

Tank 42 Sludge-Only Process Development for the Defense Waste Processing Facility (DWPF)

by

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TANK 42 SLUDGE-ONLY PROCESS DEVELOPMENT FOR THE
DEFENSE WASTE PROCESSING FACILITY (DWPF) (U)

INFORMATION ONLY

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SAVANNAH RIVER SITE

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

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***TANK 42 SLUDGE-ONLY PROCESS DEVELOPMENT
FOR THE DEFENSE WASTE PROCESSING
FACILITY (DWPF) (U)***

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EXECUTIVE SUMMARY

DWPF requested the development of a sludge-only process for Tank 42 sludge (HLW-DWPF/TTR-970134) since at the current processing rate, the Tank 51 sludge has been projected to be depleted as early as August 1998. Testing was completed using a non-radioactive Tank 42 sludge simulant. The testing was completed under a range of operating conditions, including worst case conditions, to develop the processing conditions for radioactive Tank 42 sludge. The existing Tank 51 sludge-only process is adequate with the exception that 10% additional acid is recommended during SRAT processing to ensure adequate destruction of nitrite during the SRAT cycle.

The following points summarize the major conclusions of the testing:

- Hydrogen and nitrous oxide (N₂O) generation rates were well below DWPF operating limits and design bases, even under the worst case operating conditions of this testing.
- The SME product composition passed the Product Consistency Test (PCT) and met all requirements for SME acceptability of the Process Composition and Control System except for the processing constraints of high viscosity and liquidus.
- No processing problems such as foaming were noted in these small-scale experiments.
- The process was demonstrated under both the more oxidizing conditions of the current DWPF sludge-only process and the more reducing conditions recommended by SRTC to minimize foaming in the melter and minimize corrosion of melter refractory and Inconel™ 690.
- Testing in the SRTC shielded cells with radioactive Tank 42 sludge will demonstrate the process conditions under nominal operating conditions.
- The addition of 137.5% of the stoichiometrically required acid will ensure the nitrite is sufficiently destroyed during the twelve hour boiling period. This is 10% higher than had been recommended by the Tank 51 Alternative Sludge Only Flowsheet.

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BACKGROUND

The Defense Waste Processing Facility began processing radioactive Tank 51 Sludge in 1996. At the current processing rate, the Tank 51 sludge has been projected to be depleted as early as August 1998. After this time, DWPF will process a combination of the Tank 51 heel and the washed Tank 42 sludge after transfer to Tank 51 (sludge batch 1B).

Because the precipitate feed into DWPF was delayed three months, a sludge-only process was developed for Tank 51 sludge¹. In Tank Precipitation (ITP) startup delays led to the development of a more efficient, REDOX adjusted sludge-only flowsheet^{2,3} (REDOX is a measure of the slurry's reduction/oxidation potential). Both of these flowsheets were developed for Tank 51 (Sludge Batch 1A) only. Further testing is required to demonstrate a sludge-only process for Tank 42 (Sludge Batch 1B). This document details the testing performed in support of the Tank 42 sludge-only processing.

INTRODUCTION

To determine the processing parameters for the new sludge batch, several experiments were run at differing processing conditions. During the experiments, the process was monitored for problems such as foaming, excessive offgas deposits leading to plugging, and for target parameters, such as nitrite destruction and offgas composition and generation.

The objective of this testing was to complete a series of experiments (duplicating the expected SRAT and SME processing conditions in laboratory scale vessels) to supply the shielded cells with a set of processing parameters for their verification run. The important parameters that were determined include:

- The maximum hydrogen generation rate during each SRAT and SME processing cycle.
- The maximum nitrous oxide (N₂O) generation rate during each SRAT and SME processing cycle.
- The minimum time necessary for completing the steam stripping of mercury to reach the 0.45 wt % mercury limit in the SRAT product.
- The correct acid addition scheme necessary to produce a meter feed with a redox of 0.1 -0.2 Fe²⁺/ΣFe.
- The acid stoichiometry necessary to achieve complete nitrite destruction in the SRAT (as defined as the nitrite concentration less than 1000 ppm).
- The quality of the melter feed as measured by SME acceptability tests (PCCS and PCT).

DISCUSSION

EXPERIMENTAL

Method

Six four-liter scale SRAT/SME processing runs were completed in the 772-T lab at TNX. Each of the six runs consisted of a prototypic DWPF SRAT and SME cycle. The experimental setup was designed to volumetrically scale the DWPF vessels, flows, and feed-rates. For example, 1.8 L of sludge was used in each of the six runs. This is a scale factor of 1/11,360th of DWPF scale based on a 6000-gallon DWPF sludge batch. Thus the 2-gallon/minute acid addition rate was scaled down to 0.67 ml/min. Appendix A contains a sketch of the experimental setup, the laboratory run plan and the scaled conditions used for these experiments.

The SRAT cycle includes all of the important DWPF processing steps as agreed to by DWPF Engineering and ITS. The SRAT cycle includes sludge preparation, sludge analysis, batching calculations, heat-up to 93°C, addition of first nitric acid, then formic acid, heat-up to boiling, concentration down to 6000 gallons, then 12 hours of boiling. The key activities in the SRAT cycle include the neutralization of the sludge, reduction of various metals including manganese and mercury, and destruction of nitrite (defined as <1000-ppm nitrite ion left in solution). Key data includes hydrogen and N₂O generation rates, nitrite destruction, and foaming likelihood.

The SME cycle includes the initial addition of a frit 200-water-formic acid slurry, boiling off water added with the frit-water-slurry, a second addition of a frit 200-water-formic acid slurry, boiling off water to reach a target solids loading of 45 wt% total solids, cool-down, and sampling. Key data includes hydrogen generation rate, glass quality, glass redox and foaming likelihood.

Table 1 compares the different parameters of each of the six runs.

Table 1 - Experimental Plan

<u>Run No.</u>	<u>Sludge*</u>	<u>Noble Metals</u>	<u>Redox Adjustment</u>	<u>Acid Target</u>
1V	1B	Prototypic Tank 42+10%	SME	125%
2V	1B	Prototypic Tank 42+10%	SRAT	125%
3V	1B	HM†	SRAT	125%
4V	1B	Prototypic Tank 42+10%	SRAT	Maximum‡
5V	1B	Prototypic Tank 42+10%	SRAT	137.5%
6V	1B	Prototypic Tank 42+10%	SRAT	125%

* 1B refers to the sludge combination of Tanks 42 with the heel of Tank 51 which will be the next sludge batch that DWPF processes.
† The HM rhodium concentration is approximately 7 times higher than the expected Tank 42 noble metal concentration.
‡ Maximum acid is an addition of a full tank of formic acid (480 gallons) to the SRAT.

- 1V. The first batch was processed identically to the current DWPF sludge-only processing strategy. 125% of the stoichiometric acid is added in the SRAT. Some formic acid is added but not enough to produce an appropriate redox. After the SME cycle was complete, 90 wt % formic acid was added to increase the redox (as defined by the difference between formate concentration and nitrate concentration) $F-N = 0.4 M^{\S}$. The SME was boiled after the formic acid addition to determine the maximum hydrogen generation.
- 2V. The second batch was identical to the recommended redox adjusted sludge-only flowsheet the DWPF plans to implement in FY98. 125% of the stoichiometric acid was added in the SRAT. Both 50 wt % nitric acid and 90 wt % formic acid were added to target a glass redox of $0.2 Fe^{2+}/\Sigma Fe$. No formic acid was added to the SME to adjust the redox.
- 3V. The third batch was identical to 2V except the DWPF design basis levels of noble metals and mercury were added to the sludge to create a bounding hydrogen generation rate.
- 4V. The fourth batch was also identical to 2V except that enough formic acid was added to the SRAT to simulate the addition of the entire content of the formic acid feed tank to the SRAT. This experiment was designed to represent the maximum credible deviation in the amount of formic acid that could be added to the SRAT in a single addition during Tank 42 Sludge only processing.
- 5V. The fifth batch was also identical to 2V except that 10% additional formic acid was added to the SRAT to ensure the nitrite was destroyed in the SRAT.
- 6V. The sixth batch was identical to 2V.

In all of the runs, the nitric and formic acids were fed at 93 °C. The SRAT contents were then heated to boiling and held there for at least 12 hours. In the case of the high mercury run (3V), the sludge was held at boiling longer in order to steam strip the mercury from the sludge. At the conclusion of the SRAT cycle, the SME cycle was commenced. Frit, water, and formic acid were added to the kettle in two installments. The SME cycle was considered over when the correct amount of water was collected to bring the kettle solids to 45-wt %. During all of the runs, slurry pH, offgas composition and inlet gas flow-rates were monitored online. Samples were pulled every two hours to monitor the nitrite, nitrate and formate ion concentrations in the slurry.

Acid Addition Strategy

Concentrated formic acid (90-wt %) and nitric acid (50-wt%) were used during processing. Total acid additions were based on total acid to achieve stoichiometry and an acid mix to produce a redox target (0.1 – 0.2 $Fe^{2+}/\Sigma Fe$ redox ratio in the glass). Appendix B contains the calculations used to determine the acid additions for each run. Dow Corning 544 antifoam was added per the DWPF antifoam strategy (100 ppm on a total solution basis, 1 part antifoam: 19 parts water). An ammonia scrubber was also installed on the SRAT/SME vent stream.

[§] The F-N number is the old description of the redox of the melter feed. Because the new redox adjustment method (based on the definition of redox as the ratio of $Fe^{2+}/\Sigma Fe$ in the glass) has yet to be implemented, all redox numbers will be reported in both terms throughout this document.

Sludge Composition

The sludge used in each of these runs contained approximately 16 wt % solids and represented the type of sludge that DWPF is expected to process. The sludge was prepared using a Tank 51 sludge-simulant (a nonradioactive simulant containing all the major sludge components except Uranium). The Tank 51 simulant was chosen because it is the sludge simulant closest to the Tank 42 composition. The Tank 51 simulant was doped with manganese, aluminum, and SiO₂ since these components are significantly higher in the Tank 42 simulant. In addition, the noble metals and mercury were added prior to each run as discussed in the previous section (10% higher noble metals than Tank 42 concentration). The Tank 42 noble metals and mercury were significantly higher than Tank 51. The trimmed sludge 1B simulant was then analyzed for solids, elementals, total base (pH 5.5) and density.

