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Processing of Oak Ridge B&C Pond Sludge Surrogate in the Transportable Vitrification System

by

J. R. Zamecnik

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

S. R. Young

D. K. Peeler

M. E. Smith

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Authors: John R. Zamecnik, David K. Peeler, Steven R. Young, Michael E. Smith

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PROCESSING OF OAK RIDGE B&C POND SLUDGE SURROGATE IN THE TRANSPORTABLE VITRIFICATION SYSTEM

John R. Zamecnik, David K. Peeler, Steven R. Young, Michael E. Smith
Westinghouse Savannah River Company
Savannah River Technology Center
Aiken, SC 29808

ABSTRACT

The Transportable Vitrification System (TVS) is a joule-heated, fully transportable melter system designed to demonstrate the treatment of low-level and mixed hazardous and radioactive wastes and is large enough to completely treat small to medium sized waste streams. An extended shakedown test of the TVS was completed in 1996 to demonstrate the processing of a surrogate wastewater sludge material. The surrogate waste was formulated to simulate the B&C Pond sludge waste from the Oak Ridge Reservation (ORR) K-25 Site. This surrogate contained several RCRA metals and a surrogate for depleted uranium.

The glass formulation used was soda-lime-silica (SLS), due to the high quantity of Ca in the waste. Glass forming materials were SiO_2, Li_2CO_3, and Na_2CO_3. While the melter was designed to produce 68 kg/hr of glass when slurry fed, improvements increased this rate to 86 kg/hr. Operational experience led to numerous improvements that increased the overall waste processing rate. During operation, the predicted glass viscosity was maintained between 44-62 poise. Glass produced was of acceptable durability compared to the Universal Treatment Standards for RCRA metals. The glass oxidation/reduction state was not found to influence the quality of the glass produced, even though redox increases due to the addition of organics were significant. The melter glass residence time was found to be approximately described by a well-mixed tank model with a plug flow time delay. The potential for devitrification of the glass melt during shutdown was minimized by flushing the melter with 1.5 melter turnovers of SLS glass.

INTRODUCTION[1,2]

Vitrification has been declared to be the "Best Demonstrated Available Technology" for heavy metals and high-level radioactive waste by the EPA. In the past, high capital costs have prevented vitrification from being considered as a viable option for treating low-level and mixed wastes (LLMW). By using a low cost, transportable unit, these up front capital costs can be significantly lowered and can actually result in lower life cycle costs (as compared to other existing technologies) for certain waste streams, especially if the resultant waste glass can be delisted from a LLMW to a low-level waste. The Transportable Vitrification System (TVS) was built for this reason. It is a large scale, fully integrated, vitrification system designed to treat LLMW in the form of soils, wastewater treatment sludges, incinerator ash, and similar material.

While the TVS is primarily intended for waste demonstration activities, it is large enough to completely treat waste streams of up to 500 000 kg. The Savannah River Technology Center (SRTC) procured the TVS per a Department of Energy (DOE) request and was designed and fabricated by EnVitCo (Toledo Engineering, TECO); SRTC provided substantial input to EnVitCo during the design process. The TVS was fabricated by Dreicor (a division of TECO) in Erwin, Tennessee. Acceptance testing was performed by EnVitCo and SRTC personnel, and then the TVS was shipped to Clemson, S.C. for more extensive testing.

The first stream chosen to be treated in the TVS is B&C Pond Sludge from the Oak Ridge Reservation (ORR) K-25 site, which is a sludge recovered from several wastewater ponds. Scoping tests on a smaller melter were first done at the Clemson University Environmental

Systems Engineering Department's DOE/Industry Center for Vitrification Research.[3] This smaller scale melter of similar design to the TVS Melter was used to successfully treat surrogate. B&C waste. Upon completion of these tests, SRTC personnel then ran extended tests of the TVS at Clemson using the same B&C surrogate waste composition. These tests produced about 11,500 kg of surrogate B&C waste glass. After these tests were completed, some equipment modifications were made, and the TVS was then shipped to ORR. Additional tests using surrogate B&C waste were also done at ORR to verify system integrity after reassembly and to train ORR operations personnel. Startup with mixed waste is scheduled to begin in June 1997 with either B&C Pond Sludge or a Central Neutralization Facility (CNF) sludge/B&C sludge mixture.

