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**Report for Westinghouse Hanford
Company: Makeup Procedures and
Characterization Data for Modified
DSSF and Modified Remaining
Inventory Simulated Tank Wastes**

R. O. Lokken

March 1996

**Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest National Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute**



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Pacific Northwest National Laboratory
Richland, Washington 99352

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SUMMARY

The majority of defense wastes generated from reprocessing spent reactor fuel at Hanford are stored in underground Double-Shell Tanks (DST) and in older Single-Shell Tanks (SST). The Tank Waste Remediation System (TWRS) Program has the responsibility of safely managing and immobilizing these tank wastes for disposal. A reference process flowsheet is being developed that includes waste retrieval, pretreatment, and vitrification. Melter technologies for vitrifying low-level tank wastes are being evaluated by Westinghouse Hanford Company. Chemical simulants are being used in the technology testing.

For the first phase of low-level waste (LLW) vitrification simulant development, two waste stream compositions were investigated. The first waste simulant was based on the analyses of six tanks of double-shell slurry feed (DSSF) waste and on the projected composition of the wastes exiting the pretreatment operations. A simulant normalized to 6 M sodium was based on the anticipated chemical concentrations after ion exchange and initial separations. The same simulant concentrated to 10 M sodium would represent a waste that had been concentrated by evaporation to reduce the overall volume. The second LLW simulant, referred to as the remaining inventory (RI), included wastes not included in the DSSF tanks and the projected LLW fraction of single-shell tank wastes.

Several compositions of LLW simulants were developed for use in the Phase 2 melter vendor tests. The simulants included DSSF, modified DSSF, RI and modified RI. The DSSF was the same composition that was developed and tested for the Phase 1 tests. One of the objectives of the Phase 2 melter vendor tests is to investigate the impacts of waste composition variability.

The waste composition variability issue was addressed by looking at tank inventory data files. Based on these data, it was found that, in general, most of the Cl and F is concentrated in DST sources and the majority of P and S is concentrated in SST sources. The current DSSF simulant is intended to be a surrogate for the DST wastes and the current RI is intended to be a surrogate for the much larger volume SST wastes. The modified DSSF simulant

was spiked with Cl and F at or near levels which would result in concentrations at their solubility limit in normal silicate waste glasses. Based on a 25 wt% waste oxide loading in the glass, the simulants were spiked at two, three, and four times their glass solubility limits. Similarly, the modified RI contained two, three, and four times the solubility of P_2O_5 and SO_3 .

Two mixtures of various trace metals were prepared for possible inclusion in the Phase 2 melter testing. Certain metals that have been identified in small quantities in the LLW composition data base have the potential for causing processing problems. The metals were selected from inventories of metals greater than 0.5 metric tons. The first mixture contained Bi, Cd, Ni, Pb, Zn, Sb, and Mo; the second mixture contained Cu, Zn, Pb, Mn, Ni, Sn, Sb, Mo, As, and Cr.

Laboratory prepared simulants were characterized for settled solids, density, viscosity, moisture content, and chemical analyses. Following laboratory development and characterization of the simulants, a procedure was recommended for use in preparing large quantities of LLW simulant for use in the Phase 2 melter vendor tests. Procedures were also developed for preparing the two metals mixtures.

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ACRONYMS

DSSF	Double-Shell Slurry Feed
DST	Double-Shell Tank
ICP	Inductively Coupled Plasma spectroscopy
LLW	Low-Level Waste
PNL	Pacific Northwest Laboratory
RI	Remaining Inventory
SST	Single-Shell Tank
TWRS	Tank-Waste Remediation System
WHC	Westinghouse Hanford Company
M-DSSF	Modified Double-Shell Slurry Feed
M-RI	Modified Remaining Inventory
TCLP	Toxicity Characteristic Leaching Procedure

INTRODUCTION

The majority of defense wastes generated from reprocessing spent reactor fuel at Hanford are stored in underground Double-Shell Tanks (DST) and in older Single-Shell Tanks (SST) in the form of liquids, slurries, sludges, and salt cakes. The Tank Waste Remediation System (TWRS) Program has the responsibility of safely managing and immobilizing these tanks wastes for disposal. A reference process flowsheet is being developed that includes waste retrieval, pretreatment, and vitrification.

PNL is assisting in the development of waste forms for vitrifying Hanford LLW and is supporting the Westinghouse Hanford Company melter technology selection. An integral part of the process is to identify, prepare, and characterize representative LLW simulants for use in these studies. During FY-94, procedures were developed for preparing waste simulants for use in laboratory vitrification studies and for Phase 1 melter vendor tests (Lokken and Martin 1994; Lokken 1995) as described in the PNL Project Work Plan and the WHC Vendor Test Statement of Work (Wilson 1994), respectively. Current activities have focused on developing simulants for use in the Phase 2 melter vendor tests. This report describes the basis for the simulants developed for Phase 2 testing, chemical and physical properties of the simulants and the recommended recipe for preparation.

PHASE 1 LOW-LEVEL WASTE SIMULANTS

For the first phase of LLW vitrification simulant development, two "reference" waste streams were investigated. The first waste stream simulant was based on the analyses of six tanks of Double-Shell Slurry Feed (DSSF) waste and on the projected composition of the wastes exiting the pretreatment operations (Shade 1994). A simulant normalized to 6 M sodium was based on the anticipated concentration after ion exchange and initial separations. A 10 M sodium simulant would represent the same waste that has been concentrated by evaporation to reduce the overall volume. The only difference between these two simulants is the water content; the species and their relative amounts are the same.

