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PROCESSING CONSTRAINTS ON HIGH-LEVEL
NUCLEAR WASTE GLASSES FOR HANFORD
WASTE VITRIFICATION PLANT

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PROCESSING CONSTRAINTS ON HIGH-LEVEL NUCLEAR WASTE GLASSES FOR HANFORD WASTE VITRIFICATION PLANT

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ABSTRACT

The work presented in this paper is a part of a major technology program supported by the U. S. Department of Energy (DOE) in preparation for the planned operation of the Hanford Waste Vitrification Plant (HWVP). Because composition of Hanford waste varies greatly, processability is a major concern for successful vitrification. This paper briefly surveys general aspects of waste glass processability and then discusses their ramifications for specific examples of Hanford waste streams.

GENERAL CONSIDERATIONS

Property Limits

Processability of a waste material to be vitrified is limited by the melting technology available. Processability conditions for Savannah River melter are discussed by Bickford et al. (1990a and 1990b) and for HWVP melter by Piepel et al. (1993). Because of limitations imposed by melt processability and glass acceptability, the following upper and lower bounds restrict the melt temperature (T), viscosity (η), electrical conductivity (ϵ), liquidus temperature (T_L), and glass dissolution extent (r) measured by a standard test:

$$T < T_{Max} \quad (1a)$$

$$\eta_{Min} < \eta(T_{Max}) < \eta_{Max} \quad (1b)$$

$$\epsilon_{Min} < \epsilon(T_{Max}) < \epsilon_{Max} \quad (1c)$$

$$T_L < T_{Min} \quad (1d)$$

$$r < r_R \quad (1e)$$

Here T_{Max} and T_{Min} are the maximum and minimum temperatures in the melter; η_{Min} and η_{Max} are melt viscosity limits at T_{Max} ; ϵ_{Min} and ϵ_{Max} are melt electrical conductivity limits at T_{Max} ; and the subscript R stands for the reference glass. Condition (1a) imposes a safe maximum temperature of melting ($T_{Max} = 1150^\circ\text{C}$ for a melter with Inconel electrodes). Condition $\eta(T_{Max}) < \eta_{Max}$ guarantees sufficient rates of bubble removal from the melt, melt homogenization in the melter, and melt pouring into the canister; condition $\eta(T_{Max}) > \eta_{Min}$ is necessary to limit melter corrosion. Condition $\epsilon(T_{Max}) > \epsilon_{Min}$ is imposed by the conductivity of furnace walls and $\epsilon(T_{Max}) < \epsilon_{Max}$ by the maximum current density tolerated by the electrodes. Condition (1d) prevents accumulation of solid materials in the melter. Inequality (1e) states that the dissolution extent the waste glass experiences in a standard durability test does not exceed that of a reference glass under identical conditions. It is a condition for glass acceptability rather than melt processability, but inequality (1e) cannot be dropped from the list because all conditions are mutually coupled through glass composition.

Condition (1d) can be ignored if the flow of melt through the melter is fast enough to prevent settling of solids (stirred melters), if the concentration of solids is very low (noble metals), or if the solid particles are very small (of the order of $1 \mu\text{m}$ or smaller) and of a similar density as the melt. However, excessive settling of conductive materials (RuO_2 , Rh, Pd) that can cause electric shorting must be prevented. Crystallinity in the glass product is of little concern if the overall chemical durability is high. Another concern is formation of immiscible melts produced by molten salts, such as phosphates, sulfates, or molybdates. These salts tend to form a separate layer at the bottom (molybdates) or on the top (phosphates, sulfates, or chlorides) of the melt. Formation of these layers should be avoided to prevent melter corrosion, excessive volatilization, and the potential for a steam explosion.

In summary, conditions (1a-d) define waste processability. The waste is processable if requirements (1a-d) can be met by adjusting

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melt composition. Condition (1e) defines glass acceptability if the durability test used to determine r is approved for the repository.

Glass Forming and Modifying Additives

Two basic waste glass families exist: borosilicate glasses and phosphate glasses. The former family is the best presently known choice for wastes with low levels of phosphates. Most of the Hanford high level nuclear wastes fit in this category. Borosilicate glasses are produced by adding to the waste sources of SiO_2 , B_2O_3 , and alkali oxides (Li_2O or Na_2O). Small additions of alkali earth oxides (MgO and CaO) and Al_2O_3 may also be used to lower T_L and reduce dissolution. While SiO_2 endows the glass with high resistance against dissolution, B_2O_3 and alkalis adjust the melt properties (η , ϵ , and T_L). The most effective oxide to adjust electrical conductivity is Li_2O (if Na_2O is used instead of Li_2O , the resulting glass has a lower durability).² Some B_2O_3 is needed to adjust viscosity independently of electrical conductivity, to keep T_L at a low level, and to enhance the glass forming tendency of the melt.