SYSTEM DESCRIPTION

The TVS consists of four modules which contain all the equipment required to perform vitrification. It was designed to be disassembled into containers which can be shipped on standard trailers. Fourteen trailers are needed to ship the entire TVS. Disassembly, transport, and reassembly takes about 4-6 weeks. The TVS requires a process area of about 25 m by 30 m. A schematic diagram of the TVS is shown in Figure 1.

In the waste and additives module, liquid or dry wastes and dry glass forming materials are added to the 900 L mix tank to prepare the feed for the melter. These materials are mixed to the proper proportions by weight; the mix tank weight is monitored by a set of load cells. Dry wastes are added directly to the mix tank via a screw feeder. Slurry or other aqueous wastes can be added via a liquid addition port using an external, customer supplied pumping system. Glass forming additives are delivered to the TVS in bulk bags which are hung over small hoppers. Metering screw feeders transport the additives from the hoppers to the mix tank. The feed slurry mixture is then sampled prior to transfer to the feed tank to verify the targeted composition. If analyses show the feed composition does not meet specifications, the feed can be remediated with additional additives.

The feed tank is similar in design to the mix tank. The slurry feed is recirculated through a loop which passes into the melter module, where a side stream is drawn off to feed the melter. The design production rate for the TVS is 68 kg/hr of glass produced when slurry fed. The waste and additives module is enclosed in a sealed container, which provides ventilation through HEPA filters. This system also maintains the module at a slight negative pressure to prevent inadvertent leakage of contaminated material. An airlock is provided for workers to dress out before entering the portion of the module containing radioactive materials. Surfaces inside the container are designed to allow easy decontamination.

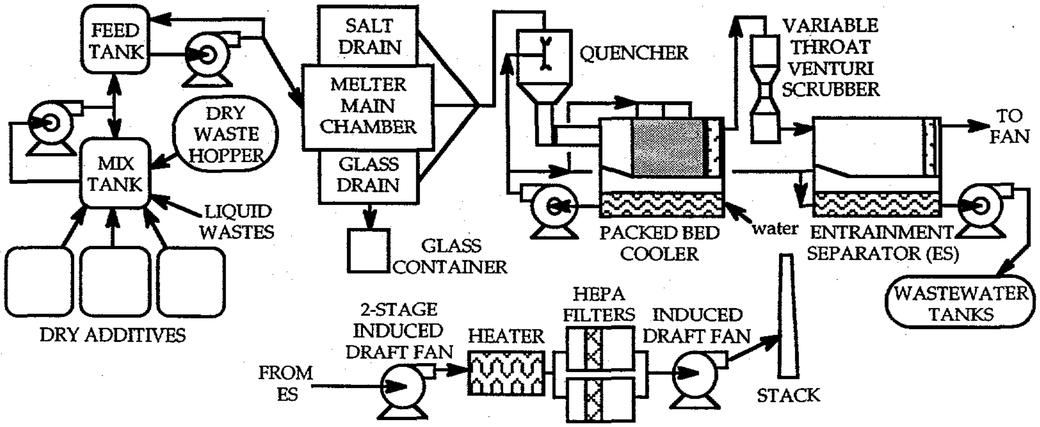


Figure 1. Transportable Vitrification System

The melter module contains the joule-heated glass melter and auxiliary equipment. The melter is divided into three refractory lined chambers. Slurried feed is introduced into the main, central chamber of the melter. Heat input is provided by molybdenum electrodes. The melter operates with glass temperatures in the range of 1100 to 1400°C. Control of the glass temperature is achieved indirectly by controlling the power to the joule heating electrodes. A propane gas burner supplies additional energy input into the main melt chamber plenum. The propane burner is used during cold startup of the melter to heat the solid glass in the melter to a temperature sufficient for joule heating to begin (ca. 700°C). The propane burner is also used during normal operation to maintain the melter plenum temperature; the TVS melter is a "cold top" melter, wherein the "cold cap" of feed slurry on top of the glass insulates the plenum from the heat of the glass, so additional energy input into the plenum is needed to maintain the offgas temperature. Operation with a cold cap also serves to limit volatilization of metals from the glass. Those metals that are not incorporated in the glass collect at the bottom of the central chamber where they can be drained off as needed.

Two side chambers are provided in the melter; the glass drain chamber contains a mechanical plug-type glass drain. Separate joule and plenum resistance heaters are provided to maintain the correct temperature in the glass drain chamber. Continuous glass production rates of 50 to 150 kg/hr can be maintained, depending on the type of feed used and the percent moisture in the feed. Glass is poured into either specially designed stainless steel containers or drums. The second side chamber contains a salt skimmer to remove molten salts from the glass surface. Accumulation of molten salts is not expected with most of the waste feeds planned for the TVS. This chamber also has separate plenum heaters and a drain.