Table 2 - Sludge Properties Comparison

	Batch 1B	Tank 42 (before storage)	Tank 42 (after 2 years of storage)
Total solids (wt %)	16.26	16.03	15.00
Specific gravity	1.13	1.123	1.113

Table 3 - Sludge Composition Comparison

Species	Batch 1B <i>Simulant</i> Wt % Solids	Tank 42 (before storage) Wt % Dried Solids	Tank 42 (after storage) Wt % Dried Solids	
Insoluble Species	Al	8.19	8.16	7.52
	Ag	0.01	0.01	0.01
	Ca	2.50	2.30	2.12
	Cl	0.00	0.02	0.01
	Cr	0.17	0.16	0.15
	Cu	0.02	0.02	0.02
	F	0.01	0.03	0.03
	Fe	25.10	22.24	20.49
	Hg	0.99	0.99	0.91
	K	0.35	0.33	0.30
	Li	0.00	0.00	0.00
	Mg	1.23	1.29	1.19
	Mn	3.70	3.69	3.40
	Na	9.55	8.34	11.15
	Ni	0.38	0.36	0.33
	P	0.04	0.99	0.91
	Pd	5.36E-04	1.36E-03	1.26E-03
	Pu	0.00	0.01	0.01
	Rh	4.33E-03	4.45E-03	4.10E-03
	Ru	0.00	0.02	0.02
Se	5.36E-04	1.22E-03	1.13E-03	
Si	1.27	1.27	1.17	
Te	0.00	0.01	0.01	
Ti	0.04	0.02	0.02	
U	0.00	2.79	2.57	
Zn	0.16	0.09	0.08	
Soluble Species	C ₂ O ₄	0.12	0.12	0.16
	Cl	0.02	0.02	0.02
	CO ₃	2.16	2.16	9.69
	Cr	0.00	0.00	0.00
	F	0.03	0.03	0.04
	K	0.00	0.00	0.00
	Li	0.00	0.00	0.00
	Na	9.55	8.34	11.15
	NO ₂	5.49	6.65	11.18
	NO ₃	1.84	2.10	0.29
	OH	0.22	1.60	0.17
	PO ₄	0.79	3.03	4.19
	SO ₄	0.45	0.45	0.62

Frit Addition Strategy

Two equal additions of frit 200, water and formic acid were designed to duplicate the frit slurry that is transferred into the SME. No water was added to simulate the addition of Frit-decon water to the SME. The frit 200 was added dry through a funnel to the kettle, followed by the addition of 90-weight percent formic acid, and then water. The addition of frit was predicted by PCCS based on the SRAT solids quantity remaining at the completion of the SRAT cycle. PCCS was based on the SRAT receipt sample that was analyzed once for all the experiments.

RESULTS

Nitrite Destruction

The DWPF Chemical Process Cell hydrogen flammability control system is comprised of control air purges to the SRAT and SME to dilute the concentration of the hydrogen that is generated. In addition, the hydrogen concentration is monitored to prevent the formation of a flammable mixture. The maximum hydrogen generation rate (design basis) was determined in previous experiments to be 0.65 lbs/hr and 0.23 lbs/hr for the SRAT and SME respectively).⁴

Nitrite destruction is believed to be necessary for the generation of hydrogen during SRAT and SME processing. The sludge has approximately 8,000 mg/L of nitrite in the SRAT feed and the nitrite must be destroyed** during the twelve hour boiling phase in the SRAT to produce the maximum hydrogen. This twelve hour boiling time is consistent with the recommendation in the Tank 51 processing⁵. The more acid added the faster the nitrite destruction.

The nitrite destruction is summarized in Table 5 and Figure 1. When 125% of the stoichiometric acid was added and Tank 42 level noble metals were present in the sludge (runs 1V, 2V, and 6V), the nitrite was not sufficiently destroyed during the twelve hours of boiling. When 125% of the stoichiometric acid was added and HM levels of noble metals were present in the sludge (run 3V), the nitrite was sufficiently destroyed during the twelve hours of boiling. When 137.5% or more of the stoichiometric acid was added and Tank 42 level noble metals were present in the sludge (runs 4V and 5V), the nitrite was sufficiently destroyed during the twelve hours of boiling.

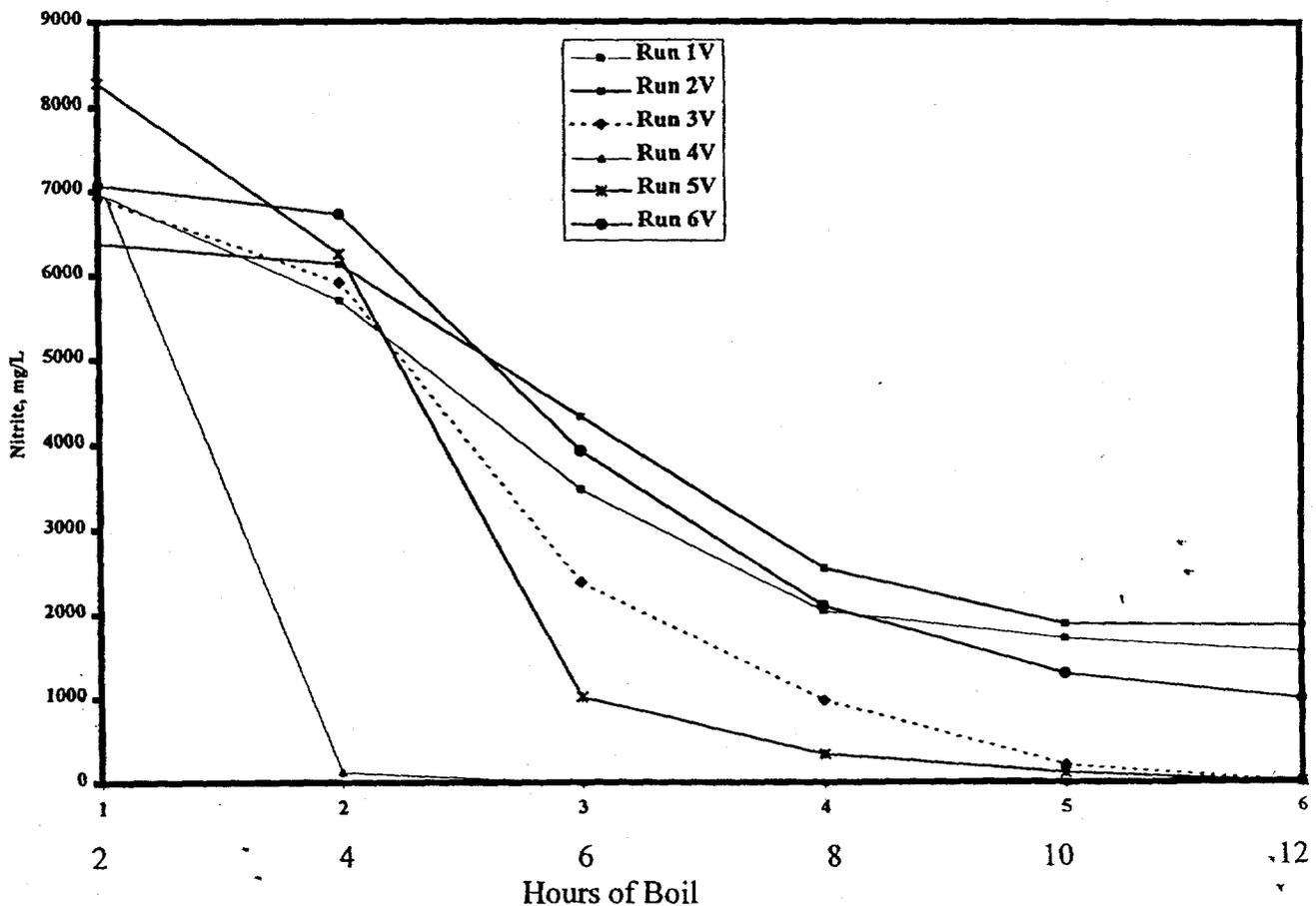
** Nitrite destruction has been defined previously as <1000 ppm in solution but ghis is difficult to measure in ...

Table 4 - Tank 42 SRAT Product Nitrite Concentration

Run	Nitrite	Acid	Noble Metals
Run 1V	1,703	125%	Tank 42
Run 2V	1,879	125%	Tank 42
Run 3V	200	125%	HM
Run 4V	1	>137.5%	Tank 42
Run 5V	111	137.5%	Tank 42
Run 6V	1,288	125%	Tank 42

Recommendation: Based on this testing, the addition of 137.5% of the stoichiometrically required acid will ensure the nitrite is sufficiently destroyed during the twelve hour boiling period. This is 10% higher than had been recommended by the Tank 51 Alternative Sludge Only Flowsheet.

Figure 1 - Nitrite Destruction



Offgas Composition

The offgas was monitored to compositionally characterize the offgas. An internal helium standard was used to calculate the outlet flow^{††}. The offgas was monitored using a Gas Chromatograph. Figure 2 shows the hydrogen generation rate during each of the six SRAT cycles. Figure 3 shows the hydrogen generation rate during each of the six SME cycles. The flow rates reported were scaled to a 6000-gallon DWPF batch. The highest amount of hydrogen occurred in the run where excess formic acid was added, run 4V. The second highest peak occurred in run 3V, the run in which HM levels of noble metals were added. Note also that the hydrogen peak occurred in the SME in all runs except run 4V.

The maximum hydrogen generation occurred in the two extreme runs, 3V (HM levels of noble metals) and 4V (maximum acid addition). The maximum peak observed during processing corresponds to a rate of 0.31 lbs/hr in the DWPF SRAT during run 3V and 0.17 lbs/hr in the DWPF SME during run 4V. The upper limits in the SRAT and SME are 0.65 lbs/hr and 0.23 lbs/hr respectively. Table 6 shows the peak hydrogen generation rate in each of the 6 runs.

The hydrogen generation was very low in runs with Tank 42 level noble metals and normal (125%) acid addition. In these runs the maximum hydrogen in the SRAT was 0.01 lb/hr in the SRAT and 0.085 lb/hr in the SME.

Because of incomplete destruction of nitrite during the SRAT cycle in Run 2V and the extremely low hydrogen generation, Run 2V was duplicated to ensure that nitrite could be adequately destroyed and conservative hydrogen generation rates were determined. First, run 2V was repeated with 10% additional acid (137.5% versus 125%). In run 5V, nitrite was destroyed in the SRAT and a higher hydrogen generation rate was calculated. In run 6V, designed to duplicate 2V, nitrite was not completely destroyed in the SRAT and a lower hydrogen generation rate was calculated.

Table 5 - Hydrogen Generation Summary (DWPF Basis)

Description ^{††}	Experiment					
	1V	2V	3V	4V	5V	6V
Noble Metals	42	42	42	42	42	42
Redox Adjustment	SME	SRAT	SRAT	NA	SRAT	SRAT
Acid	125%	125%	125%	254%	137.5%	125%
Max SRAT H ₂	0.010 lb/hr	0.001 lb/hr	0.104 lb/hr	0.310 lb/hr	0.004 lb/hr	0.001 lb/hr
Max SME H ₂	0.059 lb/hr	0.000 lb/hr	0.167 lb/hr	0.162 lb/hr	0.085 lb/hr	0.012 lb/hr
Max H ₂ Concentration	0.158 vol %	0.001 vol %	0.368 vol %	0.443 vol %	0.005 vol %	0.001 vol %

^{††} A known rate of helium was added to the inlet air stream to the SRAT. The outlet gas flow was monitored for helium. The total outlet flow was calculated based on the dilution of the helium by air and other generated gases.