Property Models

Mathematical functions that express relationships between glass composition and glass properties are called property models. Property-composition relationships and mass balance equations for individual components are combined and solved to find the amounts of SiO_2 , B_2O_3 , and Li_2O that must be added to the waste to make a processable and durable glass.³ Property models help to reduce extensive laboratory testing and melter runs. However, laboratory studies and melter runs are necessary to check model predictions and to investigate those phenomena that are not included in the models, such as melting reactions or settling of solids. Property-composition models also simplify blending optimization for different waste streams and facilitate immediate response to processing problems during melter operation.

Waste Loading

The important criterion for vitrification economy is the mass fraction of waste in the waste glass, called waste loading. If the waste contains a large fraction of SiO_2 , its loading in glass can be high because little or no SiO_2 needs to be added. If the waste contains a large fraction of Na_2O , the waste loading is limited by glass durability because it is difficult to make a durable glass containing more than about 25% Na_2O . If the waste contains a high fraction of Cr_2O_3 , Al_2O_3 , ZrO_2 , or other refractory oxides, the waste loading is likely to be limited by liquidus temperature. It is unlikely that viscosity or electrical conductivity would limit waste loading because these properties are easy to adjust by additions of B_2O_3 and M_2O ($M = \text{Li}, \text{Na}$).

VITRIFICATION OF HANFORD HIGH LEVEL NUCLEAR WASTES

The remainder of this paper will show in some detail results of calculating fractions of glass forming and glass modifying additives

² This argument is discussed in greater detail in a paper by Irma, P., and R.J. Robertus. 1993. Waste Glass Design Based on Property Composition Functions, submitted to Am. Ceram. Soc. Bull.

³ Property models for HWVP glasses are discussed by Irma, P., and R.J. Robertus. See footnote 2.

to make processable and acceptable waste glasses from selected Hanford Waste streams. These results are preliminary. Estimates of waste stream compositions, pretreatment methods, property models, frit and glass design techniques, and melter design are continuously being improved. The main purpose of this paper is to demonstrate the usefulness of property composition models for developing durable waste glasses that will satisfy melt processability requirements.

Three examples of Hanford high level nuclear wastes will now be discussed (May and Watrous, 1992): Complexant concentrate waste (CCW), Plutonium finishing plant waste (PFPW), and Neutralized cladding removal waste (NCRW). The discussion is based solely on calculated compositions of processable and acceptable waste glasses using first-order property models.⁴ The purpose of these preliminary calculations is to detect possible problems with the vitrification of these wastes.

Complexant Concentrate Waste

The first column in Table 1 presents an estimated composition of the CCW from East Area tanks pretreated by destruction of organics and sludge washing to decrease the content of readily soluble components. Mass fractions (w_i) of nine major components are shown in the top portion of the table. The total mass fraction of the remaining 30 or so components is under the heading "others." "Others" include radionuclides and troublesome components, namely Cr_2O_3 , F , P_2O_5 , SO_3 , and noble metals. Mass fractions of Cr_2O_3 , P_2O_5 , and SO_3 are shown in the middle portion of the table. Troublesome components do not significantly affect glass properties as long as their concentrations are low, but they can cause cumulative damage during a long time of melter operation.

The recycle represents a small addition produced from process waste treatment and canister decontamination (Watrous et al., 1991). Recycle composition generally depends on the waste and frit compositions. To keep things simple, mass fractions (r_i) of the recycle components were not determined for each waste stream in this study. Instead, a generic composition, shown in the second column Table 1 (originally estimated for so-called neutralized current acid waste—see May and Watrous, 1992), is used for all wastes. This simplification does not affect the general purpose of this paper to discuss aspects of waste glass processability. The ratio of recycle to waste was 0.122 for all wastes.

