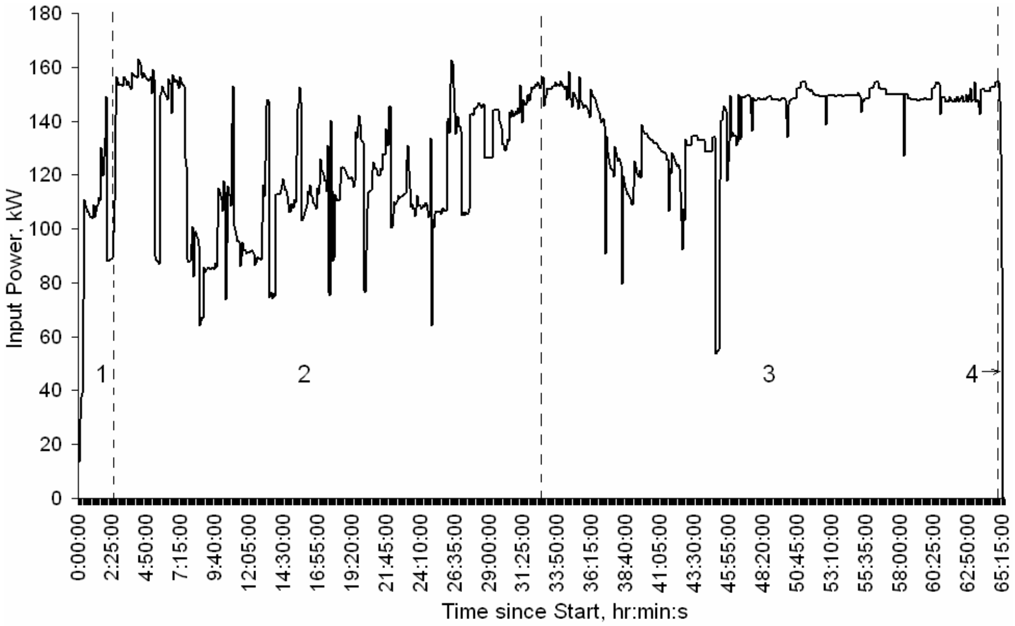

Figure 4. Variation of Input Power during the Starting Melt Production (1), Feeding of the Mixture of Chemicals and Frit (2), Waste Surrogate Prepared by SRT-MST-2007-00070 Procedure (3) and Process Stopping (4).

During step 3, the feed with Frit 503-R4 with increased water content (50-52 wt.%) was processed. Totally, the slurry in amount of ~1300 kg was fed and ~460 kg of glass was produced. Under these conditions, both glass productivity and specific glass productivity remained similar to the values for the step 2 at lower water content (~27 wt.%). This demonstrates advantages of the use of prepared waste surrogate and frit rather than chemicals as glass forming additives to increase melting rate. In spite of higher energy expenses on water evaporation, application of the feed prepared from waste surrogate and Frit 503-R4 compensates these expenses due to higher homogeneity of the feed and reduction in power expenses on silicate forming reactions. As a result, feed rate during step 3 was even higher (~40 kg/hr) than that during step 2, specific glass productivity remained nearly the same [~2450 kg/(m^2×d)], and melting ratio was increased by only a factor of ~1.1 (~10%)

Energy parameters of the CCIM process may be summarized as a heat power distribution diagram (Fig. 5). It is seen that about 60% of total power was spent on cooling of the generator's tube and cold crucible. Minor heat (up to ~4% of total) was removed with the off-gas. About 35% of total power was consumed on feed heating, water evaporation, calcine melting and melt homogenization.

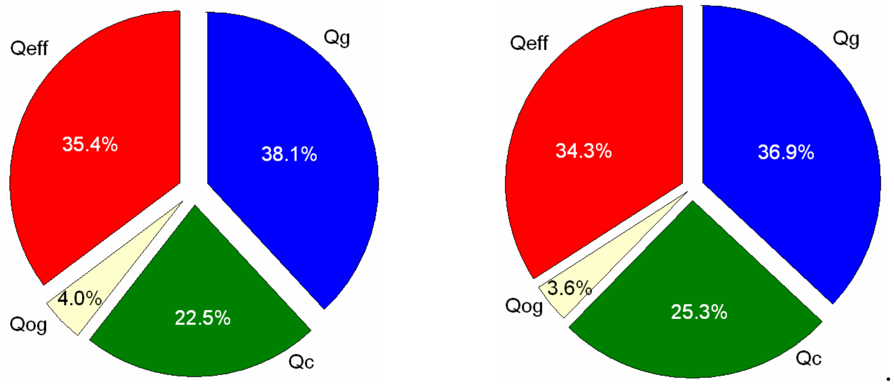

Figure 5. Heat Power Distribution at the CCIM Process at Treatment of Slurries with Water Contents of ~27 (left) and ~50 wt.% (right).