^{††} DWPF limits in parentheses

Figure 2 - SRAT Cycle Hydrogen Generation

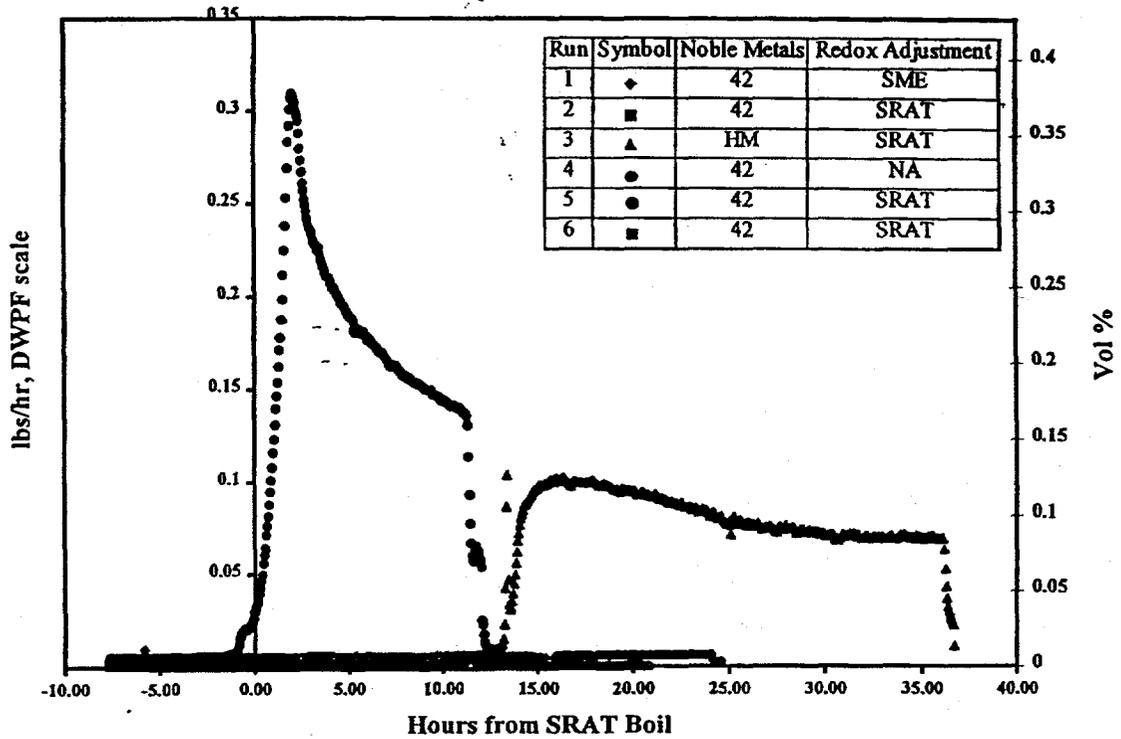
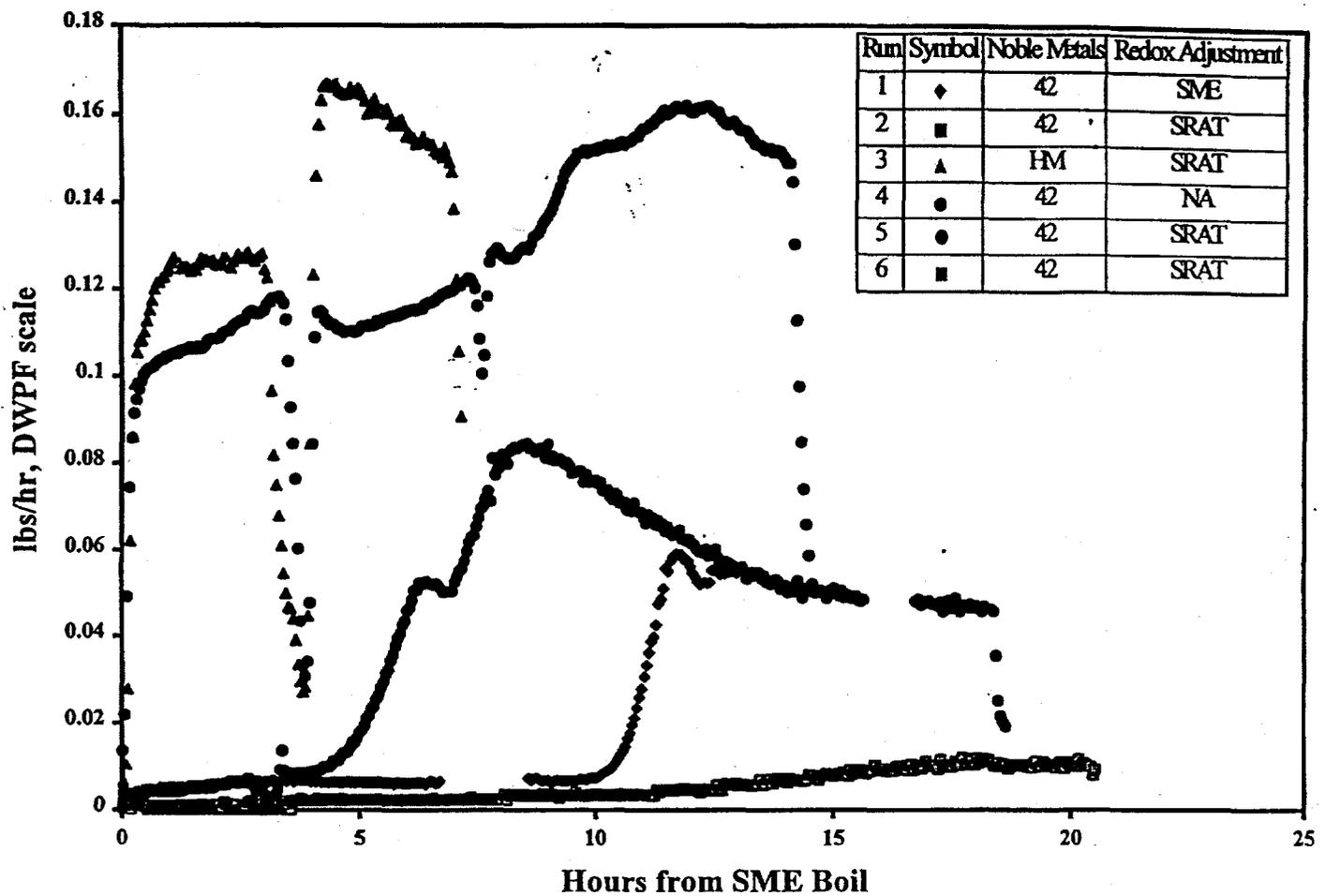


Figure 3 - SME Cycle Hydrogen Generation



The maximum N₂O generation rate was 77.7 lb/hr which resulted in a N₂O concentration of 3.85 volume % in the SRAT cycle of Run 4V. This concentration is well below the 20-volume % basis for DWPF flammability calculations. There was no measured N₂O in the SME cycle during any of the runs. Table 7 summarizes the N₂O generation rate and volume % in each of the 6 runs.

Table 6 - Nitrous Oxide Summary

Description ^{SS}	Experiment					
	1V	2V	3V	4V	5V	6V
Noble Metals	42	42	HM	42	42	42
Redox Adjustment	SME	SRAT	SRAT	NA	SRAT	SRAT
Acid	125%	125%	125%	Maximum	137.5%	125%
Max SRAT N ₂ O	23.802 lb/hr	5.411 lb/hr	21.228 lb/hr	47.746 lb/hr	45.102 lb/hr	23.152 lb/hr
Max SME N ₂ O	0.000 lb/hr	0.000 lb/hr	0.000 lb/hr	0.000 lb/hr	0.000 lb/hr	0.000 lb/hr
Max N ₂ O (<20%)	1.258 vol %	0.204 vol %	1.097 vol %	3.348 vol %	2.442 vol %	1.009 vol %

Steam Stripping

The mercury concentration in the SRAT product must be below 0.45 wt % mercury (design basis) to minimize corrosion in the melter offgas piping and minimize the mercury emission to the environment. Mercury is present as mercuric oxide (HgO) in the sludge. Mercuric oxide must be reduced to elemental mercury by formic acid (the reductant). The elemental mercury is then removed from the sludge by steam stripping during the boiling phase in the SRAT. The mercury is collected and accumulates in the Mercury Water Wash Tank (MWWT). The mercury in Tank 42 sludge is approximately 1.0 wt % in the sludge solids. It requires approximately 6.9 hours of boiling at a steam flowrate of 5000 lb/hr to strip enough mercury from the SRAT to meet the 0.45 wt % SRAT product goal with Tank 42 sludge and 35.9 hours of boiling in batch 3V where HM levels of mercury were added. Note this is based on the assumption that 750 pounds of steam are required to remove one pound of mercury. It also assumes complete separation of mercury in the MWWT. Table 8 summarizes the mercury in the SRAT product for the six runs.

Table 7 - SRAT Product Mercury Concentration

Run	Mercury, ppm	Mercury, wt %
1V	101	0.051
2V	139.7	0.070
3V	1457/1568	0.73/0.79
4V	29.6	0.015
5V	60.3	0.030
6V	105.0	0.053

All runs except the high (HM) mercury and noble metal run met the mercury target of 0.45 wt%. A longer stripping time may be required for high mercury sludge but the twelve-hour SRAT boil is sufficient for the Tank 42 sludge. The SRAT product from Run 3V was reanalyzed to confirm the high mercury content.

^{SS} DWPF limits in parentheses

Acid Additions and Redox Targets

Kevin Brown, SRTC/ITS, developed a spreadsheet to calculate the nitric and formic acid addition to reach a redox target for each run. The calculation for each of the runs is summarized below in Table 9. The details of the calculations are summarized in Appendix B.