Frit and glass compositions (the remaining columns in Table 1) were calculated using mass balances and mathematical models for viscosity and electrical conductivity (Redgate et al., 1993). Frit composition was to provide waste glass with $\eta = 4$ to 9 Pa.s and $\epsilon = 20$ to 40 S/m, both at 1150°C. With viscosity and electrical conductivity fixed at these levels, the waste loading can be determined by setting the value of liquidus temperature. The maximum waste loading corresponds to $T_L = T_{\text{min}} = 1050^\circ\text{C}$. Because the liquidus temperature model was under development and thus not available at the time when the calculation was performed, the maximum waste loading was estimated using two composition constraints:

⁴ See footnotes 2 and 3.

⁵ The constraints W_1 and W_2 are called reduced waste loadings because they do not consider the basic glass matrix components SiO_2 , B_2O_3 , Na_2O , and Li_2O (W_1) or these oxides plus CaO and MgO (W_2) even if these oxides are present in the waste. For example, a waste containing a high level of SiO_2 may allow a very high waste loading because less SiO_2 is needed in frit, but the reduced waste loadings will not be affected.

TABLE 1. WASTE GLASS COMPOSITIONS (IN TERMS OF MASS FRACTIONS) FROM CCW EAST AREA TANKS PRETREATED BY DESTRUCTION OF ORGANICS AND SLUDGE WASHING.

Component	Waste ¹	Recycle	Example 1		Example 2	
			Frit	Glass	Frit	Glass
SiO ₂	0.1750	0.4368	0.7510	0.5176	0.6606	0.5837
B ₂ O ₃			0.1577	0.0905	0.1848	0.1537
Na ₂ O	0.2510	0.4253		0.1151	0.0300	0.0704
Li ₂ O			0.0912	0.0523	0.0746	0.0621
CaO	0.0238	0.0030		0.0092		0.0036
MgO	0.0149	0.0032		0.0058		0.0023
Fe ₂ O ₃	0.0957	0.0107		0.0369		0.0146
Al ₂ O ₃	0.2900	0.0410		0.1121	0.0500	0.0858
ZrO ₂	0.0065			0.0025		0.0010
Others	0.1431	0.0801		0.0581		0.0229
Cr ₂ O ₃	0.0327			0.0124		0.0049
P ₂ O ₅	0.0051			0.0019		0.0006
SO ₃	0.0061			0.0023		0.0008
Viscosity at 1150°C (Pa.s)				8		8
Electrical conductivity at 1150°C (S/m)				40		30
Waste loading				0.38		0.15
W ₁ ²				0.22		0.13
W ₂ ³				0.21		0.12
MCC-1 28-day normalized B release (g/m ²)				7.7		10.1
PCT 7-day normalized B release (g/m ²)				0.31		1.01

¹ CCW, East Area, Sludge Washing Pretreatment.

² Sum of the mass fractions of all oxides except SiO₂, B₂O₃, Na₂O, and Li₂O.

³ Sum of the mass fractions of Fe₂O₃, Al₂O₃, ZrO₂, and "others."

(A) The difference between 1 and the sum of mass fractions of SiO₂, B₂O₃, Na₂O, and Li₂O, called reduced⁵ waste loading W₁, must not exceed 0.24.

(B) The sum of mass fractions of Fe₂O₃, Al₂O₃, ZrO₂, and "others," called reduced waste loading W₂, must not be larger than 0.22.

These constraints have been established empirically, being estimated from laboratory scoping studies with numerous glass compositions. In combination, they roughly delineate glass compositions which have fairly high probability of being crystal-free at 1050°C. Once the liquidus temperature models are available, the reduced waste loading constraints, W₁ and W₂, will no longer be necessary, and the waste loading will be predictable with a higher confidence. Until then, experimental testing will be required to check estimates based on the composition constraints.

The bottom portion of Table 1 summarizes the calculated values of glass properties, the estimated waste loading,⁶ and the reduced waste loadings. The properties shown are viscosity, electrical conductivity, and extent of glass dissolution measured as the mass of glass dissolved from a unit surface area (which is identical to normalized boron release)⁷ by two standard tests, MCC-1 (Material Characterization Center) and PCT (Product Consistency Test).

⁶ Waste loading is the mass fraction of the waste material (oxides) in the glass. Recycle is not included.

⁷ The boron release from a unit surface area of glass divided by the fraction of boron in glass, usually calculated on mass basis.

Two examples of glass composition are shown in Table 1. The reduced waste loading limits resulted in the actual waste loading of 0.38 in Example 1. This waste loading seems realistic because none of the mass fractions of the components that produce crystallinity in glass, such as Fe₂O₃, Al₂O₃ and ZrO₂, are excessively high. However, because of the present lack of a liquidus temperature model, laboratory tests for crystallinity are recommended for a final check.