The second LLW stream simulant, referred to as the remaining inventory (RI), included wastes not included in the DSSF tanks and the projected LLW fraction of single-shell tank wastes (Shade 1994). Essentially, this waste stream encompasses the entire inventory of tank wastes except for that included in the DSSF waste stream discussed above.

Development and characterization of these simulants are described in Lokken and Martin (1994) and Lokken (1995). Table 1 lists the nominal compositions of the three simulants. The 10 M DSSF simulant was used in the Phase 1 melter vendor tests (Wilson 1994).

TABLE 1. Concentrations of Low-Level Tank Waste Simulants Tested
During Phase 1 Simulant Development, moles/L

<u>Component</u>	<u>DSSF 6 M Na</u>	<u>DSSF 10 M Na</u>	<u>Remaining Inventory</u>
Al	0.61	1.02	0.16
Ca	0.00063	0.00105	0.0004
Cr	0.0052	0.00867	0.0042
Fe	0.00046	0.000767	0.00024
K	0.30	0.50	0.0058
Mg	0.00062	0.00103	0.0000011
Mn	0.00025	0.000417	0.001
Mo	0.01	0.0167	0.01
Na	6.0	10.0	6.0
Sr	0.01	0.0167	0.01
Cs	0.01	0.0167	0.01
PO ₄	0.026	0.0433	0.11
IO ₄	0.01	0.0167	0.01
CO ₃	0.16	0.267	0.05
Cl	0.096	0.127	0.0092
F	0.15	0.25	0.13
SO ₄	0.026	0.0433	0.038
NO ₃	1.9	3.11	3.5
NO ₂	1.0	1.67	0.26
OH	2.3	3.8	1.5
TOC	0.81	1.35	0.11

PHASE 2 MELTER VENDOR SIMULANTS

Eight compositions of LLW simulants were tested for use in the Phase 2 melter vendor tests being conducted for Westinghouse Hanford Company. The simulants included DSSF, three variants of modified DSSF (M-DSSF), RI and three variants of modified RI (M-RI). The DSSF was the same composition that was developed and tested for the Phase 1 tests (Lokken and Martin 1994; Lokken 1995). One of the objectives of the Phase 2 melter vendor tests is to investigate some issues of the vitrification process that were not addressed in the Phase 1 tests. The issues can be grouped into two classes, related to processing issues and to product issues:^(a)

Processing Issues:

- Recycle of Melter Effluents,
- Semi-Volatile Metals,
- Solubility of P, S, F, Cl,
- Maximum Liquidus Temperature Operating Range, and
- Waste Compositional Variability Related to Flowsheet.

Product Issues:

- Waste Loading,
- Glass Product Durability.

Waste composition variability was addressed by analyzing tank inventory data files. Based on these data, it was found that, in general, most of the Cl and F is concentrated in DST sources and the majority of P and S is concentrated in SST sources. The current DSSF simulant is intended to be a surrogate for the DST wastes and the current RI is intended to be a surrogate for the much larger volume SST wastes. Consequently, it was decided to spike the DSSF simulant with Cl and F and to spike the RI with P and S as discussed below.

The modified DSSF simulant was spiked with Cl and F at levels which would result in concentrations at or near their solubility limit in normal silicate waste glasses^(a). Based on a 25 wt% waste oxide loading in the glass, the

^(a) Shade, J. W. January 5, 1995, Update on Phase 2 Simulants, via cc:Mail.

simulants were spiked at four, three, and two times their glass solubility limits. Similarly, the modified RI contained four, three and two times the solubility of P_2O_5 and SO_3 . The "modified" oxide composition of the simulants was normalized to 100% oxides after the addition of the Cl and F1 or P_2O_5 and SO_3 . The nominal oxide compositions of the "modified" LLW simulants are listed and compared with the original compositions in Tables 2 and 3.

TABLE 2. Oxide Composition of the Original and Modified DSSF Simulants.

Oxide	Wt% Oxide			
	DSSF	M-DSSF(2x)	M-DSSF(3x)	M-DSSF(4x)
Na ₂ O	72.67	69.12	65.49	61.86
K ₂ O	5.77	5.49	5.20	4.91
Al ₂ O ₃	12.70	12.08	11.45	10.81
CaO	0.01	0.01	0.01	0.01
Cr ₂ O ₃	0.16	0.15	0.14	0.14
Cs ₂ O	0.58	0.55	0.52	0.49
Fe ₂ O ₃	0.01	0.01	0.01	0.01
MgO	0.01	0.01	0.01	0.01
MnO ₂	0.01	0.01	0.01	0.01
MoO ₃	0.59	0.56	0.53	0.50
SrO	0.42	0.40	0.38	0.36
P ₂ O ₅	0.75	0.71	0.68	0.64
SO ₃	0.85	0.81	0.77	0.72
NaCl	2.29	3.30	4.95	6.59
NaF	2.57	6.21	9.31	12.42
NaI	0.61	0.58	0.55	0.52

TABLE 3. Oxide Composition of the Original and Modified Remaining Inventory (RI) Simulants

Oxide	Wt% Oxide			
	RI	M-RI(2x)	M-RI(3x)	M-RI(4x)
Na ₂ O	85.35	83.65	80.50	77.37
K ₂ O	0.13	0.13	0.12	0.12
Al ₂ O ₃	3.84	3.76	3.62	3.48
CaO	0.01	0.01	0.01	0.01
Cr ₂ O ₃	0.15	0.15	0.14	0.14
Cs ₂ O	0.66	0.65	0.62	0.60
Fe ₂ O ₃	0.01	0.01	0.01	0.01
MgO				
MnO ₂	0.04	0.04	0.04	0.04
MoO ₃	0.68	0.67	0.64	0.61
SrO	0.49	0.48	0.46	0.44
P ₂ O ₅	3.68	5.00	7.50	10.00
SO ₃	1.43	2.00	3.00	4.00
NaCl	0.25	0.24	0.24	0.23
NaF	2.57	2.52	2.42	2.32
NaI	0.71	0.70	0.67	0.64

Tables 4 and 5 list the ionic concentrations for the Phase 2 DSSF and RI simulants, respectively. The concentrations are based on 10 M Na concentrations for the DSSF and RI. The sodium concentrations for the modified simulants were allowed to "float" because of the additional Cl and F in the modified DSSF and additional PO₄ and SO₄ in the modified RI which were added as sodium salts.