The offgas module was designed by Andersen 2000 under contract to EnVitCo. Offgases from each of the three chambers of the melter are conveyed to the offgas system through a refractory lined pipe. Flow through the offgas system and melter vacuum is maintained by two induced-draft fans in this module. The other components of the module are the quencher, packed bed cooler, variable throat venturi, entrainment separator, heater, HEPA filters, and a 50 foot stack. The quencher cools the offgas by evaporative cooling and removes the coarser particulates. The packed bed cooler further cools the gas and removes some additional particulates from the gas stream; fresh water is added to the packed bed cooler. The variable throat venturi, which uses recirculated pH adjusted water, is designed to remove 95% of the particulates greater than 1 micrometer and the entrainment separator removes water droplets. A constant blowdown of water from the entrainment separator is used to maintain the solids content in the water below a maximum level. A reheater prevents condensation before the offgas enters the HEPA filters, which are designed to remove the remaining particulate to a total removal of 99.99%. The second fan maintains the HEPA filters at a slight negative pressure and sends the offgas into the stack. Sampling ports are provided at the stack for performing gas and particulate measurements in accordance with Environmental Protection Agency (EPA) methods. Continuous sampling for radioactive particulates is done by isokinetic sampling.

The final module is the control and services module which contains the melter power supplies and a control room. Most of the process is monitored and controlled from this module via a programmable logic controller interface and surveillance cameras.

SURROGATE WASTE FORMULATION

The actual ORR B&C Pond Sludge is a sludge material contains both waste sludge and excavated soil. RCRA metal components found in this waste include Cr, Ni, and Pb. The main radioactive component is depleted uranium. The surrogate was prepared from bulk technical grade chemicals, Indian red pottery clay, and a "metals spike" mixture (metal chlorides, nitrates, and acetates in HCl). The bulk chemical components were mixed in a large tumbler with the Indian red pottery clay. This clay was used since it is similar in composition to the excavated soil. The metals spike mixture contained minor component metals (Cr, Cu, Ni, Pb, Ce) in an acid solution. Cerium was the surrogate for uranium.

Table I shows target, calculated, and analyzed surrogate compositions. The calculated composition is the composition determined from a material balance on the masses of the components batched into the process. The calculated results are the average of the results for 24 batches of feed; the analytical results are the average of the analyses of nine of the batches from the mix tank. Small quantities of Ba and Zn (impurities in the clay), and Cl^-, NO_3^-, and acetate (from the metals spike solution) were also present. These data show that the analyzed composition of actual surrogate batches made matched the target values reasonably well. The most significant differences occur in the minor components; also, the presence of Li in the surrogate is the result of residual material in the mix tank from mixing the previous batch of surrogate with the glass formers.

The glass formulation for this waste was a soda-lime-silica (SLS) glass, which is the best glass choice for wastes containing significant amounts of calcium since it maximizes waste loading by taking advantage of the glass forming potential of the waste.[4] Borosilicate glasses were not chosen due to the large immiscibility gap in the CaO-B_2O_3-SiO_2 system. The glass forming additives used were silica, lithium carbonate, and sodium carbonate. Table II shows the targeted, calculated, and analyzed composition of the melter feed. The data show that the batching of the surrogate and glass forming materials into the TVS was also accurate, with the exception of Al, for which the actual concentrations were higher than the target. There are slight discrepancies when comparing the feed analyses with the glass analyses; the glass analyses for Li, Mn, Na and P were all somewhat different than the feed analyses, but were comparable to the target concentrations, indicating that there may be an analytical bias when analyzing these components in the feed. Again, the differences in the minor components were generally greater than the differences in the major components.