Qg is a power spent on cooling of HF generator, Qc is a power spent on cooling of the cold crucible, Qog is a power removed with off-gas, Qeff is a power spent on batch heating, melting and melt homogenization.

Off-Gas Analysis

The off-gas sampling system flowsheet is shown on Fig. 6. Major results are summarized in Table 3.

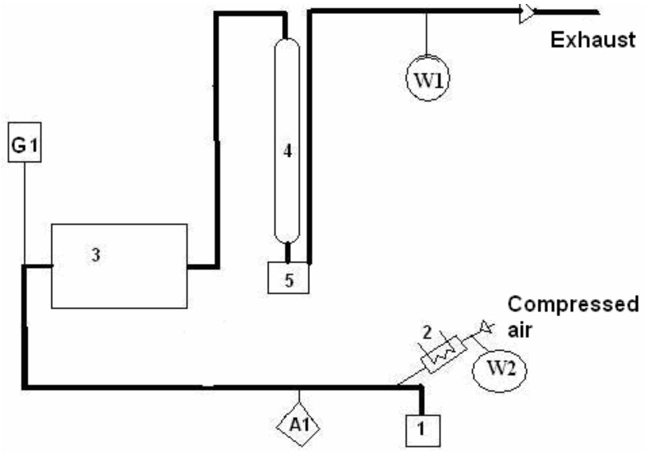

Figure 6. Flowsheet of Aerodynamic Measurements and Off-Gas Sampling Points.

1 – melter, 2 – air heater, 3 – coarse filter, 4 – cooler, 5 – condensate collector.

Measurement and sampling points: A1 – aerosol sampling, G1 – gas components sampling, W1 – off-gas flow rate measurement, W2 – compressed air flow rate measurement.

Volatilization of Feed Constituents

Volatilization of feed constituents was evaluated using two methods: 1) by measuring of elemental concentrations in off-gas using aerosol filters and 2) by difference between amounts of constituents (recalculated to oxides) fed in the cold crucible and found in glass. Both give significant error, therefore, only rough evaluation is possible.

Table 3. Average Concentrations and Variability of Off-gas Constituents

Parameters		Values
Off-gas temperature after dilution with compressed air, °C		150 - 207
Off-gas flow rate (25 °C, 1 atm), m^3/h		149 (72 - 234)
Concentration of components in off-gas	[1] NO. mg(N)/m^3	~0 - >3000
	[1] NO_2. mg(N)/m^3	~0 - >3000
	[1] CO. mg(C)/m^3	~0 - 50.0
	[2] HCl. mg/m^3	262 (26.2 - 846)
	[2] F^-. mg/m^3	1.32 (0.67 - 3.44)
	[2] SO_2. mg/m^3	132 (2.5 - 271)
	[3] Aerosols. mg/m^3	4410 (430 - 24100)

[1] "Kaskad-512" gas analyzer, [2] chemical analysis, [3] weighing procedure.

Major sources of error in determination of losses using the first technique are the variable off-gas flow rate and variation in elemental concentrations due to the semi-periodic mode of operation consisting of slurry feeding, melting/homogenization and melt pouring steps. Significant error in the second method of determination arises from differences in chemical composition of the glasses both in canisters and "dead volume" of the cold crucible and difficulties in precise determination of amount of the slurry fed in the cold crucible and amount of glass poured into canisters (unknown amount of glass was poured into a catcher under transportation system in the tunnel furnace). Results are given in Table 4.

Table 4. Oxide Losses from Cold Crucible by Various Methods of Measurement

Oxides	Loss, wt.%			
	XRF	EDS	ICP-AAS	OGA*
Li_2O	-	-	-	8.0
B_2O_3	12	-	7.3	4.5
Na_2O	3.5	7.9	2.6	6.7
Al_2O_3	0.1	-	0.3	0.3
SiO_2	-	-	-	0.1
CaO	0.5	-	4.3	2.4
MnO	3.9	5.1	7.1	5.3
Fe_2O_3	7.4	2.7	4.5	2.2
Cs_2O	56.3	48.4	71.9	61.6

* Determined by ICP-MS analyses of off-gas deposits on aerosol filters.