Four batches of the Phase 2 simulants were prepared according to the recipes listed in Tables 6 and 7 using reagent grade chemicals and tap water. During the chemical additions, the solutions were stirred and heated to aid dissolution. After all the chemicals had been added and allowed to dissolve, the volumes were brought to slightly less than one liter through evaporation. The hot solutions were then transferred into graduated cylinders, the volumes

adjusted to one liter, and the densities were determined. After cooling to room temperature, the volumes were readjusted to one liter and the densities were remeasured. The one-liter samples were then transferred to polypropylene jars and maintained at room temperature.

TABLE 4. Ionic Concentration of Phase 2 DSSF Simulants

Species	Moles/Liter			
	DSSF	M-DSSF(2x)	M-DSSF(3x)	M-DSSF(4x)
Na	1.00E+01	1.04E+01	1.09E+01	1.11E+01
K	5.00E-01	4.78E-01	4.56E-01	4.33E-01
Al	1.02E+00	9.72E-01	9.27E-01	8.81E-01
Ca	7.28E-04	6.96E-04	6.63E-04	6.30E-04
Cr	8.60E-03	8.21E-03	7.83E-03	7.44E-03
Cs	8.40E-03	8.03E-03	7.66E-03	7.28E-03
Fe	5.11E-04	4.89E-04	4.66E-04	4.43E-04
Mo	1.67E-02	1.60E-02	1.52E-02	1.45E-02
Sr	1.66E-02	1.58E-02	1.51E-02	1.43E-02
PO ₄	4.32E-02	4.12E-02	3.93E-02	3.74E-02
SO ₄	4.34E-02	4.14E-02	3.95E-02	3.75E-02
Cl	1.60E-01	2.31E-01	3.49E-01	4.69E-01
F	2.50E-01	6.07E-01	9.16E-01	1.23E+00
IO ₄	1.66E-02	1.59E-02	1.51E-02	1.44E-02
NO ₂	1.70E+00	1.70E+00	1.70E+00	1.70E+00
NO ₃	3.20E+00	2.96E+00	3.20E+00	2.96E+00
CO ₂	2.70E-01	2.70E-01	2.70E-01	2.70E-01
OH	3.83E+00	3.83E+00	3.83E+00	3.83E+00
TOC	1.40E+00	1.40E+00	1.40E+00	1.40E+00

TABLE 5. Ionic Concentration of Phase 2 Remaining Inventory (RI) Simulants

Species	Moles/Liter			
	RI	M-RI(2x)	M-RI(3x)	M-RI(4x)
Na	1.00E+01	1.03E+01	1.08E+01	1.14E+01
K	9.77E-03	9.77E-03	9.77E-03	9.96E-03
Al	2.67E-01	2.67E-01	2.67E-01	2.67E-01
Ca	6.31E-04	6.31E-04	6.31E-04	6.27E-04
Cr	6.99E-03	6.99E-03	6.99E-03	6.99E-03
Cs	8.29E-03	8.29E-03	8.29E-03	8.29E-03
Fe	4.44E-04	4.44E-04	4.44E-04	4.41E-04
Mo	1.67E-02	1.67E-02	1.67E-02	1.66E-02
Sr	1.67E-02	1.67E-02	1.67E-02	1.66E-02
PO ₄	1.84E-01	2.55E-01	3.97E-01	5.51E-01
SO ₄	6.33E-02	9.03E-02	1.41E-01	1.95E-01
Cl	1.51E-02	1.51E-02	1.51E-02	1.54E-02
F	2.17E-01	2.17E-01	2.17E-01	2.16E-01
IO ₄	1.68E-02	1.68E-02	1.68E-02	1.67E-02
NO ₂	4.30E-01	4.30E-01	4.30E-01	4.30E-01
NO ₃	6.00E+00	6.00E+00	6.00E+00	6.00E+00
CO ₂	8.30E-02	8.30E-02	8.30E-02	8.30E-02
OH	2.37E+00	2.37E+00	2.37E+00	2.37E+00
TOC	1.80E-01	1.80E-01	1.80E-01	1.80E-01

TABLE 6. Batch Sheet for Phase 2 DSSF Simulants

Compound	grams/Liter			
	DSSF	M-DSSF(2x)	M-DSSF(3x)	M-DSSF(4x)
NaNO ₂	117.30	117.30	117.30	117.30
KOH (85%)	33.03	31.55	30.08	28.59
SrCl ₂ 6H ₂ O	4.41	4.22	4.02	3.82
NaH ₂ PO ₄ H ₂ O	5.96	5.69	5.42	5.16
NaIO ₄	3.29	3.40	3.24	2.85
Na ₂ CO ₃	28.62	28.62	28.62	28.62
NaCl	7.42	11.68	18.66	25.72
NaF	10.50	25.47	38.45	51.59
Na ₂ SO ₄	6.16	5.88	5.61	5.33
NaOH (50 wt%)	520.08	510.73	501.21	491.57
Al(NO ₃) ₃ 9H ₂ O	381.65	364.62	347.63	330.42
Na ₂ CrO ₄ 4H ₂ O	1.28	0.96	0.92	1.11
Na ₂ MoO ₄ H ₂ O	4.05	3.87	3.69	3.51
CsNO ₃	1.64	1.57	1.49	1.42
NaNO ₃	11.35	22.98	34.59	25.91
Na ₄ EDTA	58.27	58.27	58.27	58.27
Fe(NO ₃) ₃ 9H ₂ O ^(a)	0.21	-	-	0.18
Mg(NO ₃) ₂ 6H ₂ O ^(a)	0.26	-	-	0.22
Mn(NO ₃) ₂ (50%) ^(a)	0.16	-	-	0.14