Table I. Target, Calculated, and Analyzed Composition of ORR B&C Surrogate

	Element Basis			Oxide Basis		
Element or Anion	Calculated (wt %)	Analyzed (wt %)	Oxide	Target (wt %)	Calculated (wt %)	Analyzed (wt %)
Al	2.350	2.437	Al_2O_3	5.400	5.356	5.842
Ca	13.15	11.92	CaO	23.97	22.19	21.16
Ce	0.091	0.106	Ce_2O_3	0.134	0.128	0.158
Cr	0.028	0.036	Cr_2O_3	0.045	0.049	0.067
Cu	0.027	0.029	CuO	0.047	0.041	0.046
Fe	4.647	4.339	Fe_2O_3	8.330	8.013	7.870
K	1.339	1.325	K_2O	2.060	1.945	2.024
Li	0.000	0.213	Li_2O	0.000	0.000	0.581
Mg	0.418	0.449	MgO	0.814	0.836	0.945
Mn	0.396	0.139	MnO_2	0.777	0.756	0.279
Na	0.260	0.837	Na_2O	0.246	0.422	1.431
Ni	0.172	0.163	NiO	0.276	0.265	0.264
P	0.334	0.327	P_2O_5	0.995	0.922	0.950
Pb	0.021	0.013	PbO	0.028	0.027	0.018
Si	22.62	21.28	SiO_2	56.21	58.36	57.77
Ti	0.339	0.278	TiO_2	0.667	0.681	0.589
$CO_3^{=}$	19.63	15.79				

The major anion present in the B&C sludge was carbonate ($CO_3^=$). Therefore, the calcium component of the surrogate was added as the carbonate. The lithium and sodium glass formers used were also carbonates. About 20% of the carbonates in the surrogate sludge and 35% in the melter feed were found to decompose prior to sampling. The acidic metals spike solution may have accounted for a small amount of this decomposition. The accuracy of the measured carbonate compositions was indirectly verified by measurements of the amount of CO and CO_2 in the offgas from the melter.

The total solids content of the melter feeds ranged from 39 to 61 wt%, while the density ranged from 1.3 to 1.6 kg/L. The processibility of these feeds depended greatly on the type of glass formers used. Several different types of glass formers were used, ranging from very fine (precipitated silica) to very coarse (sodium carbonate). Augering of the very fine precipitated silica was so difficult and the feed slurry made was so viscous that coarser silica was eventually substituted.

Table II. Target, Calculated, and Analyzed Composition of Melter Feed

	Element Basis			Oxide Basis			Glass
Element or Anion	Calculated (wt %)	Analyzed (wt %)	Oxide	Target (wt %)	Calculated (wt %)	Analyzed (wt %)	@ Three Melter Turnovers
Al	1.138	1.450	Al_2O_3	2.700	2.724	3.580	3.716
Ca	6.370	6.018	CaO	11.99	11.29	11.00	11.60
Ce	0.044	0.042	Ce_2O_3	0.070	0.065	0.065	0.079
Cr	0.014	0.019	Cr_2O_3	0.020	0.025	0.036	0.042
Cu	0.013	0.013	CuO	0.020	0.021	0.021	0.018
Fe	2.251	2.168	Fe_2O_3	4.170	4.076	4.050	3.822
K	0.649	0.720	K_2O	1.030	0.989	1.132	1.038
Li	2.644	2.881	Li_2O	7.500	7.209	8.103	7.308
Mg	0.203	0.251	MgO	0.410	0.425	0.544	0.519
Mn	0.192	0.069	MnO_2	0.390	0.384	0.142	0.374
Na	4.706	4.255	Na_2O	7.620	8.034	7.493	7.946
Ni	0.084	0.079	NiO	0.140	0.135	0.131	0.122
P	0.162	0.229	P_2O_5	0.500	0.469	0.687	0.451
Pb	0.010	<0.010	PbO	0.010	0.014	0.014	<0.010
Si	23.55	22.42	SiO_2	63.11	63.79	62.66	62.62
Ti	0.164	0.157	TiO_2	0.330	0.347	0.342	0.355
$CO_3^=$	26.92	17.51					

The particle size of the glass formers can affect the rate of melting in the melter and also the amount of waste that can be incorporated into a given glass composition. Smaller particle sizes for glass formers usually result in higher possible waste loadings and melt rates; however, the overall processing time suffered due to difficulties with augering the very fine precipitated silica and with highly viscous feed slurries that were difficult to pump. Therefore, a compromise between waste loading and feed processibility was needed. Use of a coarser silica improved augering and feed viscosity greatly, such that the overall processing rate increased. Additional improvement in the reliability of all of the glass former augers was also made by inserting solid cores into the augers.