Considering data presented in Table 4 note, that analyses of aerosol filters in the off-gas system indicated much lower boron volatilization as compared with the value obtained from analyses of glass. Boron loss occurs in two ways: mechanical carry-over mainly in aerosol form and vaporization from the melt surface at high temperatures. Appreciable glass depletion with boron is due to both boron vaporization from the open melt surface in the cold crucible and mechanical carry-over of fine-grained frit. These mechanisms were responsible for the predominant fraction of the carry-over deposits on the inner cold crucible walls and off-gas pipe (Table 5). Only a minor fraction of boron as vapors and aerosols is volatilized.

Sodium losses determined from both off-gas and glass analyses are similar. XRF spectroscopy shows lower sodium loss than EDS but the value obtained from EDS is closer to that of the off-gas analysis.

Table 5. Chemical Compositions (wt.%) of Deposits on the Inner Surfaces of Cold Crucible Wall and Cover

Oxides	Wall Deposit	Cover Deposit	Oxides	Wall Deposit	Cover Deposit
Li_2O*	4.00	4.00	MnO	2.26	1.31
B_2O_3**	5.80	10.40	Fe_2O_3	22.37	13.52
Na_2O	8.55	22.94	NiO	3.85	2.40
MgO	1.29	1.37	CuO	0.00	0.00
Al_2O_3	20.52	4.31	ZnO	5.24	3.41
SiO_2	9.95	2.13	ZrO_2	0.07	0.02
P_2O_5	0.25	0.10	Cs_2O	1.45	4.59
SO_3	2.77	7.37	BaO	0.00	0.00
Cl	3.38	18.14	La_2O_3	0.00	0.00
K_2O	0.19	0.49	Ce_2O_3	0.00	0.00
CaO	1.58	2.25	PbO	0.75	0.51
TiO_2	0.00	0.00			
Cr_2O_3	0.34	0.31	Total	90.51***	95.57***

* Li and B are not determined by XRF spectroscopy, ** B was determined by potentiometric titration, *** analytical sum (without Li_2O), OH-groups may be present.

Cesium is the most volatile component. The values obtained from both off-gas (~60 wt.%) and glass analyses (~50-55 wt.%) are in rather good agreement. Some volatility was observed even for iron and manganese (Table 4 and 5). Probably, mechanical carry-over as aerosols was the major contribution to their volatilization. Nevertheless, as it has been shown in our previous work [11], appreciable losses of these and other iron group elements may be due to volatility of their chlorides that is consistent with analytical data for the wall and cover deposits (Table 5).

As follows from Table 5, if wall deposits are due predominantly to the mechanical carry-over, then vaporization of easily volatile constituents yields significant contribution to the cover deposit formation. The latter is strongly enriched with boron, sodium, cesium, sulfur, and chlorine. XRD data (Fig. 7) show that the wall deposits consisted of major spinel type phase and minor sodium chroride (halite, NaCl) and sulfate (Na_2SO_4 – hexagonal). Phase composition of the cover deposit is much more complex. It contained major sodium sulfate and chloride, with spinel secondary in abundance and minor sodium zinc sulfate - β-$Na_2Zn(SO_4)_2$ and possibly cesium sulfate. Cs ion is too large and unable to substitute for Na ion in the crystal lattice.

Spinel might occur in the deposits due to mechanical carry-over of the slurry droplets. XRD pattern (Fig. 3) demonstrates that the spinel (variety different from that occurred in the vitrified product) is present in the air-dried waste surrogate prepared by SRT-MST-2007-00070 procedure. Other volatiles (sodium chloride, sulfate, sodium-zinc sulfate, cesium sulfate) are the products of both aerosol carry-over and vaporization during calcination and melting and from open surface volatilization of the melt in the cold crucible.

Other feed constituents (magnesium, aluminum, silicon and calcium compounds) are not volatile although minor mechanical carry-over with gas-vapor flow occurs as well.

As follows from X-ray diffraction and scanning electron microscopy data, the product of SB4 waste vitrification is composed of major vitreous and minor (~10 vol.%) spinel structure (trevorite and zincochromite) phases. Vitrified waste characterization details will be presented in a separate paper.