(a) Added only to batch 3.

TABLE 7. Batch Sheet for Phase 2 RI Simulants

Compound	grams/Liter			
	RI	M-RI(2x)	M-RI(3x)	M-RI(4x)
NaNO ₂	29.67	29.67	29.67	29.67
KOH (85%)	0.65	0.65	0.65	0.66
SrCl ₂ 6H ₂ O	4.47	4.47	4.47	4.43
NaH ₂ PO ₄ H ₂ O	25.34	35.13	54.76	75.99
NaIO ₄ ^(a)	3.32	3.59	3.59	3.30
Na ₂ CO ₃	8.80	8.80	8.80	8.80
NaCl	0.00	0.00	0.00	0.00
NaF	9.10	9.10	9.10	9.07
Na ₂ SO ₄	8.98	12.82	19.99	27.74
NaOH (50 wt%)	284.98	296.22	318.97	343.58
Al(NO ₃) ₃ 9H ₂ O	100.07	100.07	100.07	100.08
Na ₂ CrO ₄ 4H ₂ O	1.04	0.82	0.82	1.04
Na ₂ MoO ₄ H ₂ O	4.05	4.05	4.05	4.01
CsNO ₃	1.62	1.62	1.62	1.62
NaNO ₃	440.72	440.72	440.72	440.72
Na ₄ EDTA	7.49	7.49	7.49	7.49
Fe(NO ₃) ₃ 9H ₂ O ^(a)	0.18	-	-	0.18
Mg(NO ₃) ₂ 6H ₂ O ^(a)	0.00	-	-	0.00
Mn(NO ₃) ₂ (50%) ^(a)	0.58	-	-	0.58

(a) Added only to batch 3.

The first batches of the simulants were prepared as dilute (~3 M Na) solutions and then evaporated to reach a final volume of one liter, resulting in the nominal concentrations listed in Tables 4 and 5. The dilute solutions would represent the concentrations of wastes exiting the ion exchange process, and the final concentration would be representative of wastes after going through the evaporator.

Modified DSSF and modified RI were also prepared in concentrated form (i.e., at the concentrations listed in Tables 4 and 5). This procedure would most likely resemble that used by the vendor chosen to produce the simulants for the melter tests.

The third batch of M-DSSF(4x) and M-RI(4x) were prepared as in the first batch. However, during volume reduction, 40-ml samples were taken at various levels of concentration.

REDUCIBLE METALS AND SEMI-VOLATILES SIMULANT ADDITIVES

Two compositions of various trace metals were prepared for possible inclusion in the Phase 2 melter testing. Certain metals that have been identified in small quantities in the LLW composition data base have the potential for causing processing problems. The metals were selected from inventories of metals greater than 0.5 metric tons.^(a)

Because of the uncertainty associated with the concentrations, two metals mixtures containing equal weights of the metals listed in Tables 8 and 9 were selected. The batch sheet used for preparing metals mixture number 1 is listed in Table 8. An alternate, low-hazardous metals mix was also prepared. This mix contained equal concentrations of Cu, Zn, Pb, Sn, Cr, Mn, Ni, Mo, Sb, and As (on a metals basis). These metals should demonstrate similar chemical behavior to those in the first mix, but they are usually contained in glass sufficiently well to pass TCLP criteria, thus minimizing or eliminating the need for disposal as hazardous waste. The batch sheet for the metals mixture number 2 is shown in Table 9.

The first two batches of the metals mixtures were prepared by adding the chemicals listed in Table 8 and 9 to deionized water while maintaining constant agitation. The final concentration of the solutions/slurries was 100 grams of total compound weight in a volume of one liter. Both mixtures

(a) Shade, J. W. January 5, 1995, Update on Phase 2 Simulants, via cc:Mail.

contained undissolved or precipitated solids that were easily maintained in suspension, and after settling, they were easily resuspended.

The next step in preparing the mixtures was to make three separate solutions for each mixture. The first solution contained the nitrate salts. The second solution contained the chloride salts, and the third solution contained the remaining salts.

For the first solution for metals mixture number 1, the nitrate salts of Bi, Cd, Ni, Pb, and Zn were combined with approximately an equal weight of a 1 M nitric acid solution. The nitric acid solution was required to completely dissolve the $\text{Bi}(\text{NO}_3)_3$. The second solution was prepared by dissolving SbCl_3 in a concentrated HCl solution of equal weight. The SbCl_3 cannot be directly dissolved in water because the compound converts to the insoluble antimony oxychloride when contacting water. The third solution was prepared by dissolving $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ in twice its weight of water. There were no undissolved or precipitated solids in these three solutions.