The high content of Cr₂O₃ in the Example 1 glass (1.2 wt%) is a concern because this quantity of Cr₂O₃ will probably not dissolve in the glass completely or will generate spinel with FeO that is produced from Fe₂O₃ at melting temperatures. If Cr₂O₃ or spinel particles are very small (< 1 μm), they may not settle in the melter in an intolerable quantity. A small settling will not cause problems because Cr₂O₃ is one of the refractory components of furnace walls and thus will neither cause excessive corrosion nor form a conductive layer. Example 2 shows waste and frit composition when the concentration of Cr₂O₃ is reduced to a conservative value of 0.5 wt%. Then the waste loading is decreased to a very low value of 0.15, which would substantially increase vitrification cost.

Note that it is crystallization (liquidus temperature), i.e., a processing property, and not glass dissolution extent, i.e., a product property, that limits the maximum waste loading for this particular waste. This leaves considerable room for improvement of the processing technology. If an advanced technology is developed that would allow making glasses with higher liquidus temperature, the waste

loading can be increased until the durability limit (expressed as a dissolution extent, measured or predicted) is reached.

The predicted dissolution extent by the 7 day PCT is far below the limiting value of 8.4 g/m², which is characteristic for the Environmental Assessment (EA) glass (Jantzen et al., 1992) used as a standard reference for American high level nuclear waste glasses. However, first-order models for glass dissolution possess considerable uncertainty ($R^2 = 0.78$), and thus laboratory testing is recommended until more accurate models are developed.⁸ Note that the glass with a lower waste loading (Example 2) exhibits a slightly higher dissolution extent by both MCC-1 and PCT despite the reduced alkali content achieved by reducing electrical conductivity to 30 S/m at 1150°C. This negative effect is caused by a lower level of alumina in the glass which was only partly compensated by adding alumina to the frit.

Figures 1 and 2 display glass dissolution by PCT as a function of the melt viscosity (at $T = 1150^\circ\text{C}$) and melting temperature (at $\eta = 10$ Pa.s), respectively. All property values were calculated using first-order models. The frit composition ($\text{SiO}_2 + \text{B}_2\text{O}_3 + \text{Li}_2\text{O}$) was allowed to vary in such a way that the parameters listed in the upper right corners of each figure were kept at constant values. The figures show that composition changes that produce either higher melt viscosity (at constant melting temperature) or higher melting temperature (at constant melting viscosity) reduce glass dissolution. But these more durable glasses may have an increased tendency to crystallize. Higher viscosity also makes melting more difficult. An optimum viscosity recommended by the Kernforschungszentrum in Germany is around 5 Pa.s (Lukscheiter, 1993).

Table 2 shows the composition of CCW from East Area tanks pretreated by the (projected) dissolution of solids and extraction of transuranic components. The waste is higher in SiO_2 and SO_3 , and lower in Na_2O , Al_2O_3 , and Cr_2O_3 than the same waste pretreated by organic destruction and sludge washing (Table 1). Two examples of waste glasses are shown. In Example 3, glass is made from the waste by mere addition of B_2O_3 and Li_2O . All the SiO_2 needed for glass network is from the waste. This allows a very high waste loading of 0.75. However, the resulting glass would contain 1.8 wt% SO_3 , which somewhat exceeds the upper limit of SO_3 solubility in silicate glasses. Whether this concentration of SO_3 will cause processing problems must be tested experimentally. The current maximum level of SO_3 accepted for HWVP waste glasses is 0.5 wt%. This would necessitate a large reduction in the waste loading (to 0.21, see Example 4) and thus is more expensive. To keep Li_2O content below 7 wt% in Example 4, some Na_2O (5 wt%) was added to the frit and somewhat lower electrical conductivity and higher viscosity of glass were allowed. To keep glass dissolution at a low level, 10 wt% of Al_2O_3 was added to the frit (although, according to Van Iseghem and Grambow (1988), the long term effect of Al_2O_3 on glass dissolution is uncertain). Because of the considerable potential increase of the waste loading, it would be worthwhile to investigate the effects of the elevated levels of SO_3 on melter performance or to reduce it by pretreatment or blending with wastes that have a low level of SO_3 .

Plutonium Finishing Plant Waste and Neutralized Cladding Removal Waste

Table 3 compares two wastes (PFPW and NCRW), of which PFPW was pretreated either by sludge washing (Examples 5 and 6)

⁸ Second-order models, which were also applied to our data base (Redgate et al., 1993) bring some, but not sufficient, increase in the R^2 value.