Table 8 - Acid Addition and Redox Target

Description	Experiment					
	1V	2V	3V	4V	5V	6V
Noble Metals	42	42	HM	42	42	42
Redox Adjustment	SME	SRAT	SRAT	NA	SRAT	SRAT
Redox Target ($Fe^{+2}/\Sigma Fe$)	None	0.20	0.20	None	0.20	0.20
Acid Target	125%	125%	125%	254%	137.5%	125%
Batch Size	6000 gallons					
90 wt % Formic Acid	171.2 gal	212.6 gal	219.1 gal	480.0 gal	229.6 gal	210.4 gal
50 wt % Nitric Acid	200.0 gal	106.8	107.6 gal	106.8 gal	119.7 gal	105.0 gal

The glass redox (reduction/oxidation potential) was measured for the first four experiments by the SRTC Mobile lab. The results are summarized in Table 10 (reporting redox as F-N as DWPF does currently) and Table 11 (reporting redox as $Fe^{+2}/\Sigma Fe$, a better measure of redox than F-N). Table 10 summarizes the redox (F-N) data for the first four experiments. The F-N (difference between the Formate and Nitrate Molar concentrations) was calculated for each of these runs based on the Formate and Nitrate analyses corrected for a 45-wt% slurry. Table 11 summarizes the redox ($Fe^{+2}/\Sigma Fe$) data for the first four experiments. The "Target Redox" column was based on the redox target as input into the Kevin Brown spreadsheet. The "Calculated Redox" column was a calculation of the redox based on the Kevin Brown redox correlation⁶. The "measured redox" column was the redox as measured by the SRTC Mobile Lab of glasses vitrified by Alex Cozzi.

The measured redox for the first three experiments was very oxidizing. This is much lower than had been targeted for Runs 2V and 3V. It should be noted that it is very difficult to vitrify and measure the redox of the resulting glass without overly oxidizing the glass. It is believed that the actual glass produced in the DWPF melter should be much more reducing than the glasses that were analyzed by the SRTC Mobile Lab. Note that in glasses made in sludge only testing, higher redox measurements have been achieved only in glass produced in the 774-A melter run with Tank 51 sludge⁷.

Table 9 - Predicted versus Actual Redox (F-N) of Melter Feeds

Run	Calculated F-N	
	Mobile Lab	TNX
1V	0.33	0.19
2V	0.62	0.58
3V	0.35	0.35
4V	0.99	1.26

Table 10 - Predicted versus Actual Redox ($Fe^{+2}/\Sigma Fe$) of Melter Feeds

Run	Target Redox	Predicted Redox Based on Melter Feed Formate and Nitrate		Measured Redox
		SRTC	TNX	
		Mobile Lab	TNX	
1V	<0.03	0.03	0.03	<0.03
2V	0.20	0.23	0.22	<0.03
3V	0.20	0.18	0.20	<0.03
4V	0.45	0.33	0.39	0.36

SME Product Acceptability Tests

Product Composition Control System (PCCS) Acceptability

The SME products were vitrified in alumina crucibles by Alex Cozzi and Pat Toole. The SRTC Mobile Lab completed the elemental analyses to verify that the process produced quality glass quality glass. The glass elemental analysis was entered into PCCS acceptance spreadsheet (PCCS Version 3.2, appendix B). The SME products passed all but two of the PCCS acceptability tests. The two PCCS acceptability tests that failed were viscosity and liquidus, both DWPF processability constraints. Note that these glasses failed the Liquidus Temperature and High Viscosity Constraint at a 95% confidence level.

The probable explanation for failing to meet all PCCS acceptability constraints was that frit was underadded to the sludge because the glass was lower in frit components (Li and B) than had been predicted by PCCS. Also, some of the sludge components were higher than expected (Al, Mn). However, the major glass components (Fe, Si, Na) were very close to the PCCS prediction. In addition, the ratio of key sludge components (such as Fe/Al) was different in the glass than it was in the sludge suggesting that Fe was lost during testing or the Al analysis in the sludge was low.

One way to understand what may have caused the failure of the test is to look at the ratio of the measured glass composition to the expected glass composition. The expected glass composition was predicted by PCCS based on the sludge sample. If frit was over-added, the frit components (Si, Li,

and B) will be higher than predicted. If sludge was over-added, the sludge components (Fe, Al, Mn, Mg, Ca, and Na) will be higher than predicted. Note that many of the sludge ratios are greater than 1 and many of the frit ratios are <1, suggesting that frit was under-added. However, the iron and silicon ratios are approximately 1 as expected (iron and silicon oxides are the major components in the sludge). The results of the PCCS acceptability tests are summarized in Table 12.

Table 11 - PCCS Acceptability Data
Passed PCCS Acceptance version 2.5?

Criteria	1V	2V	3V	4V
B Leaching	Yes	Yes	Yes	Yes
Li Leaching	Yes	Yes	Yes	Yes
Na Leaching	Yes	Yes	Yes	Yes
Liquidus Temp	No	No	No	No
High Viscosity	No	No	No	No
Low Viscosity	Yes	Yes	Yes	Yes
Homogeneity	Yes	Yes	Yes	Yes
Al ₂ O ₃	Yes	Yes	Yes	Yes
High Conserve	Yes	Yes	Yes	Yes
Low Conserve	Yes	Yes	Yes	Yes
Low Frit	Yes	Yes	Yes	Yes
High Frit	Yes	Yes	Yes	Yes
TiO ₂	Yes	Yes	Yes	Yes
NaCl	Yes	Yes	Yes	Yes
NaF	Yes	Yes	Yes	Yes
Cr ₂ O ₃	Yes	Yes	Yes	Yes
Na ₂ SO ₄	Yes	Yes	Yes	Yes
Cu	Yes	Yes	Yes	Yes
P ₂ O ₅	Yes	Yes	Yes	Yes

An analysis of the data indicated that an over-addition of frit might have led to the high viscosity and liquidus. The Li and B concentrations in the glass were higher than predicted (compared to the initial PCCS prediction based on the sludge analysis). In addition, many of the sludge components, including aluminum, calcium and manganese were lower than predicted. The major components in the glass (>1 wt %) are summarized in Table 13.

Table 12 - Glass composition versus PCCS prediction

Sludge Component	PCCS Expected, wt %	Actual, wt %	Ratio, Actual/Expected
Al	2.65	3.16	1.19
Ca	0.98	1.21	1.24
Fe	8.46	8.63	1.02
Mg	1.28	1.26	0.98
Mn	1.10	1.22	1.11
Na	8.73	8.34	0.96
Frit Component	PCCS Expected, wt %	Actual, wt %	Ratio, Actual/Expected
B	2.77	2.26	0.81
Li	1.72	1.47	0.85
Si	24.75	25.03	1.01

However, another explanation for failing to meet the PCCS acceptance criteria is there could have been an error in the sludge composition or the glass composition. Since the only source of iron and aluminum is from the sludge, the ratio of iron to aluminum (Fe/Al) should be the same in the sludge and the glass. However, the ratio of Fe/Al was 3.19 in the sludge versus 2.81 in the glass suggesting there was 10% more aluminum than had been predicted based on the sludge receipt sample. Future testing should be done with the SRAT product sample instead of the sludge receipt sample (consistent with DWPF practice) to minimize the error in calculating the frit addition.

Product Consistency Test (PCT) Acceptability

The glasses produced from each of the first four experiments were tested using the ASTM C1285 (Product Consistency Test, PCT) protocol to determine the seven day normalized release for Na, Li, K, B, and Si versus the EA Glass Standard⁸. The normalized release values for these cations is significantly^{***} lower than the EA Glass Standard.

The PCT results (average of three analyses) are summarized in Table 14. Glass compositions and complete PCT results are tabulated in Appendix C. The table in Appendix C shows that the blind multi-element standard for the element Na was not in control, e.g. >10% difference between the standard and the value reported. Therefore, the PCT procedure does not permit reporting/usage of the Na values. It should also be noted that for the B results from the 7-day PCT shown in Table 14 are in excellent agreement with the B release predicted by PCCS (Table C-1, Appendix C), e.g. 0.56 g/L predicted vs. 0.59, 0.56, 0.62, 0.61 g/L measured (Table 14).

^{***} Normalized boron release values for the PCT are less than two standard deviations of the variability of EA glass.

Table 13 - Product Consistency Test (PCT)

Average Normalized Release

Glass B	Si	Na	Li	K	
	g/L	g/L	g/L	g/L	
ARM	0.59	0.32	##	0.68	N/A
T1V	0.56	0.33	##	0.63	N/A
T2V	0.62	0.38	##	0.66	N/A
T3V	0.61	0.35	##	0.65	N/A
T4V	0.84	0.39	##	0.69	N/A
EA	15.03	3.96	##	7.91	2.59

Measurements of Na in the multi-element solution standard were not in the acceptable range to permit reporting data.

Additional Hydrogen Testing

Additional hydrogen testing was completed because there was higher hydrogen production in the run with more nitric acid (1V) than the runs with less nitric acid (runs 2V and 6V). Two additional SRAT/SME cycles (Runs 5V and 6V) were added in an attempt to understand the lower hydrogen generation in Run 2V compared with Run 1V. Run 6V was a duplicate of 2V. Run 5V was similar to Run 2V, except that an additional 10 % acid was added. Extra acid was added because it appeared that inadequate acid had been added during experiments 1V and 2V (nitrite was not completely destroyed). Table 15 summarizes the results of these similar experiments.

Table 14 - Hydrogen Generation in Similar Experiments

Description ^{†††}	Original Experiments		Additional Experiments	
	1V	2V	5V	6V
Noble Metals	42	42	42	42
Acid Mix	More Nitric	More Formic	More Formic	More Formic
Acid	125%	125%	137.5%	125%
Max SRAT H ₂ (<0.65)	0.010 lb/hr	0.001 lb/hr	0.004 lb/hr	0.001 lb/hr
Max SME H ₂ (<0.23)	0.059 lb/hr	0.000 lb/hr	0.085 lb/hr	0.012 lb/hr

Based on the two additional experiments, it is apparent that the hydrogen generation in Run 2V and 6V led to a minimal hydrogen production in both the SRAT and SME cycle. This hydrogen generation is much less than Run 1V.

In an attempt to understand why the experiments with less nitric acid led to lower hydrogen generation, eight additional hydrogen study experiments were completed. The experiments were all completed using the same melter feed, a combination prepared by combining the melter feed from

^{†††} DWPF limits in parentheses, Hydrogen generation calculated based on a 6000 gallon DWPF Batch.

Runs 5V and 6V. These melter feeds were chosen because they were relatively low in hydrogen generation.

At the beginning of each experiment, 250 ml of melter feed was added to a one-liter kettle. Next, the appropriate acid was added to the agitated mixture using an autotitrator. The mixture was heated to boiling and maintained at boiling for four hours. An air purge with an internal helium standard was used to measure the hydrogen generation. The conditions for the eight additional runs are summarized in Table 16.