The first solution for metals mixture number 2 was prepared by dissolving the nitrate salts of Cu, Zn, Pb, Mn, and Ni in a volume of water equal in weight to the total weight of the salts. (Note: Mn was added as a 50 wt% solution). The second solution contained SbCl_3 and SnCl_2 dissolved in an equal weight of concentrated HCl. The third solution was prepared by dissolving $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$, and $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$ in twice the total compound weight of water. There were no undissolved or precipitated solids in these three solutions.

TABLE 8. Batch Sheet for Metals Mixture No. 1

<u>Metal</u>	<u>Compound</u>	<u>wt% Comp.</u>
Bi	Bi(NO ₃) ₃ 5H ₂ O	11.29%
Cd	Cd(NO ₃) ₂ 4H ₂ O	13.35%
Ni	Ni(NO ₃) ₂ 6H ₂ O	24.09%
Pb	Pb(NO ₃) ₂	7.77%
Zn	Zn(NO ₃) ₂ 6H ₂ O	22.13%
Sb	SbCl ₃	9.11%
Mo	Na ₂ MoO ₄ 2H ₂ O	12.26%

TABLE 9. Batch Sheet for Metals Mixture No. 2

<u>Metal</u>	<u>Compound</u>	<u>wt% Comp.</u>
Cu	Cu(NO ₃) ₂ 3H ₂ O	11.57%
Zn	Zn(NO ₃) ₂ 6H ₂ O	13.84%
Pb	Pb(NO ₃) ₂	4.86%
Mn	Mn(NO ₃) ₂ ^(a)	12.96%
Ni	Ni(NO ₃) ₂ 6H ₂ O	9.27%
Sn	SnCl ₂	7.45%
Sb	SbCl ₃	4.74%
Mo	Na ₂ MoO ₄ 2H ₂ O	7.23%
As	Na ₂ HAsO ₄ 7H ₂ O	9.82%
Cr	Na ₂ CrO ₄ 4H ₂ O	18.25%

(a) Added as a 50 wt% solution

PHYSICAL PROPERTIESSETTLED SOLIDS

The quantity of settled, undissolved or precipitated solids in the Phase 2 simulants was estimated by allowing the freshly prepared solutions/slurries to remain undisturbed in a closed container until a clear supernate was observed and then estimating the total volume of solids by comparing the depth of solids to the total depth of sample. Table 10 lists the estimated amount of settled solids in samples at room temperature and for the same samples after heating to ~75°C. The RI and M-RI samples had the most solids, ranging between 19 and 23 vol% for the RI sample and between 14 and 58 vol% for the M-RI samples. After cooling from 75°C to room temperature the M-RI samples appeared "gelatinous" as a network of crystals formed throughout the sample. The crystals are likely sodium phosphate because of the high levels of sodium and phosphate present in the M-RI simulants and because of the high temperature dependence on the solubility of sodium phosphates.

TABLE 10. Estimated Volume Percent Settled Solids

<u>Simulant</u>	<u>Solids, vol%</u>		<u>Batch^(b)</u>
	<u>RT^(a)</u>	<u>75°C</u>	
DSSF	7	2	1
M-DSSF(2x)	7	6	3
M-DSSF(3x)	9	6	3
M-DSSF(4x)	8	6	1
M-DSSF(4x)	16	14	2
RI	23	19	1
M-RI(2x)	28	14	3
M-RI(3x)	40	22	3
M-RI(4x)	58	40	1
M-RI(4x)	46	37	2

(a) RT = room temperature

(b) Batch 1 was prepared dilute and then concentrated;
 Batches 2 and 3 were prepared concentrated.

An important property of slurries is the ability to resuspend the solids once they have settled in order to obtain a uniform feed to the vitrification facility. A qualitative evaluation of the "resuspendability" of the solids in the simulants was done by allowing the solids to settle in a 1-L polypropylene bottle overnight and then slowly turning the bottles end-over-end several times, each time inspecting the bottom for the presence of remaining solids. The solids in all the samples, except the room temperature M-DSSF(4x) sample from Batch 2, were resuspended by only a few rotations of the sample. The M-DSSF(4x) sample had to be shaken vigorously to resuspend the settled solids. Based on these observations, it appears that the solids in all the simulants could be resuspended by moderate to vigorous agitation.

DENSITY

Density of the solutions/slurries, i.e., of the total sample, was measured by weighing a known volume within a graduated cylinder. The density of the

supernates was determined by weighing a 100-ml sample in a volumetric flask at room temperature. Table 11 summarizes the density results. The densities of the slurries at room temperature were generally about 2 to 3% higher than at the elevated temperatures.

TABLE 11. Density of Phase 2 Simulants

Simulant	Density, g/cm ³			Batch(c)
	Supernate	Total(a)	Total(°C)(b)	
DSSF	1.445	1.46	1.42 (71)	1
M-DSSF(4x)	1.447	1.47	1.44 (64)	1
M-DSSF(4x)	1.443	1.48	1.44 (70)	2
M-DSSF(3x)	ND(d)	1.47	1.45 (85)	4
M-DSSF(2x)	ND	1.47	1.43 (84)	4
RI	1.393	1.45	1.44 (75)	1
M-RI(2x)	ND	1.48	1.43 (85)	4
M-RI(3x)	ND	1.50	1.46 (82)	4
M-RI(4x)	1.397	1.52	1.49 (65)	1
M-RI(4x)	1.403	1.52	1.49 (75)	2

(a) Measured at room temperature

(b) Measured at the temperature in parentheses

(c) Batch 1 was prepared dilute and then concentrated;
Batches 2 and 3 were prepared concentrated.