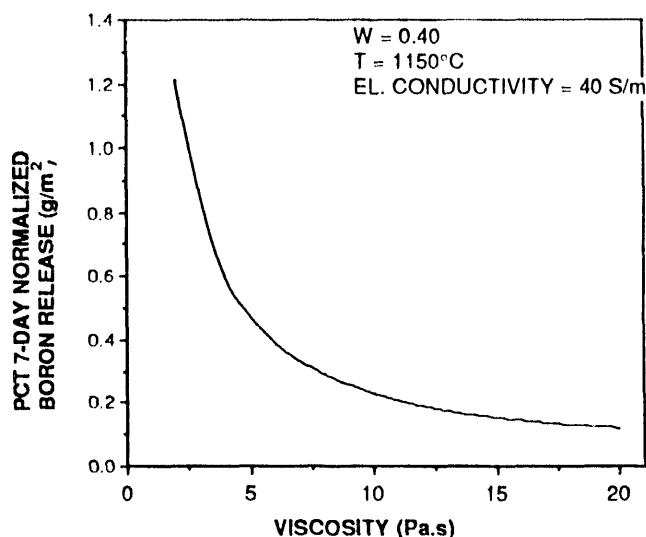


FIGURE 1. PREDICTED MASS OF GLASS (MEASURED AS NORMALIZED BORON RELEASE) DISSOLVED PER UNIT SURFACE AREA IN DEIONIZED WATER AT 90°C DURING 7 DAYS OF THE PRODUCT CONSISTENCY TEST AS A FUNCTION OF GLASS VISCOSITY AT 1150°C. DIFFERENT VALUES OF VISCOSITY WERE ACHIEVED BY CHANGING $\text{B}_2\text{O}_3/\text{SiO}_2$ AND $\text{Li}_2\text{O}/\text{SiO}_2$ RATIOS TO KEEP ELECTRICAL CONDUCTIVITY AT 40 S/m AT 1150°C. A CONSTANT CCW LOADING WAS MAINTAINED AT 0.4.

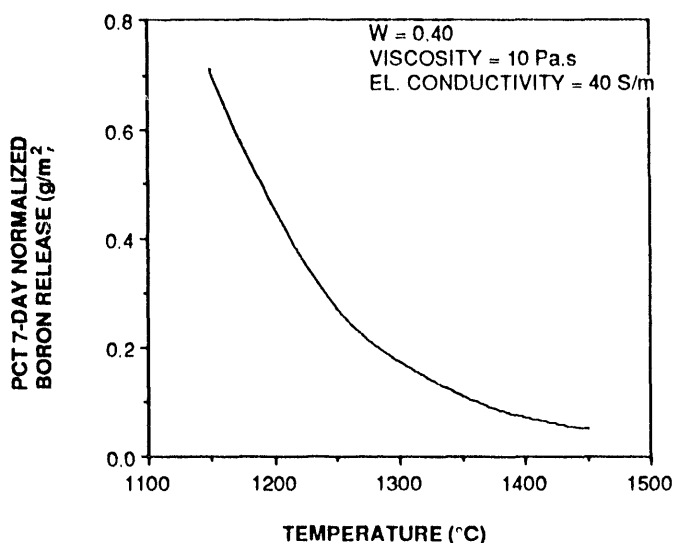


FIGURE 2. PREDICTED MASS OF GLASS DISSOLVED PER UNIT SURFACE AREA DURING 7 DAYS OF THE PRODUCT CONSISTENCY TEST AS A FUNCTION OF TEMPERATURE (T_{10}) AT WHICH GLASS VISCOSITY WAS 10 Pa.s. THE CHANGE IN T_{10} WAS ACHIEVED BY CHANGING $\text{B}_2\text{O}_3/\text{SiO}_2$ AND $\text{Li}_2\text{O}/\text{SiO}_2$ RATIOS TO KEEP ELECTRICAL CONDUCTIVITY AT 40 S/m AT T_{10} . A CONSTANT CCW LOADING WAS MAINTAINED AT 0.4.

TABLE 3. WASTE GLASS COMPOSITIONS (IN TERMS OF MASS FRACTIONS) FROM PFPW, PRETREATED BY DESTRUCTION OF ORGANICS AND SLUDGE WASHING; PFPW, PRETREATED BY SOLID DISSOLUTION AND TRANSURANIC EXTRACTION; AND NCRW, PRETREATED BY DESTRUCTION OF ORGANICS AND SLUDGE WASHING.