Table 15 - Plan for Additional Hydrogen Study

Run	Added Acid Molarity	Acid
11C	0.00 M	none
12C	0.376 M	formic
13C	0.752 M	formic
14C	0.376 M	nitric
15C	0.376 M	HCl
16C	0.188 M	formic
17C	0.094 M	formic
18C	0.376 M	formic

The results of the experiments are summarized in Table 17. Note that four of the runs (shaded in Table 16 and 17) were completed with the addition of 0.376 moles of formic, nitric or hydrochloric acid (planned to raise acidity by 0.376 M). The runs with added formic acid had much higher hydrogen generation than the runs with the addition of strong acids, nitric and hydrochloric acid. This is the opposite of the trend noted in the variability runs. Note also that the measured acidity of the melter feed after the addition of the acid was less than had been predicted. As a result of the inconsistent data collected during this experimentation, additional experiments are recommended to study the generation of hydrogen during SRAT and SME cycles.

Table 16 - Hydrogen Generation during Additional Hydrogen Study

Run	11C	17C	16C	12C	18C	13C	14C	15C
Added Acid	None	formic	formic	formic	formic	formic	nitric	HCl
Added acid, M	0.000	0.094	0.188	0.376	0.376	0.752	0.376	0.376
Expected Acidity, M	-0.037	0.057	0.151	0.339	0.339	0.715	0.339	0.339
Measured Total Acid after acid addition, M	-0.037	0.042	0.071	0.169	0.187	0.436	0.189	0.195
H ₂ Peak, volume %	0.009	0.022	0.034	0.092	0.089	0.118	0.049	0.050
H ₂ Peak, lb/hr ^{†††}	0.002	0.005	0.008	0.023	0.022	0.029	0.012	0.012

^{†††} Hydrogen generation calculated based on a 6000 gallon DWPF Batch.

ACKNOWLEDGMENTS

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Thanks also to Paul Monson, Russ Eibling and Jim Marek, who provided technical insight throughout the study. Alex Cozzi and Pat Toole completed the drying and vitrification of the melter feed. David Best and Eric Frickey completed the PCT, anion, elemental analysis, and redox measurements in the SRTC Mobile Lab.

Kim Willoughby, Andy Birk, Mary Johnson, and Chris Boley prepared the sludge simulant.

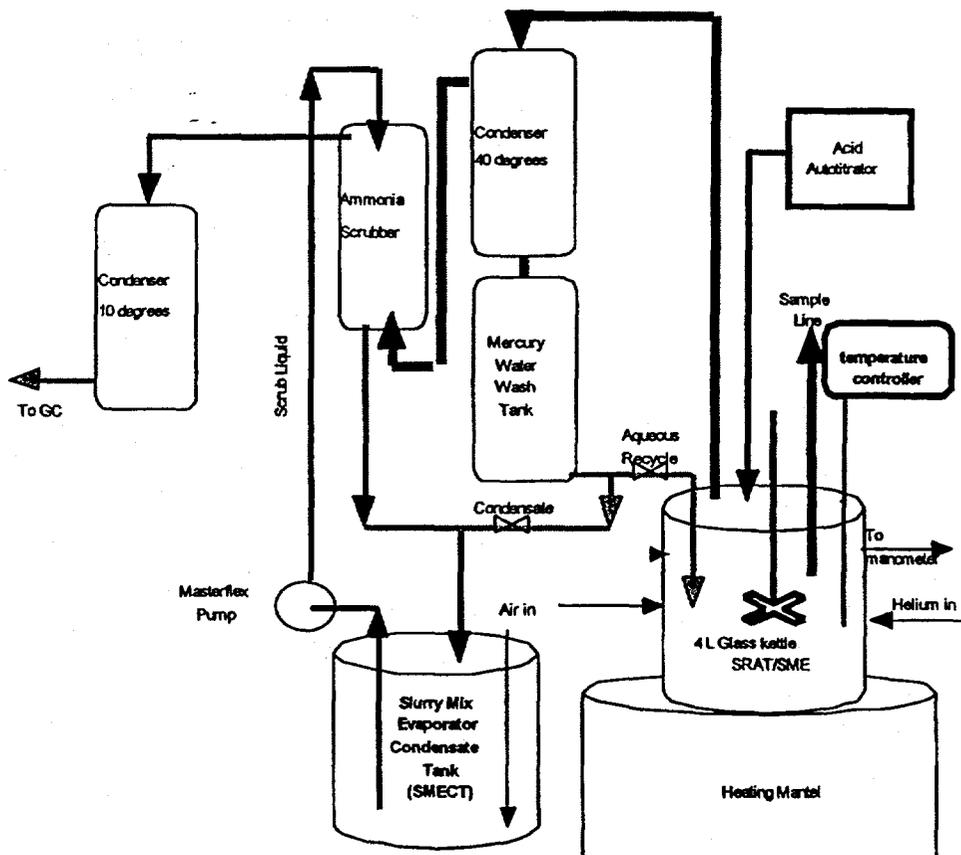
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 - ⁸ C.M. Jantzen, N.E. Bibler, D.C. Beam, C.L. Crawford, and M.A. Pickett, Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material (U), WSRC-TR-92-346, Rev. 1, June 1, 1993.

Appendix A Experimental Setup and Laboratory Procedure

The six variability study experiments were conducted in the 772-T Laboratory. The experimental setup is summarized in Figure A1. The equipment and instruments used are described below:

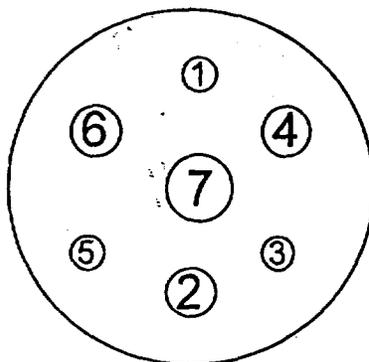
Figure A1



SRAT/SME Kettle

A four-liter Pyrex kettle with a glass lid was used for the variability study. The kettle lid contained the following seven openings or ports (Figure A2). The instrument that uses each of the ports is described below:

Figure A2 - SRAT/SME Kettle Lid Ports



Port 7 - Agitator

The contents of the SRAT/SME kettle was mixed using an agitator. The agitator had two impellers, a bottom flat blade impeller and the upper marine impeller. A Variable Speed Mixer powered the agitator. A variac speed controller controlled the agitation speed.

Port 4 - pH Probe

A calibrated pH electrode was installed through the kettle top into the slurry. The electrode was connected to an Omega pH meter.

Port 6 - Slurry Sampler

A sampler was installed to allow the sampling of the SRAT/SME kettle contents as needed.

Port 3 - Antifoam addition Port

An antifoam addition port was designed to allow the addition of antifoam as needed.

Port 2 - Acid Addition

Nitric and formic acid were accurately metered into the kettle use a Mettler Toledo Autotitrator. Only one acid was present in the hood at a time to prevent the inadvertent addition of concentrated nitric acid to concentrated formic acid.

Port 5 - Air and Helium purge

The inlet gas flows to the kettle were controlled by individual MKS Flow Meter connected to a MKS four-channel set-point controller. A separate air and helium purge was added to the kettle

to produce a 0.5 volume % helium flow entering the kettle. This was used as an internal standard to calculate the outlet offgas flow since it is difficult to measure reliably during SRAT and SME operations.

Port 2 - Thermocouple

RTD probes measured the Kettle slurry and SMECT liquid temperatures. The temperature signal was an input to the Dyna-sense[®] Benchtop Temperature Controller that controlled the slurry temperature as needed.

Port 5 - Pressure Surge U-tube manometer

The kettle pressure was limited to approximately 20 inwc by water filled U-tube manometer. Approximately 10 inches of water were in each leg of the manometer. The manometer would relieve if the pressure in the kettle exceeded approximately 20 inwc. It was also used to measure the pressure in the kettle vapor space during processing.

SRAT/SME Kettle Mantle

The four-liter Pyrex kettle rested in an 800 W mantle to allow temperature control of the kettle slurry. A Dyna-sense[®] Benchtop Temperature Controller controlled the power to the mantle.

SRAT/SME Condenser

The SRAT/SME condenser is a counter current heat exchanger. The offgas from the kettle enters the top of the condenser, flows through the shell of the heat exchanger and the cooled offgas exits near the bottom of the condenser. The cooled offgas enters the ammonia scrubber after exiting the condenser. The condensate drains by gravity to the Mercury Water Wash Tank (MWWT). The cooling water enters the top of condenser, flows through the heat exchanger tubes and exits at the bottom of the condenser. The cooling water was controlled at 40°C by a Haake constant temperature water bath.

Mercury Water Wash Tank (MWWT)

The MWWT collects condensate and traps elemental mercury. The condensate drains by gravity to the Mercury Water Wash Tank. An underflow/overflow weir prevents the transfer of the dense mercury or floating organic from exiting the MWWT. The condensate is transferred either back to the kettle through port 6 during the reflux phase or forward to the Slurry Mix Evaporator Condensate Tank (SMECT).

Ammonia Scrubber

A laboratory scrubber filled with glass Raschig rings removes ammonia from the offgas between the SRAT condenser and the FAVC. The gas enters at the bottom of the scrubber and flows up through scrubber packing and exits at the top of the scrubber. The offgas from the scrubber is fed to the FAVC. The SMECT was filled with 500 ml of a pH 2 condensate at the

start of each experiment. This liquid, plus any condensate produced during the experiments is used as the scrubbing liquid for removing ammonia from the offgas. A Masterflex[®] Pump pumped the liquid from the SMECT to the top of the ammonia scrubber. The liquid flows down the column through the packing and exits at the bottom of the scrubber. This liquid drains by gravity to the SMECT.

Formic Acid Vent Condenser (FAVC)

The FAVC is a counter current heat exchanger. The offgas from the kettle enters the top of the condenser, flows through the shell of the heat exchanger and the cooled offgas exits near the bottom of the condenser. The condensate drains by gravity to the collection area at the bottom of the FAVC. The cooling water enters the top of condenser, flows through the heat exchanger tubes and exits at the bottom of the condenser. The cooling water was controlled at 5°C by a Haake constant temperature water bath.

SMECT Kettle

The SMECT is used to contain the condensate collected from the SRAT/SME kettle and serve as the liquid scrubbing solution source for the ammonia scrubber. The SMECT liquid was maintained at temperature of 50°C. A four-liter Pyrex kettle with a glass lid was used for the variability study.

SMECT Kettle Mantle

The four-liter Pyrex kettle rested in an 800 W mantle to allow temperature control of the kettle slurry. A Dyna-sense[®] Benchtop Temperature Controller controlled the power to the mantle.

SMECT Air Sparge

An air purge was introduced into the SMECT liquid to promote mixing. The air flowrate was controlled by a MKS flow controller and fed to the liquid through a fritted glass disk. This was to simulate the air sparge ring in the DWPF SMECT.