(d) ND = Not Determined

VISCOSITY

Viscosity of the simulants was measured using a Fann viscometer. The measured values are shown in Table 12. The nature of the solids in the RI simulants did not allow for viscosity measurements on the total samples because the size of some of the solid particles was larger than the gap size in the viscometer and because the solids settled out very quickly in the viscometer cup. However, based on a qualitative evaluation of the simulants, the apparent viscosities of the slurries should be sufficiently low to allow adequate handling and transport in the melter vendor processes.

TABLE 12. Viscosity of Phase 2 Simulants

Simulant	Viscosity, cP ^(a)		Batch
	Supernate	Slurry	
DSSF	15	16	1
M-DSSF(2x)	16	18	4
M-DSSF(3x)	16	19	4
M-DSSF(4x)	16	17	1
M-DSSF(4x)	16	18	2
RI	8	ND ^(b)	1
M-RI(2x)	7	ND	3
M-RI(3x)	8	ND	3
M-RI(4x)	8	ND	1
M-RI(4x)	8	ND	2

(a) Values at a shear rate of 511 s^{-1}

(b) ND = Not Determined because of the presence of large crystalline solids

CHEMICAL PROPERTIES

Samples of the simulants were dried at 105°C - 120°C in a vacuum oven for several days to determine evaporable water or moisture content. Table 13 compares results calculated from the nominal composition and from the measured density, and the experimental results measured after allowing the samples to cool in a desiccator. The experimental and calculated values are in general agreement, with an average moisture content of about 50% by weight. The larger experimental values for the RI samples are likely caused by the difficulty in obtaining truly representative specimens from the simulant samples. The large quantity of solids, and their rapid settling rate resulted in specimens that contained less undissolved solids, resulting in a higher experimentally determined moisture contents.

TABLE 13. Moisture Content of Phase 2 Simulants

Simulant	Moisture Content, wt%		Batch
	Calculated	Experimental	
DSSF	53.6	50.0	1
M-DSSF(2x)	52.7	47.7	4
M-DSSF(3x)	51.6	48.7	4
M-DSSF(4x)	50.4	48.9	1
M-DSSF(4x)	50.7	48.9	2
RI	50.8	52.5	1
M-RI(2x)	50.8	52.0	4
M-RI(3x)	49.4	51.6	4
M-RI(4x)	47.9	52.5	1
M-RI(4x)	47.9	52.7	2

Chemical analyses were conducted on selected simulants to compare the analyzed concentrations with the target values. Inductively coupled argon plasma emission spectrometry (ICP) was conducted with a Thermo Jarrell-Ash Model 61E spectrometer according to Method APSL-14. The solutions were acidified using 2 vol% nitric acid. Anion concentrations were measured using a Dionex Series 4000i ion chromatograph according to procedure PNL-ALO-212 Rev. 1. Carbon analyses was conducted using a Xertex-Dohrmann Model DC-80 carbon analyzer according to procedure PNL-ALO-382.1 Rev. 0. The free hydroxide content was determined by titration. A summary of the analytical results are shown in Tables 14 through 19.

Table 14 compares the nominal composition of DSSF and modified DSSF and the analyzed composition of the supernates from the samples. It should be noted that the analyzed Al values may be higher than actual because the samples were stored in plastic vials with aluminum-lined caps. The concentration of several of the analyzed species are considerably below the nominal composition, indicating that these species either precipitated from solution, or were not completely dissolved. These included Cr, PO₄, SO₄, and Sr.

TABLE 14. Nominal and Analyzed Chemical Composition of DSSF and M-DSSF, mg/L (Analyzed Composition is of the Supernate only).

Ion	DSSF		M-DSSF(4x)		
	Nominal	Batch 1	Nominal	Batch 1	Batch 2
Al	27,450	27,015	23,765	22,320	24,910
Ca	29	11	25	<5	<5
Cl	5,673	5,056	16,616	15,910	15,869
CO ₃	16,200	25,280	16,200	15,910	18,750
Cr	447	275	387	214	244
F	4,749	ND ^(c)	23,344	ND	ND
K	19,562	19,214	16,936	15,716	16,814
Mo	1,606	1,552	1,390	1,309	1,411
Na	229,900	213,970	254,760	209,330	226,280
NO ₂	78,200	76,560	78,200	73,770	79,340
NO ₃	198,400	187,800	183,497	188,050	187,500
OH	65,067	46,660	65,110	51,640	46,600
PO ₄	4,099	(580)	3,549	(870)	(580)
SO ₄	4,166	1,730	3,606	1,740	1,730
Sr	1,450	564	1,256	459	566
TOC	16,800	15,601	16,800	16,060	15,440

(a) Values in parentheses are close to the analytical detection limit

(b) "Less-than" values are the analytical detection limit

(c) ND = Not Determined

Table 15 compares the nominal composition of RI and modified RI and the analyzed composition of the supernates from the samples. These samples had considerable quantities of undissolved solids as discussed above. Based on the PO₄ results, it appears that most of the PO₄ precipitated, probably as a sodium phosphate. Sulfate, Cr, and Sr are also lower in concentration than the nominal, overall concentrations. The analyzed carbonate concentrations are considerably higher and the hydroxide concentrations lower than desired, due possibly to conversion of OH to CO₃ by atmospheric CO₂ during agitation and volume reduction.

Tables 16 and 17 show the analyzed concentrations of species of modified DSSF and modified RI at different stages of concentration. The modified DSSF samples showed a fairly uniform increase in concentration of all species in the supernates as the samples were concentrated. The modified DSSF sample at a nominal 10 M Na concentration (last column of Table 16) did not indicate the degree of precipitation as suggested by the compositions of the previous modified DSSF supernates (see Table 14), suggesting that the precipitation may be kinetically controlled, i.e., occurring over time.