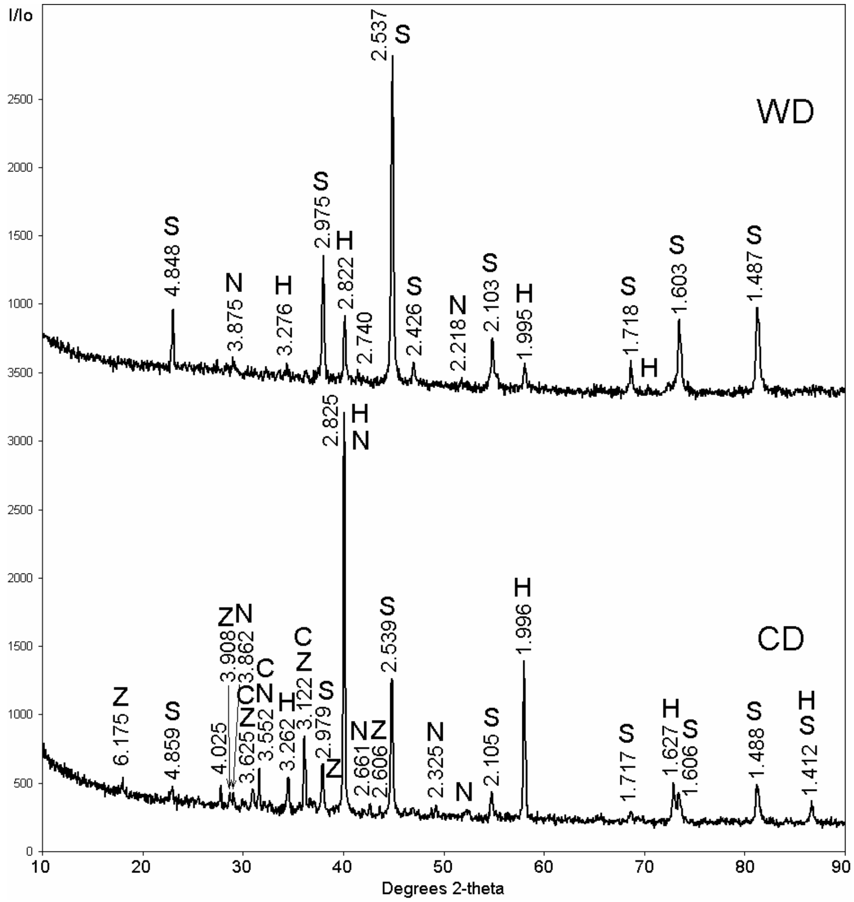

Figure 7. XRD Patterns of the Sublimates Deposited on the Inner Surface of the Cold Crucible Wall (WD) and Cover (CD).

C – $CsSO_4$, H – NaCl, N – Na_2SO_4, S – spinel, Z – $Na_2Zn(SO_4)_2$.

Conclusion

The full-scale cold crucible test using a 418 mm inner diameter stainless steel crucible was carried-out for 66 hrs. Commercially available Frit 503-R4 (8 wt.% Li_2O, 16 wt.% B_2O_3, 76 wt.% SiO_2) was used as a glass forming additive at a calcine to frit ratio of 1:1 (50 wt.% calcine, 50 wt.% frit). Two portions of slurry prepared from frit and mixture of chemicals simulating waste in amount of ~750 kg and from frit and waste surrogate prepared by the SRT-MST-2007-00070 procedure in amount of ~1,300 kg with water content of ~27 and ~50 wt.% was processed and ~415 kg and ~460 kg of the vitrified products was obtained. Major process variables achieved were as follows:

- Average Vibration Power, kW: 121.6 to 134.1;
- Average Feed Rate (Capacity), kg/hr: 25.1 to 39.8;
- Average Glass Pour Rate (Productivity), kg/hr: 14.0;
- Specific Energy Expenses for Feed Processing, kW×hr/kg: 4.8 to 3.4;
- Specific Energy Expenses for Glass Production (Melting Ratio), kW×hr/kg: 8.7 to 9.6;
- Specific Glass Productivity, kg/(m^2×d): 2453.

The product was composed of major vitreous and minor spinel structure phases (trevorite and zincochromite). No nepheline phase was found. Average degree of crystallinity was estimated to be ~12 vol.%. Cesium was found to be the most volatile component (~60 wt.% of total). Lithium, sodium and boron were less volatile. Other major feed constituents (Al, Si, Mg, Fe, Mn) were not volatile but mechanical carry-over with gas-vapor flow occurs.

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