MELTER OPERATION

The design glass production for the TVS melter is 68 kg/hr when slurry fed with an approximately 50 wt% slurry. Initially, a maximum overall glass production rate of only 50 kg/hr could be achieved due to lower than design melt rate. The vacuum in the melter could only be maintained at about 25-75 Pa due to a large amount of air inleakage (475 m^3/hr) into the main melter chamber. Modifications done between the surrogate runs at Clemson and at Oak Ridge reduced this inleakage to about 135 m^3/hr, increased the operating vacuum to 250-375 Pa, and resulted in an increase in the average plenum temperature during feeding of about 200 °C. The bottom line was an increase in the attainable steady state glass production rate from 50 kg/hr to 86 kg/hr.[5]

Since the melter was started up with an inventory of SLS glass, the composition and physical properties of the glass in the melter changed as the surrogate waste was introduced. The most significant impact on operation is the change in the glass viscosity as the less viscous B&C surrogate glass formed displaces the SLS startup glass. Since viscosity is a parameter of critical importance to both glass pouring and refractory life, the melter operating temperature was adjusted periodically during the transition from SLS to the B&C surrogate glass. The composition and viscosity of the glass in the melter was estimated by treating the melter as a well-stirred tank and using viscosity-composition correlations developed by the SRTC. Table III summarizes the predicted viscosity as a function of melter turnovers of B&C surrogate glass. Viscosities between 20 and 100 poise is considered to be acceptable for pouring; higher viscosities in this range are better for refractory wear. Viscosities in brackets [] in Table III show the approximate actual temperature and the corresponding viscosities during the transition; viscosities in braces {} show temperatures that would have resulted in viscosities outside the acceptable range.

Table III. Predicted Glass Viscosity (Poise) During the Transition from SLS to B&C Glass

Melter Turnovers	1150 °C	1200 °C	1250 °C	1300 °C	1350 °C
0	{172}	119	84	61	**[46]**
1/8	{143}	98	70	**[51]**	38
1/4	{120}	83	**[59]**	43	32
3/8	{103}	71	**[50]**	37	27
1/2	89	**[62]**	44	32	24
5/8	79	**[54]**	39	28	21
3/4	71	**[49]**	35	25	{19}
7/8	64	**[44]**	31	23	{17}
1	**[59]**	40	29	21	{15}

Crystallization or devitrification in the melter can result in accumulation of a viscous layer on the bottom of the melter which could interfere with glass pouring or lead to localized corrosion. To mitigate the adverse effects of crystallization, the melter was maintained at a temperature of at least 100°C above the liquidus temperature predicted as a function of composition. The refractory was inspected following the demonstration and only minor wear was observed.

The maximum steady-state glass production rate was found to be limited by the maximum melt rate (86 kg/hr glass) that could be achieved in the melter. The maximum instantaneous rate of glass pouring achieved was about 365 kg/hr. However, the glass pouring rate should be limited to reduce the potential for formation of devitrified glass in the glass container due to slow cooldown. Pouring at up to 140 kg/hr showed no evidence of devitrification.

Joule heated glass melters are often assumed to be well mixed, such that their residence time distribution can be described by the continuously stirred tank reactor (CSTR), or well-mixed tank model. Several residence time distribution models for the concentrations of iron and lithium, which were not present in the SLS glass cullet used to initially charge the melter, were fit to the TVS data. Figure 2 shows the concentrations of Li as a function of melter mass turnover.