Tables 18 and 19 compare the nominal total metal composition of the two metals mixtures to the analyzed composition of the mixture supernates. As discussed above, considerable precipitation occurred in these mixtures, and the data in the tables suggest which species are present in the supernate and which precipitated. Table 18 shows that, within experimental and/or analytical errors, the Cd, Ni, and Zn remained in solution in the metals mixture number 1, while most of the other metals precipitated. Similarly, in the metals mixture number 2, the Cu, Zn, Mn, and Ni appeared to remain in solution, while Sn and Mo were almost entirely precipitated. About half the Pb and two thirds of the Cr remained in solution. Although Sb was not analyzed, based on observation during preparation of the mixtures, it is believed that nearly all of the Sb would be present in the solid phase.

TABLE 15. Nominal and Analyzed Chemical Composition of RI and M-RI, mg/L
(Analyzed Composition is of the Supernate only).

Ion	RI		M-RI(4x)		
	Nominal	Batch 1	Nominal	Batch 1	Batch 2
Al	7,197	8,504	7,198	10,012	8,258
Ca	25	<5	25	<5	<5
Cl	537	696	545	783	800
CO ₃	4,980	15,320	4,980	6,990	19,650
Cr	363	234	364	291	281
Cs	1,102	ND ^(c)	1,102	ND	ND
F	4,118	ND	4,103	ND	ND
K	382	<150	389	<150	<150
Mo	1,605	1,730	1,589	2,040	1,985
Na	229,900	198,430	261,218	209,650	204,750
NO ₂	19,780	20,890	19,780	23,760	25,260
NO ₃	372,000	334,200	372,000	279,500	294,700
OH	40,315	33,140	40,290	42,760	33,400
PO ₄	17,439	(560)	52,300	2,930	3,230
SO ₄	6,077	1,950	18,760	15,370	11,930
Sr	1,467	553	1,454	137	237
TOC	2,160	2,500	2,160	2,660	2,250

- (a) Values in parentheses are close to the analytical detection limit
 (b) "Less-than" values are the analytical detection limit
 (c) ND = Not Determined

TABLE 16. Analyzed Chemical Composition of M-DSSF(4x) Supernates at Various Stages of Concentration, mg/L

Ion	Approximate Concentration Factor ^(a)			
	0.27	0.42	0.67	1.00
Al	5,945	10,006	16,603	24,344
Ca	19	27	46	37
Cl	4,064	6,271	10,328	14,510
CO ₃	5,081	7,839	15,492	25,393
Cr	67	102	153	235
Cs	ND ^(b)	ND	ND	ND
F	ND	ND	ND	ND
Fe	5	1	7	17
IO ₄	ND	ND	ND	ND
K	4,656	7,073	10,816	16,133
Mg	<12 ^(c)	<12	<12	<12
Mn	<1	<1	<1	<1
Mo	380	579	873	1,320
Na	67,400	100,700	133,000	224,000
NO ₂	20,320	31,360	51,640	76,900
NO ₃	50,810	77,180	129,100	192,980
OH	13,630	20,092	30,730	46,870
PO ₄	960	1,568	1,549	2,031
SO ₄	1,005	1,447	2,324	3,337
Sr	154	247	378	553
TOC	4,290	4,462	10,840	15,530

(a) Concentration Factor is the concentration relative to a 10 M Na solution

(b) ND = Not Determined

(c) "Less-Than" values are detection limits

TABLE 17. Analyzed Chemical Composition of M-RI(4x) Supernates at Various Stages of Concentration, mg/L

Ion	Approximate Concentration Factor ^(a)			
	0.27	0.42	0.67	1.00
Al	3,810	4,240	6,120	9,230
Ca	<2 ^(b)	<2	<2	<2
Cl	211	346	504	843
CO ₃	2,220	2,990	5,970	2,180
Cr	60	93	141	260
Cs	ND ^(c)	ND	ND	ND
F	ND	ND	ND	ND
Fe	1	3	8	12
IO ₄	ND	ND	ND	ND
K	<60	<60	<60	123
Mg	<12	<12	<12	<12
Mn	<1	<1	<1	<1
Mo	455	672	1,010	1,840
Na	70,900	100,300	132,700	164,600
NO ₂	5,430	8,600	13,800	26,150
NO ₃	98,610	167,200	252,100	290,600
OH	9,040	14,210	24,820	41,990
PO ₄	8,860	7,160	14,600	11,040
SO ₄	4,880	7,760	9,020	7,270
Sr	21	37	37	69
TOC	665	955	1,330	581

(a) Concentration Factor is the concentration relative to a 10 M Na solution

(c) ND = Not Determined

(b) "Less-Than" values are detection limits

TABLE 18. Nominal and Analyzed Composition of Metals Mixture 1 Supernate

Metal	Composition, mg/L	
	Nominal	Analyzed
Bi	4817	59.7
Cd	4817	4518
Mo	4817	7.1
Ni	4817	4669
Pb	4817	1510
Sb	4817	ND ^(a)
Zn	4817	4488

(a) ND = Not Determined

TABLE 19. Nominal and Analyzed Composition of Metals Mixture 2 Supernate

Metal	Composition, mg/L	
	Nominal	Analyzed
Cu	3109	3204
Zn	3109	2847
Pb	3109	1502
Sn	3109	5.5
Cr	3109	2028
Mn	3109	3290
Ni	3109	2914
Mo	3109	272
Sb	3109	ND ^(a)
As	3109	ND