Offgas Gas Chromatograph

The offgas was monitored by an MTI gas chromatograph. Column A was an MTI MS5A 4m column with an argon carrier gas calibrated for helium, hydrogen, oxygen and nitrogen. Column B was an MTI poraplot Q 8m column with an argon carrier calibrated for carbon dioxide and nitrous oxide. The chromatogram was recorded by the MTI EZChrom software version 4 on a PC in the lab.

Laboratory Run Plan

The experiments were controlled using the SRAT/SME Laboratory Run Plan, SRT-PTD-97-0021. The blank procedure is included below:

Run # _____

Date: _____

PREREQUISITES

1. Add sludge to kettle and add specified noble metals. Paste a copy of the noble metal additions in the log book. Transfer the required quantity and wash water #1 (from the run sheet) to the kettle. Mark the current level on the kettle with a marker.
2. Add 500 ml of pH 2 Nitric acid solution to SMECT. _____ ml added.
3. Prepare sufficient 90% formic acid and 50% nitric acid.
4. Prepare 100 ml 1M NaOH solution or make sure sufficient solution is available.
5. Turn on cooling water to offgas condenser. Setpoint = 5°C.
6. Make sure the GC computer has enough memory space for the run (at least 40 Mbyte).
7. Set the GC computer time equal to the clock time. Record the time in the log book.
8. Turn on the GC and its computer and make sure that the computer and GC are a matching set.
9. Install the calibration gas cylinder to the GC and let the GC run five times. If at the end of five runs the GC reading is within 10% of the gas composition in the cylinder, print the calibration check results and write down "pre-cal check and run number" on the printout. Otherwise, select "Calibration" "Level 1" "OK" to calibrate the GC five times. At the end of five runs the GC reading should be within 10% of the gas composition in the cylinder. If it is not, contact the engineer. Print the calibration check results and write down "pre-cal check and run number" on the printout.
10. Turn on the purge gas to kettle with 468.8 sccm of air and 2.86 sccm of helium. Turn on the air to the SMECT at 100 sccm. Connect the outlet flowmeter to perform the leak check. The outlet flow should be ≈600 sccm. If it is not, tighten all connections until the system is leak tight. Write down the leak check in the log book.
11. Disconnect outlet flowmeter.
12. Make sure the He pressure is at 18 psi (inside the lab).
13. Calibrate the PHA pump to provide a flow of 4.33 ml/min, if that has not been done.
14. Transfer required PHA and wash water #1 (from run sheet) to the feed tank. Turn on PHA feed agitator. Setpoint = _____. Pump the PHA in the line up to the kettle & rezero the pump (press the up and down arrows and the Cum.Vol.Read buttons at the same time).
15. Calibrate pH probe and set up the pH meter to provide continuous reading.
16. Add the first antifoam addition and wash water #2 (from the run sheet) to the kettle.
17. Turn on kettle agitator. Setpoint = _____.
18. Pull a 10 ml sample (SRAT-01) from the kettle, record the weight, add 1 ml 1M NaOH solution, record the weight again and other information required on the run sheet. Label and send to lab for formate, nitrate and nitrite analysis.
19. Remove the sampling device and install the nitric acid titrator to the same port.
20. Set up flow from the SMECT to the scrubber by turning on the SMECT pump. Flow = 6 ml/min.
21. Insulate vessel and offgas lines.
22. Start the GC for this run beginning with baseline reading for a few minutes. The beginning GC reading should be approx. 0.5% helium and 99.5% air. If it is not, contact the engineer. Write down the GC time, filename and etc. in the log book. Record the baseline data on the data sheet.
23. Make sure the reflux line is set up so that the condensate is sent to the kettle.
24. Make sure the MWWT is filled to the top line with water.

Run # _____

Date: _____

SRAT CYCLE

1. Start heating the kettle (Turn on power, setpoint at 93°C, voltage load setting at 10). Record the run data every 20 minutes on the data sheet.
2. Turn on heat to SMECT (Temp setpoint = 50 °C).
3. When kettle temperature reaches 70°C, turn the voltage load setting to 8.
4. When the kettle reaches 93°C, add required quantity of 50% nitric acid (from the run sheet) to the kettle at a rate specified on the run sheet and adjust the load setting to maintain the kettle at 93°C. Write down the start and stop time for the addition on the run sheet.
5. When the addition of nitric acid is complete, remove nitric acid titrator and install formic acid titrator to the port and add required quantity of 90% formic acid (from the run sheet) to the kettle at a rate specified on the run sheet and adjust the voltage load setting to maintain the kettle at 93°C. Write down the start and stop time for the addition on the run sheet.
6. When the formic acid addition is complete, remove the titrator and install the sampling device to the port.
7. Pull a 10 ml sample (SRAT-02) from the kettle, record the weight, add 1 ml 1M NaOH solution, record the weight again on the run sheet. Label and send to lab for formate, nitrate and nitrite analysis.
8. Add antifoam and wash water. Add the subsequent antifoams and wash waters (from the run sheet) to the kettle every 12 hours through the antifoam feeding port. Record the additions on the run sheet in the appropriate chronological position.
9. Change the reflux line so that the condensate is sent to the SMECT.
10. Change temperature setpoint to 110 °C to begin heating the kettle to boiling. Set the voltage load setting to 6 after boiling is reached.
11. When kettle temperature reaches boiling, concentrate until the level in the SRAT is equal with the line drawn after the initial sludge addition.
12. When the line in the kettle has been reached, change the reflux line so that the condensate is returned to the kettle. Hold the kettle at boiling in the reflux mode for 12 hours while taking samples every 4 hours. Measure boil-up rate once every two hours (desired 3.33 g/min). Record the data on the run sheet.
13. At the end of 12 hours of reflux, cool the kettle to sub-boiling. Pull a 125 ml sample (SRAT-final) from the kettle, record the weight, add 1 ml 1M NaOH solution, record the weight again on the run sheet. Label and send to lab for formate, nitrate and nitrite analysis.
14. Pump ~ 2500 grams of condensate from the SMECT into a carboy. Record the initial level and the total grams pumped out on the run sheet.

Run # _____

Date: _____

To obtain the SME Run Sheet, enter the data from the SRAT Run Sheet in to the excel spreadsheet in the lab. (Make sure the file name corresponds to the run name). Only enter numbers. Go to the Sheet "SME Run Sheet" and print.

SME CYCLE

1. Reduce the purge rate to 164.6 scc/min of air and 1.33 scc/min of helium.
2. Decrease the SMECT pump flow from 6 ml/min to 3 ml/min.
3. Add the SME antifoam addition and wash water (from the SME run sheet) to the kettle.
4. Open the PHA port for first frit addition and add frit, 90% formic acid and water per run sheet. Record the addition time and the quantity of each chemical on the run sheet
5. Heat to boiling to collect condensate to level marked on SRAT kettle at the start of the SRAT cycle.
6. After the desired level of the second condensate received, shut off the heater to cool the kettle to sub-boiling.
7. Open the PHA port for second frit addition and add frit, 90% formic acid and water per run sheet. Record the addition time and the quantity of each chemical on the run sheet.
8. Heat to boiling and begin collecting condensate #3.
9. Continue boiling until the target condensate level is received in the SMECT. (The kettle should contain 45% solids). Record the condensate level on the run sheet.
10. Remove the kettle insulation and shut off the heater to cool the kettle to sub-boiling.
11. Pull a 125 ml sample (SME-final) from the kettle, record the weight on the run sheet. Do not add any NaOH solution. Label and send to lab for formate, nitrate and nitrite analysis.
12. Stop GC and record the GC time and clock time in the log book. Stop recording run data on the data sheet.
13. Install the calibration gas cylinder to the GC and run the post-cal check. If the check indicates OK, print a copy and write "post-cal check and run number" on the printout, then place the GC in standby. If the check is not within 10% of the cal gas composition, notify the engineer.
14. Pump melter feed into a tared bottle. Record the weight on the run sheet.
15. Pump out the SMECT. Record total amount collected throughout the run on the Run sheet.
16. Complete pH meter post calibration check.
17. Install the outlet flow meter to the purge gas.
18. When the kettle is cool (<50 °C), record the outlet purge flow in the log book. (All 4 four channels).
19. Turn off all instruments.
20. Perform a wt% solids analysis on the SME product. Verify with the engineer that the target has been met.

Appendix B
Scaling and Batching Calculations

The variability study consisted of scaled DWPF SRAT and SME processing cycles. Each run was based on a 2000-gram addition of Tank 42 sludge simulant to the kettle. PCCS was used to determine the quantity of frit 200 required to make quality melter feed. Kevin Brown completed the PCCS calculations based on the sludge composition summarized in Tables B1 and B2. The PCCS calculated batching is summarized in Table B3.

Table B-1 Tank 42 Simulant Sludge Composition

Elementals, wt%	Best Estimate					
	ADS Sample #	Average	94958A	94958B	94959A	94959B
Al		7.43	7.7121	7.4313	7.3321	7.2285
B			0.0201	0.0203	0.0204	0.0199
Ca		2.74	2.718	2.67055	2.8267	2.7313
Cr		0.19	0.07685	0.07425	0.07965	0.08725
Cu		0.01	0.01295	0.0099	0.01615	0.01835
Fe		23.74	23.9423	23.6995	23.88335	23.4263
K		0.24	0.245	0.2434	0.2324	0.2341
Li		0.00	<0.0082	<0.0082	<0.0083	<0.0081
Mg		1.09	1.1056	1.08815	1.0978	1.05535
Mn		3.09	3.12035	3.0741	3.11425	3.04895
Na		7.57	7.8758	7.7344	7.4382	7.2404
Ni		0.33	0.3316	0.3207	0.3608	0.3293
P		0.26	0.255	0.2414	0.2704	0.2699
Pb		0.08	0.07115	0.0677	0.08455	0.0983
Si		1.53	1.60565	1.52735	1.58395	1.41735
Ti		0.06	0.0736	0.03525	0.06125	0.05685
Zn		0.12	0.1201	0.11795	0.12055	0.1188
Zr		0.11	0.1588	0.0977	0.0759	0.1174

Table B-2 - Tank 42 Simulant Sludge Composition

	Runs 1V-4V	Runs 5V, 6V
Total Solids, wt %	16.98%	16.05%
Insoluble Solids, wt %	13.61%	12.59%
Soluble Solids, wt %	3.37%	3.46%
Density	1.1112	1.0896
Total Base pH 7, M	0.408	0.433
Total Base pH 5.5, M	0.528	0.536
Total Base pH 4.5, M	0.686	0.647
Nitrite, mg/L	8299	8049
Nitrate, mg/L	2965	2996

Table B-3 - PCCS Batching for all experiments (DWPF Basis)