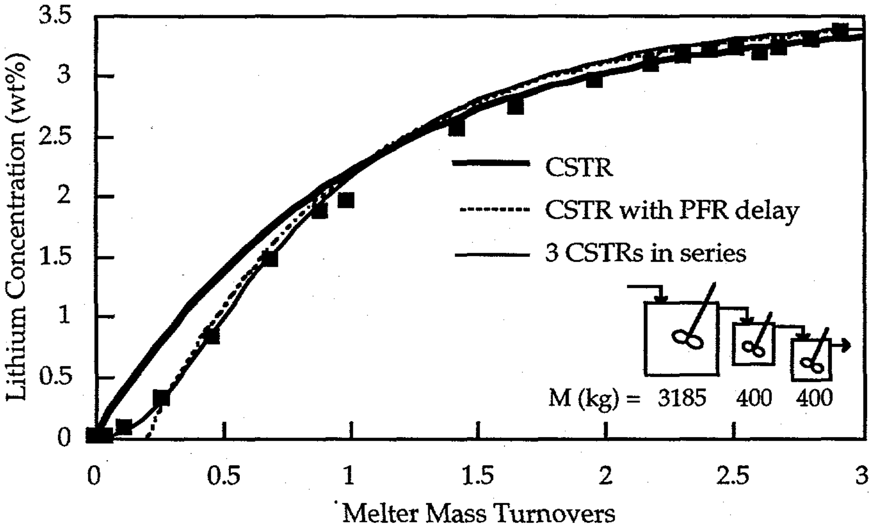
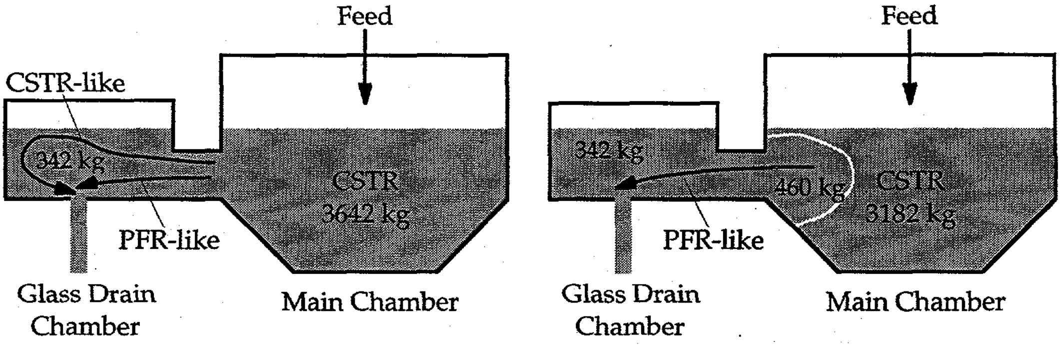


Figure 2. Lithium Concentration in Melter

The CSTR model was found to not fit the data as well as models with a plug flow reactor (PFR) delay time or with three reactors in series. The physical layout of the melter is consistent with these models. The melter consists of the large main chamber, which is likely to behave like a CSTR. The glass from the main chamber passes through a throat block to the glass drain chamber, from which it is poured. Figure 3a shows a simple interpretation of the possible glass flow patterns.



(a) Expected (b) Based on Models

Figure 3. Residence Time Behavior of the TVS Melter (not to scale)

The flow through the glass drain chamber would be expected to be somewhere between a PFR and a CSTR. This chamber contains joule electrodes that would tend to promote some convective mixing, but the lack of a cold cap and the short residence time significantly limits the actual convection. The best model (3 CSTRs in series) has one large CSTR (main chamber) followed by two smaller equal sized CSTRs in series that approximate a non-ideal PFR with substantial back mixing,[6] as shown in Figure 2 for the Li data. One would expect that the predicted mass of the main chamber CSTR would be equal to the actual mass in the main chamber; however, the best fit of the data gives about 460 kg less than the actual amount, which may indicate that the region of the main chamber near the passage to the glass drain also has PFR behavior, as shown in Figure 3b. Figure 4 shows data for most of the other elements and fitted curves for the CSTR with PFR delay model. The fitted curves use the CSTR and PFR volume determined from the Li data.