(a) ND = Not Determined

RECOMMENDED PROCEDURE FOR PHASE 2 LLW SIMULANT PREPARATION

The following procedure is recommended for preparing DSSF, M-DSSF(4x), RI, and M-RI(4x) LLW simulants for the Phase 2 melter vendor tests. Start with water at approximately 30 vol% of the final solution volume required. Add the appropriate amounts of the following compounds (solutions) in the order listed to the water while maintaining constant, moderate agitation (note: the solution may be heated to aid dissolution):

Compound	grams/Liter			
	DSSF	RI	M-DSSF(4x)	M-RI(4x)
NaNO ₂	117.30	29.67	117.30	29.67
KOH (85%)	33.03	0.65	28.59	0.66
SrCl ₂ ·6H ₂ O	4.41	4.47	3.82	4.43
NaH ₂ PO ₄ ·H ₂ O	5.96	25.34	5.16	75.99
NaIO ₄	3.29	3.32	2.85	3.30
NaCl	7.42	0.00	25.72	0.00
NaF	10.50	9.10	51.59	9.07
Na ₂ SO ₄	6.16	8.98	5.33	27.74
NaOH (50 wt% sol'n)	520.08	284.98	491.57	343.58
Na ₂ CO ₃	28.62	8.80	28.62	8.80
Al(NO ₃) ₃ ·9H ₂ O	381.65	100.07	330.42	100.08
Na ₂ CrO ₄ ·4H ₂ O	1.28	1.04	1.11	1.04
Na ₂ MoO ₄ ·H ₂ O	4.05	4.05	3.51	4.01
CsNO ₃	1.64	1.62	1.42	1.62
NaNO ₃	11.35	440.72	25.91	440.72
Na ₄ EDTA	58.27	7.49	58.27	7.49

Add a minimum amount of water to completely dissolve the following compounds prior to adding to the main solution:

Fe(NO ₃) ₃ ·9H ₂ O	0.21	0.18	0.18	0.18
Mg(NO ₃) ₂ ·6H ₂ O	0.26	0.00	0.22	0.00
Mn(NO ₃) ₂ (50% sol'n)	0.16	0.58	0.14	0.58

When all the compounds have been added and no further dissolution appears to be occurring, allow the solution to cool to room temperature (~20 to 25°C) and adjust the volume with water to the required level.

When using solutions as a substitute for the dry chemicals, the initial volume of water may need to be decreased to ensure that the final volume does not exceed the desired volume. If aluminum nitrate solution is used, it is recommended that this solution be added to the 50% NaOH solution first, and then the remaining ingredients added to this solution, again in the order listed above.

Based on the heterogeneous nature of the resulting simulants (i.e., the large amount of solids), obtaining representative samples for confirmatory analyses from large batches is very difficult. It is therefore recommended that the individual chemicals be analyzed to determine purity and the analyzed values be used for adjustment of amounts. The quantity added to the batch should then be used for determining total composition and acceptability of the simulants for use in the Phase 2 melter tests.

RECOMMENDED PROCEDURE FOR PHASE 2 METALS MIXTURES PREPARATION

The two metals mixtures can either be prepared as individual mixtures or as three separate solutions for each composition. Because of the chemicals used for the individual metals, precipitation occurs when all the chemicals are mixed together. If a single solution/slurry is satisfactory, or desired, the metals mixtures should be prepared by mixing the chemicals listed below in sufficient water to allow the solids that precipitate to be easily resuspended. (Note: in the development studies, the solutions/slurries were mixed at a total compound weight of 100 grams in a total volume of one liter.)

Metals Mixture Number 1 (Single Solution/Slurry):

<u>Metal</u>	<u>Compound</u>	<u>wt% Comp.</u>
Bi	$\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	11.29%
Cd	$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	13.35%
Ni	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	24.09%
Pb	$\text{Pb}(\text{NO}_3)_2$	7.77%
Zn	$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	22.13%
Sb	SbCl_3	9.11%
Mo	$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$	12.26%

Metals Mixture Number 2 (Single Solution/Slurry):

<u>Metal</u>	<u>Compound</u>	<u>wt% Comp.</u>
Cu	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	11.57%
Zn	$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	13.84%
Pb	$\text{Pb}(\text{NO}_3)_2$	4.86%
Mn	$\text{Mn}(\text{NO}_3)_2$	12.96%
Ni	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	9.27%
Sn	SnCl_2	7.45%
Sb	SbCl_3	4.74%
Mo	$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$	7.23%
As	$\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$	9.82%
Cr	$\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$	18.25%

Three individual solutions for each metals mixture can also be prepared. This approach results in concentrated solutions, devoid of undissolved or precipitated solids.

For the first solution for metals mixture number 1, the nitrate salts of Bi, Cd, Ni, Pb, and Zn are combined with approximately an equal weight of a 1 M nitric acid solution. The nitric acid solution is required to completely dissolve the $\text{Bi}(\text{NO}_3)_3$; the other nitrate salts can be dissolved in pure water. The second solution is prepared by dissolving SbCl_3 in a concentrated HCl

solution of equal weight. The SbCl_3 cannot be directly dissolved in water because the compound is converted to the insoluble antimony oxychloride. The third solution is prepared by dissolving $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ in twice its weight of water.

The first solution for metals mixture number 2 is prepared by dissolving the nitrate salts of Cu, Zn, Pb, Mn, and Ni in a volume of water equal in weight to the total weight of the salts. (Note: Mn was added as a 50 wt% solution). The second solution contains SbCl_3 and SnCl_2 dissolved in an equal weight of concentrated HCl. The third solution is prepared by dissolving $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$, and $\text{Na}_2\text{CrO}_4 \cdot 4\text{H}_2\text{O}$ in twice the total compound weight of water.

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