Oxide Fraction	
Sludge	0.260
Frit	0.740
Volume, gallons	
Sludge	5000
Frit	2175.6
SME	4400.9
Mass Total Solids, lbs	
Total	24,151
Sludge	7,873
Frit	16,340
Mass Vitrified Solids, lbs	
Total	22,038
Sludge	5,730
Frit	16,308
Calcine Factor	
Total	91.25%
Sludge	72.78%
Frit	99.80%

The Plan for the experiments is summarized below, based on 6000 gallon DWPF batches (Table B4)

Table B-4 - Calculated Batching for all Experiments (DWPF Scale)

	1V	2V	3V	4V	5V	6V
Acid	125%	125%	125%	Max	137.5%	125%
Redox Target	None	0.2 M	0.2 M	None	0.2 M	0.2 M
Sludge Batch size, gallons	6,000	6,000	6,000	6,000	6,000	6,000
50 wt % Nitric Acid, gal	200.0	106.8	107.6	106.8	119.7	105.0
90 wt % Formic Acid, gal	171.2	212.6	219.1	400.0	229.6	210.4

Batch Preparation

The experimental setup was put together as described in Appendix A. The procedure for controlling these experiments is summarized in Appendix C. The sludge preparation for each of the experiments is summarized in Table B5. The sludge was added to the kettle (no SRAT product heel was used), then the noble metals and mercury were added to the sludge. Finally rinse water was used to ensure all of the materials were added to the kettle, including the rinse water. Weekly the antifoam solution was mixed by combining a 1 gram Dow Corning 544 antifoam with 9 grams of water to make a 1:10 antifoam solution. At the beginning of the runs, a 0.01 M nitric acid solution (pH 2) was prepared to serve as the scrubbing liquid at the beginning of the experiments.

Table B-5 - Tank 42 Flowsheet Sludge Preparation

	1V	2V	3V	4V	5V	6V
Sludge, g	2000.1	2000.0	2000.2	2000.0	2000.2	2000.0
wt % solids	16.98	16.98	16.98	16.98	16.05	16.05
Total solids, g	339.6	339.6	339.6	339.6	321.0	321.0
RuCl ₃ , g	0.1614	0.1617	1.7658	0.1587	0.1522	0.1523
Rh(NO ₃)•2H ₂ O, g	0.3304	0.3307	1.9276	0.3247	0.3124	0.3121
Pd(NO ₃)•2H ₂ O, g	0.0312	0.0307	1.7568	0.0313	0.0294	0.0295
AgNO ₃ , g	0.0827	0.0821	0.0075	0.0821	0.0778	0.0778
HgO, g	3.6301	3.6287	11.9649	3.6256	3.4313	3.4314
SeO ₂ , g	0.0059	0.0059	0.0189	0.0061	0.0055	0.0054
TeO ₂ , g	0.0467	0.0468	0.2038	0.0467	0.0442	0.0443
Rinse Water, g	300.00	300.00	300.01	300.00	300.02	300.00

SRAT Cycle

The SRAT cycle was designed to duplicate the processing in the DWPF SRAT cycle. The sludge was heated to 93°C then, the acids were added as summarized in Table B6. First nitric acid was added, then formic acid was added at a prototypic rate of 0.67 ml/min. The acidified sludge was then heated to boiling, and the volume reduced back to 1800 ml. Then kettle was placed in reflux and boiling continue for a total of twelve hours. The kettle was sampled every four hours to track nitrite destruction. After the boiling was complete, the SRAT product was cooled and sampled.

Table B-6 - SRAT Cycle Additions

	1V	2V	3V	4V	5V	6V
Nitric Acid, ml	66.34	35.45	35.71	35.45	39.70	34.83
Formic Acid, ml	57.09	70.87	73.03	160.02	76.55	70.15

SME Cycle

The SME cycle was designed to duplicate the processing in the DWPF SRAT cycle. Frit 200, water, and 90 wt% formic acid were added to the SRAT product. The slurry was then heated to boiling, and the volume reduced back to 1800 ml. A second addition of frit 200, water, and 90 wt% formic acid was added to the kettle. The total addition of frit 200, water and formic acid is summarized in Table B7. The slurry was then heated to boiling, and the volume reduced until the product concentration was approximately 45 wt% solids. After the boiling was complete, the SME product was cooled and sampled.

Table B-7 - SME Cycle Additions

	1V	2V	3V	4V	5V	6V
Frit 200, g	603.89		592.56	637.4	608.52	601.82
Water, g	1109.25		1088.45	1170.78	1117.73	1105.46
Formic Acid, g	12.3		12.08	12.99	12.42	12.24
SME Formic Acid Addition for Redox, g	17.9	No Formic Acid was added after SME Cycle				
Melter Feed, g	1973.05		1879.47	1884.56	1992.59	1870.88
Condensate, g			2114.77	2522	2217.7	2228.6

Table B-8 - Tank 42 Simulant Sludge Composition - Sodium Fusion and Microwave Preps

Elementals, wt%	Sodium Fusion				Microwave			
ADS Sample #	94958A	94958B	94959A	94959B	94958A	94958B	94959A	94959B
Al	7.7121	7.4313	7.3321	7.2285	6.7475	6.6694	6.3397	6.1324
B	0.0201	0.0203	0.0204	0.0199				
Ca	2.4721	2.3792	2.5182	2.4225	2.9639	2.9619	3.1352	3.0401
Cr	0.1389	0.1328	0.1453	0.1458	0.0148	0.0157	0.014	0.0287
Cu	0.0114	0.0086	0.0129	0.0187	0.0145	0.0112	0.0194	0.018
Fe	22.9899	22.5339	22.8758	22.3531	24.8947	24.8651	24.8909	24.4995
Li	<0.0081	<0.0082	<0.0082	<0.0080	<0.0083	<0.0081	<0.0083	<0.0082
Mg	1.0108	0.9992	1.0147	0.9703	1.2004	1.1771	1.1809	1.1404
Mn	2.8458	2.7835	2.8189	2.7637	3.3949	3.3647	3.4096	3.3342
Ni	0.3187	0.3049	0.364	0.3159	0.3445	0.3365	0.3576	0.3427
P	0.2342	0.2191	0.2498	0.2634	0.2758	0.2637	0.291	0.2764
Pb	0.0511	0.0516	0.0593	0.0926	0.0912	0.0838	0.1098	0.104
Si	1.3595	1.2715	1.4047	1.3193	1.8518	1.7832	1.7632	1.5154
Ti	0.1048	0.0339	0.0838	0.0774	0.0424	0.0366	0.0387	0.0363
Zn	0.1115	0.1084	0.1133	0.1108	0.1287	0.1275	0.1278	0.1268
Zr					0.1588	0.0977	0.0759	0.1174
K - AA	0.245	0.2434	0.2324	0.2341				
Na - AA and ICP	6.9269	6.8585	6.4413	6.8326	7.8758	7.7344	7.4382	7.2404

Stream	PHA	Sludge	Frit	SME		
Oxide Contribution (%)	0.0%	26.0%	74.0%	11995.9	kg Calc. Basis	
Density (kg/L)	1.0355	1.111	n/a	1.37		
Total Solids (%)	5.38%	16.98%	n/a	48.00%		
Calcine Factor (oz/sol)	1.00	0.73	n/a	n/a		
Hydroxide (M)	n/a	0.528	n/a	n/a		
Mercury (ppm)	n/a	5541.0	n/a	n/a		
Total Mn (wt% sol)	n/a	3.09	n/a	n/a		
Soluble Mn (ppm)	n/a	0.0	n/a	n/a		
TIC (ppm)	n/a	0.0	n/a	n/a		
Total Acid (M)	0.240	n/a	n/a	n/a		
Nitrite (ppm)	0.0	8298.8	0.0 (kg/100kg frit)		Data Entry	
NO2 Destruction	0.0%	100.0%	0.0%			
Nitrate (ppm)	1012.1	2668.1	0.0 (kg/100kg frit)			
% nitrite to nitrate conversion		35.0%				
Formate (ppm)	22808.9	0.0	1.0 (kg/100kg frit)			
Reaction formate destruction, kg		126.02				
Factor for total formate destruction		15.0%				
Total formate destruction, kg		18.90				
Oxide Mass (kg)	0.0	3118.9	8877.0	11995.9		Intermediate Results
Solids Mass (kg)	0.00	4285.46	8876.99	13162.45		
Total Mass (kg)	0.00	25238.29	n/a	27421.77		
Volume (L)	0.00	22,712.65	n/a	20015.89		
% Stoichiometry	n/a	125.0%	n/a	n/a		
Volume HNO3@50% (L)	n/a	2,294.54	n/a	n/a		
NO2 Contribution (kg)	0.000	209.446	0.000	209.446		
NO2 Remaining (kg)	0.000	0.000	0.000	0.000		
NO3 Contribution (kg)	0.000	1613.091	0.000	1613.091		
COOH Contribution (kg)	0.000	(126.02)	93.843	(32).175		
NO2 (Molar@45%)				0.000	Nitric Only Redox	
NO3 (Molar@45%)				1.103		
COOH (Molar@45%)				-0.030		
Predicted Fe(II)/DFe			0.09	≤ BDL@0.03 ≤ 0.1		
Volume HCOOH@90% (L)			2809.18	2587.31 2846		
% Stoichiometry	n/a	125.0%	n/a	n/a		
Volume HCOOH@90% (L)	n/a	1,008.33	n/a	n/a		
NO2 Contribution (kg)	0.000	209.446	0.000	209.446		
NO2 Remaining (kg)	0.000	0.000	0.000	0.000		
NO3 Contribution (kg)	0.000	140.644	0.000	140.644		
COOH Contribution (kg)	0.000	1050.146	93.843	1143.989		
NO2 (Molar@45%)				0.000	Formic Only Redox	
NO3 (Molar@45%)				0.096		
COOH (Molar@45%)				1.078		
Predicted Fe(II)/DFe			0.09	≤ 0.423 ≤ 0.1		
Volume HNO3@50% (L)			989.77	961.74		
mol% Nitric	n/a	17.8%	n/a	n/a		
Volume HCOOH@90% (L)	n/a	829.28	n/a	n/a		
Volume HNO3@50% (L)		407.45				
NO2 Contribution (kg)	0.000	209.446	0.000	209.446		
NO2 Remaining (kg)	0.000	0.000	0.000	0.000		
NO3 Contribution (kg)	0.000	402.111	0.000	402.111		
COOH Contribution (kg)	0.000	753.196	0.000	753.196		
NO2 (Molar@45%)				0.000	Acid Mixture Redox	
NO3 (Molar@45%)				0.275		
COOH (Molar@45%)				0.709		
Predicted Fe(II)/DFe			0.09	≤ 0.200 ≤ 0.1		
mol% Nitric	n/a	17.8%	n/a	n/a		
Volume HCOOH@90% (L)	n/a	829.28	n/a	n/a		
Volume HNO3@50% (L)		407.45				
NO2 Contribution (kg)	0.000	209.446	0.000	209.446		
NO2 Remaining (kg)	0.000	0.000	0.000	0.000		
NO3 Contribution (kg)	0.000	402.111	0.000	402.111		
COOH Contribution (kg)	0.000	753.196	0.000	753.196		
NO2 (Molar@45%)				0.000		
NO3 (Molar@45%)				0.275		
COOH (Molar@45%)				0.709		
Predicted Fe(II)/DFe			0.09	≤ 0.200 ≤ 0.1		