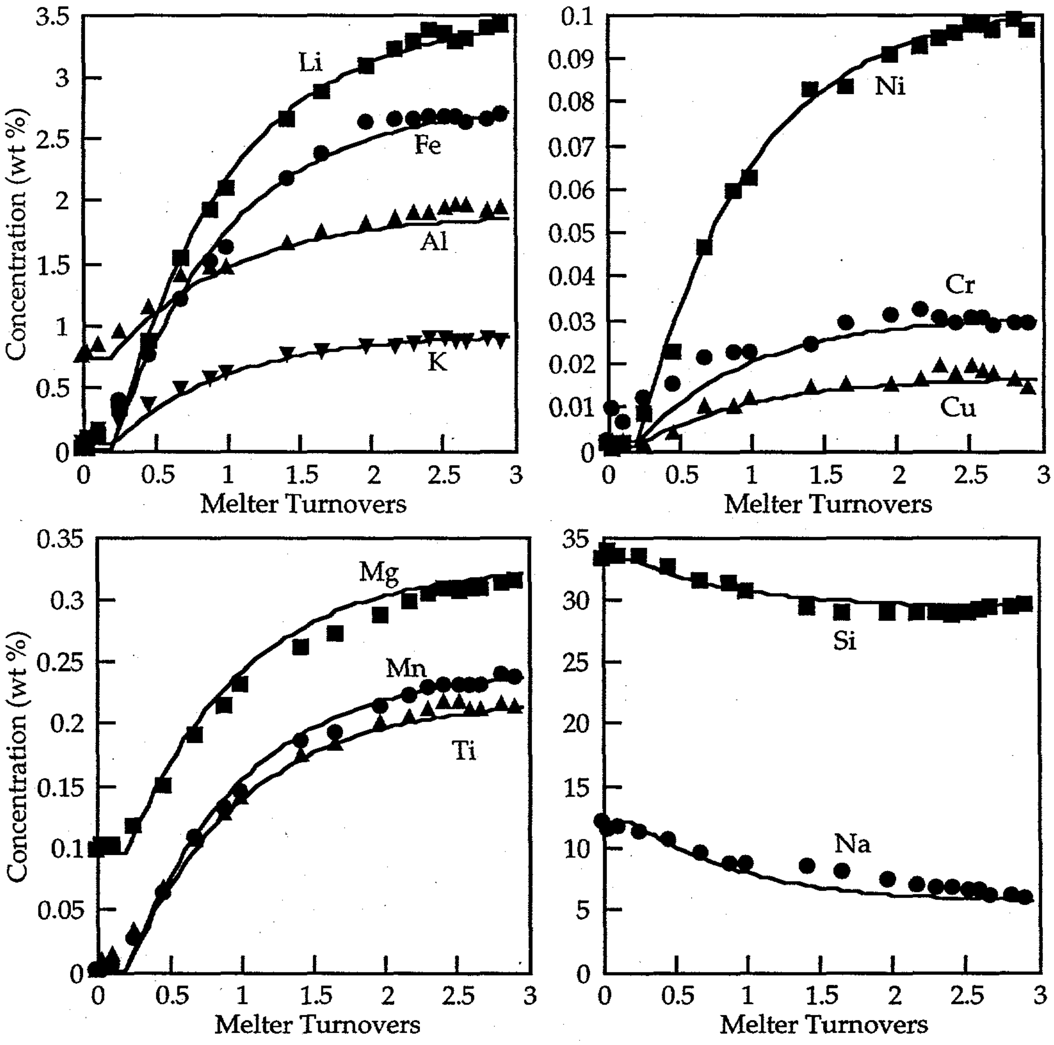


Figure 4. Melter Glass Composition Fit by CSTR-PFR Model

Although the melter does not behave exactly as a CSTR, the concentrations of all components at three melter turnovers are very similar to the concentrations that would be seen with true CSTR behavior and the CSTR model is essentially as accurate as the more complicated models at greater than one melter turnover.

The durability of the glasses produced was also of concern, as the B&C Pond sludge is a listed waste as defined in the Land Disposal Restrictions[7] (LDRs) for hazardous waste. Specifically, it is listed as F006 waste, which is for wastewater sludges from electroplating operations. The constituents of concern in F006 wastes are Cd, Cr, Ni, Ag, and cyanides; of these, B&C Pond sludge contains Cr, Ni, and Ag; this sludge also contains the RCRA metals Ba and Pb. Glass pour grab samples and samples from the tops of glass containers were taken and their durability measured with the EPA Toxicity Characteristic Leaching Procedure (TCLP).[8] Based on the TCLP analyses of all the samples, the glass produced was judged to have acceptable durability as defined by the EPA Universal Treatment Standards (UTS).[9,10] Table IV summarizes the TCLP results and compares them to the UTS limits. The metals placed in the B&C Ponds sludge surrogate that are addressed by the LDRs were Cr, Ni, and Pb. Although not intentionally added to the surrogate, trace amounts of Ba and Zn, present as impurities in the Indian red pottery clay, gave measurable TCLP responses.

Table IV. TCLP Results for Glasses Produced

	Measured Values (mg/L)		
Metal	Minimum	Maximum	UTS Limits
Ba	<0.01	0.29	7.6
Cr	<0.02	<0.02	0.86
Ni	<0.01	<0.01	5.0
Pb	<0.10	<0.10	0.37
Zn	0.17	0.36	5.3

Crystallization in the TVS melter upon melter shutdown was also a concern. Devitrification of the remaining melt pool upon shutdown could cause severe problems with melter restart. To evaluate the devitrification potential of the melt pool, representative glass samples were obtained from the glass poured as the melter was transitioned from the startup inventory of SLS glass to the glass containing the B&C surrogate. Samples were taken at approximately 0, 0.5, 1.0, 1.5, 2.0, and 3.0 melter turnovers.