Component	Waste ¹	Example 5		Example 6		Waste ²	Example 7		Waste ³	Example 8	
		Frit	Glass	Frit	Glass		Frit	Glass		Frit	Glass
SiO ₂	0.0190	0.7322	0.5299	0.6002	0.5641	0.0471	0.5712	0.4415	0.0360	0.7304	0.5040
B ₂ O ₃	0.0025	0.1537	0.1078	0.1351	0.1262	0.0025	0.2742	0.2010	0.0059	0.1677	0.1112
Na ₂ O	0.1490	0.0300	0.0752	0.1000	0.1053	0.0431	0.1000	0.0959	0.2510		0.0939
Li ₂ O	0.0000	0.0841	0.0586	0.0647	0.0603	0.0000	0.0546	0.0399	0.0000	0.1019	0.0664
CaO	0.0300		0.0082		0.0018	0.0030		0.0008	0.0022		0.0008
MgO	0.0106		0.0030		0.0007	0.0028		0.0008	0.0009		0.0004
Fe ₂ O ₃	0.1270		0.0346		0.0077	0.0321		0.0080	0.0032		0.0014
Al ₂ O ₃	0.3070		0.0842	0.1000	0.1120	0.7540		0.1822	0.0641		0.0214
ZrO ₂	0.0020		0.0005		0.0001	0.0020		0.0005	0.5440		0.1686
Others	0.3529		0.0979		0.0218	0.1134		0.0296	0.0927		0.0318

TABLE 2. WASTE GLASS COMPOSITIONS (IN TERMS OF MASS FRACTIONS) FROM CCW EAST AREA TANKS PRETREATED BY SOLID DISSOLUTION AND TRANSURANIC EXTRACTION.

Component	Waste ¹	Example 3		Example 4	
		Frit	Glass	Frit	Glass
SiO ₂	0.6910	0.0002	0.5592	0.5376	0.5672
B ₂ O ₃	0.0000	0.6610	0.1038	0.2330	0.1781
Na ₂ O	0.0988		0.1132	0.0500	0.0699
Li ₂ O	0.0000	0.3388	0.0532	0.0794	0.0607
CaO	0.0028		0.0024		0.0007
MgO	0.0018		0.0016		0.0005
Fe ₂ O ₃	0.0113		0.0095		0.0026
Al ₂ O ₃	0.1140		0.0894	0.1000	0.1014
ZrO ₂	0.0008		0.0006		0.0002
Others	0.0795		0.0671		0.0187
Cr ₂ O ₃	0.0043		0.0032		0.0009
P ₂ O ₅	0.0201		0.0151		0.0042
SO ₃	0.0239		0.0180		0.0050
Viscosity at 1150°C (Pa.s)			8.4		7
Electrical conductivity at 1150°C (S/m)			40		29
Waste loading			0.75		0.21
W ₁ ²			0.17		0.12
W ₂ ³			0.17		0.12
MCC-1 28-day normalized B release (g/m ²)			8.28		10
PCT 7-day normalized B release (g/m ²)			0.69		.60

¹CCW, East Area, TRUEX Pretreatment

²Sum of the mass fractions of all oxides except SiO₂, B₂O₃, Na₂O, and Li₂O

³Sum of the mass fractions of Fe₂O₃, Al₂O₃, ZrO₂, and "others."

or by transuranic extraction with dissolution of solids (Example 7). The PFPW pretreated by sludge washing has a high level of Al₂O₃ and "others" (mainly Cr₂O₃, P₂O₅, and MnO₂); 3 wt% of Na₂O was added to the frit and somewhat lower electrical conductivity was allowed to keep Li₂O content below 7 wt%. Even a modest waste loading 0.27—limited by crystallinity constraints W₁ and W₂ (Example 5)—makes the concentration of Cr₂O₃ (2.3 wt%) and P₂O₅ (3.2 wt%) excessively high. Reduction of Cr₂O₃ to 0.5 wt% (which

than that achieved with PFPW washed sludge (Examples 5 and 6), but the uncertainty of the predicted value requires experimental verification in glasses of extreme composition, such as that of Example 7.

The NCRW pretreated by sludge washing (Example 8) is rich in ZrO₂ and Na₂O. Using crystallinity constraints W₁ and W₂, waste loading of 0.31 is probably the maximum achievable with the present technology based on Joule heated ceramic melters. Troublesome

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