These samples were subjected in the laboratory to a thermal cooldown profile that approximated the cooldown data recorded from the TVS during a cooldown with SLS glass. The profile used was based on the temperature at approximately the center of the main melter chamber, so this profile is near the slowest cooldown rate, and thus is conservative. X-ray diffraction analysis on the as-received (pre-heat treatment) glasses showed no indication of devitrification.

Results of the thermally heat treated glasses showed that the high SLS glass (initial inventory) was "thermally stable" with respect to devitrification when the melter cooling curve was applied while the high B&C glass at three melter turnovers partially devitrified. The majority of the crystallization associated with this last sample was surface devitrification. At least three crystalline products were identified by x-ray diffraction: sodium aluminum silicate (nepheline), lithium silicate, and calcium silicate (wollastonite). With the exception of the glass at 0.5 melter turnovers, all intermediate glasses were x-ray amorphous after heat treatment. Nepheline was detected in the 0.5 melter turnover sample after heat treatment , but semi-quantitative analyses suggested an extremely small quantity.

Based on these results, it was recommended that the melter be flushed with at least one melter turnover of SLS glass to avoid or minimize devitrification in the melter. The melter was then flushed with 1.5 melter turnovers of SLS glass before shutdown, and was then successfully restarted at Oak Ridge.

Samples of the glass product poured from the melter were characterized for their oxidation/reduction (redox) state. Since the mixing in the melter can be approximately described as a well mixed tank, the glass poured from the melter is considered to be a fairly representative sample of the bulk contents of the melter. The redox state of the glass was monitored by measuring the ratio of Fe^{+2} to the total iron ($\Sigma Fe = Fe^{+2} + Fe^{+3}$), or $Fe^{+2}/\Sigma Fe$. Figure 5 shows the redox ratio as the melter glass inventory transitioned from SLS to B&C and the later parts of the demonstration when organics were added to three batches.

For three batches, 1 wt% dry basis petroleum oil, 3 wt% dry basis phenol, and 3 wt% dry basis petroleum oil were added to the feed at approximately 2.4, 2.5 and 2.8 melter turnovers, respectively. The petroleum oil used was a hydrotreated heavy naphthalenic grade lubricating oil with an approximate molecular weight of 480 and a boiling point of 300 °C. These organics were added to estimate combustion efficiency in the melter. As Figure 4 shows, the transition from SLS glass to the B&C surrogate glass did not have a significant effect on the redox state of the glass. The addition of the organics, as expected, had a considerable effect. Each 240 gallon feed batch is approximately equivalent to 1/8 of a melter turnover. The three organic additions had the cumulative effect of raising the redox ratio from 0.08 to 0.29. Although the glass became more reduced with the addition of the organics, there was no impact processibility or product quality. It should be noted that if these levels of organics had been added throughout the demonstration, the redox ratio would have likely increased to unacceptable levels.

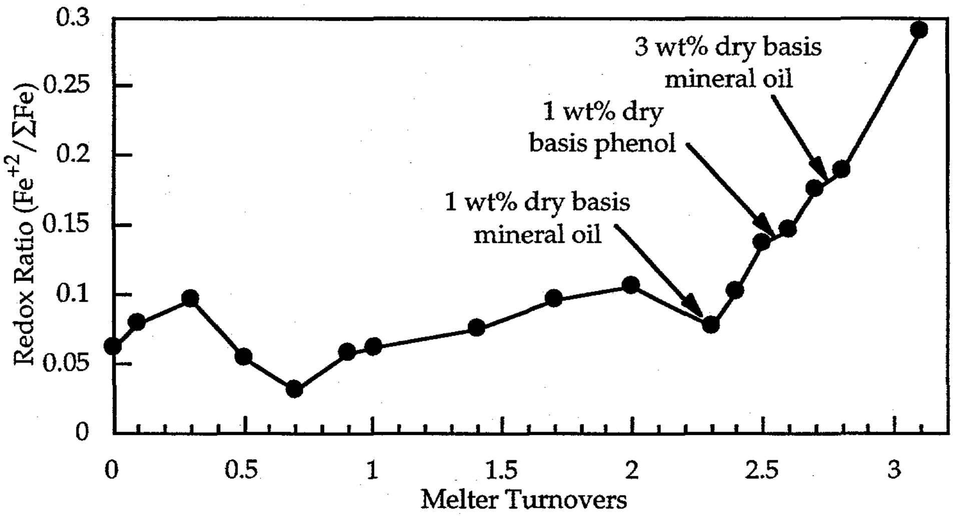


Figure 5. Redox of Glass Produced

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