Printed: 4/2/98

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Table B-9 - - Calculated Batching for all experiments (DWPF Scale)

Appendix C Analytical Data

The following is a summary of the Product Composition and Control System (PCCS) calculation of the sludge and frit batching required for the Tank 42 experiments based on the sludge (LPPPST-0) composition and the composition of frit 200 (PFSFT-0). The predicted composition of the SRAT product (SRAT Target) and SME product (SME target) are summarized in the table below:

Table C-1

Batching Information for SRAT Batch 0

Best Analyses of SRAT and FEED Vessels

Vessel	PRBT-0	LPPPST-0	PFSFT-0	SRAT Target	SME Target
Wt%	4.26	16.98	60.00	16.98	48.00
Cal'd Wt%	2.12	12.40	60.00	12.40	43.80
Wt% Ins	.00	.00	-	.00	.00
Sp. Gr.	1.03	1.11	1.50	1.11	1.37
Units	ppm	Wt%(s)	Wt%(v)	Wt%(s)	Wt%(v)
Al	.00	7.43	.00	7.43	2.65
B	2169.14	.02	3.73	.02	2.77
Ba	.00	.00	.00	.00	.00
Ca	.00	2.74	.00	2.74	.98
Cl	.00	.00	.00	.00	.00
Cr	.19	.08	.00	.08	.03
Cs	.00	.00	.00	.00	.00
Cu	209.37	.01	.00	.01	.01
F	.00	.00	.00	.00	.00
Fe	1104.97	23.74	.00	23.74	8.46
K	7852.30	.24	.00	.24	.09
Li	.00	.00	2.32	.00	1.72
Mg	12.14	1.09	1.21	1.09	1.28
Mn	202.20	3.09	.00	3.09	1.10
Na	874.50	7.57	8.16	7.57	8.73
Nd	.00	.00	-	.00	.00
Ni	120.75	.33	.00	.33	.12
P	.00	.26	.00	.26	.09
Pb	.00	.08	-	.08	.03
Si	80.87	1.53	32.72	1.53	24.75
So4	.00	.00	-	.00	.00
Th	.00	.00	.00	.00	.00
Ti	649.86	.06	.00	.06	.02
U	.00	.00	.00	.00	.00
Zn	.00	.12	-	.12	.04
Zr	43.70	.11	.00	.11	.04

Frit 20

Actual	Target
3.7	
2.3	
1.2	
8.0	
32	

SRAT Target Information

Wt Frac	Comment				
Heel	PHA	Sludge	Frit	PNMT	
.000	.000	.260	.740	-	Liquidus Temperature PRAR Target

SRAT Volumetric Target Information

Gallons	Gallons	Gallons	Gallons	Gallons	Gallons	Comment
Heel	PHA	Sludge	SRAT	SME	PNMT	
.0	.0	5000.0	2175.6	5000.0	4400.9	.0 Liquidus Temperature PRAR Target

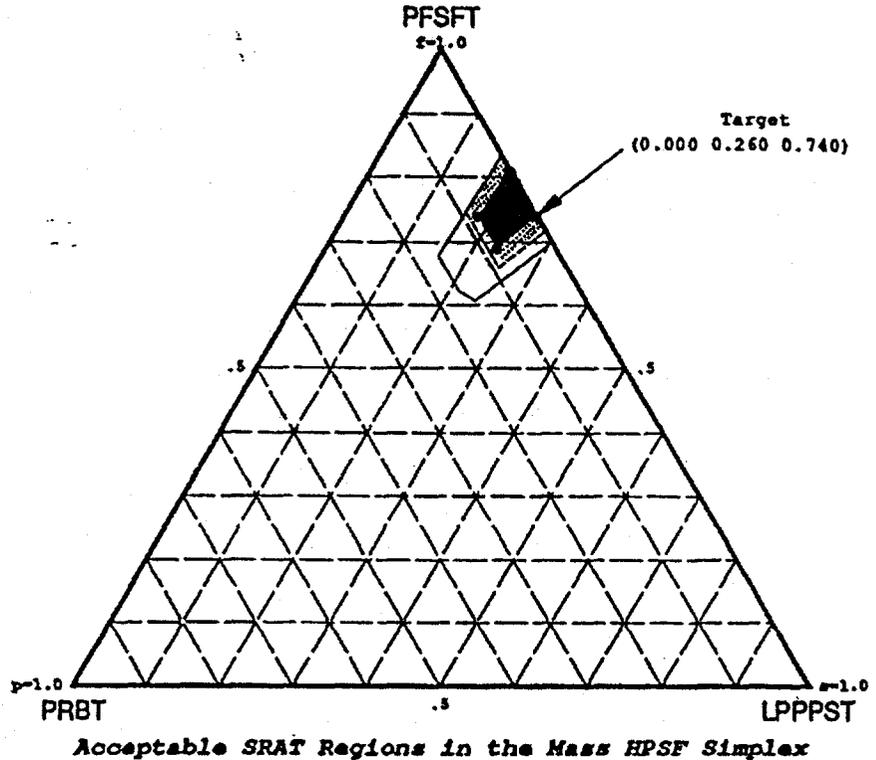
Target Properties-

NL(B)	Visc	LiqT	TiO2	Na2SO4	Cr2O3	P2O5	NaF	NaCl	Cu	Pred
0.570	73.64	997.31	0.034	0.000	0.041	0.212	0.000	0.000	0.005	-175.15

* ordered

The following ternary diagram summarizes the acceptable Tank 42 sludge and frit 200 blend necessary to make acceptable glass. Since no PHA is used, the target is 19.3 - 26.0 wt % sludge oxides and 74.0 - 80.7 wt % frit oxides. Maximum sludge waste loading was used to maximize the waste loading in the glass.

Table C-2



SRAT PRAR (mass) Coordinates (Weight Fraction):

PRBT	LPPPST	PFSFT	PRBT	LPPPST	PFSFT
0.000	0.260	0.740	0.000	0.193	0.807
0.080	0.232	0.688	0.003	0.185	0.812
0.080	0.182	0.738			

The Constraints Applicable to the Above Target Blend are:

- Liquidus Temperature (C) < 1050 at a % confidence of 95.0
- High Viscosity (poise) < 100 at a % confidence of 95.0
- Low Viscosity (poise) > 20 at a % confidence of 95.0
- ASTM C1285-94 B (NL(B) g/L) < 7.07 at a % confidence of 95.0
- TiO2 (Wt%(v)) < 1.0 at a % confidence of 95.0
- Cr2O3 (Wt%(v)) < 0.3 at a % confidence of 95.0
- P2O5 (Wt%(v)) < 2.24 at a % confidence of 95.0
- Cu (Wt%(v)) < 0.5 at a % confidence of 95.0
- Predictability (Wt%(v)) < -163.6517 at a % confidence of 95.0

The following data summarizes the results of the redox

Table C- 3 - SRTC Mobile Laboratory Analytical Results

Date: 10/16/97

Customer: Dan Lambert

Sample ID	Weight(mg)	Dil.(uls)	Fe2+ abs	FeTotal abs	Fe2+/Fe3+	Fe2+/Fe total
EASTD	26.1	300	0.129	0.691	0.229	0.186
T2V1A	35.4	500	0.021	1.666	0.013	0.012
T2V1B	40.0	500	0.021	1.836	0.012	0.011
T2V2A	37.0	500	0.028	1.669	0.017	0.016
T2V2B	31.7	500	0.029	1.415	0.021	0.021
T2V3A	30.5	500	0.021	1.201	0.018	0.018
T2V3B	31.9	500	0.024	1.305	0.019	0.019
Average						0.016
T3V1.1A	31.0	500	0.011	1.505	0.007	0.007
T3V1.1B	45.1	500	0.013	2.042	0.006	0.006
T3V1.2A	37.8	500	0.008	1.710	0.005	0.005
T3V1.2B	37.3	500	0.008	1.651	0.005	0.005
T3V2.1A	30.4	500	0.047	1.414	0.034	0.033
T3V2.1B	34.6	500	0.054	1.662	0.033	0.032
T3V2.2A	31.9	500	0.064	1.343	0.050	0.048
T3V2.2B	42.5	500	0.086	2.021	0.045	0.043
T3V3.1A	41.1	500	0.006	1.887	0.003	0.003
T3V3.1B	33.5	500	0.006	1.722	0.004	0.004
T3V3.2A	35.0	500	0.010	1.568	0.007	0.007
T3V3.2B	42.2	500	0.010	1.983	0.005	0.005
Average						0.017
EASTD	28.0	300	0.114	0.602	0.233	0.189
EAstd	26.0	300	0.101	0.548	0.225	0.183

* These were analyzed with a standard that had a higher than expected ratio. This was due to bad buffer pH. You may want to bias correct to get the correct readings on the samples that follow. Fortunately there was enough sample on one of the set to reanalyze with corrected buffer (noted with a "x").

Table C-4 - Fe²⁺/Fe Total Redox Analysis

SRTC Mobile Laboratory

725-0932

4/17/98

8:08 AM

122

DL 12/16

ID	Dilution	WEIGHT(mg)	Fe2+	FeTotal	Fe2+/Fe3+	Fe2+/Fe total
Eastd	300	27.9	0.110	0.601	0.224	0.183
T1V1-1A	500	32.1	0.010	1.490	0.007	0.007
T1V1-1B	500	32.1	0.004	1.468	0.003	0.003
T1V1-2A	500	34.5	0.007	1.544	0.005	0.005
T1V1-2B	500	32.8	0.006	1.556	0.004	0.004
T1V2-1A	500	37.1	0.005	1.646	0.003	0.003
T1V2-1B	500	34.0	0.009	1.516	0.006	0.006
T1V2-2A	500	31.6	0.008	1.410	0.006	0.006
T1V2-2B	500	34.9	0.009	1.539	0.006	0.006
T1V3-1A	500	36.9	0.008	1.722	0.005	0.005
T1V3-1B	500	34.7	0.009	2.900	0.003	0.003
T1V3-2A	500	34.8	0.016	1.623	0.010	0.010
T1V3-2B	500	38.0	0.017	1.680	0.010	0.010
T4V1-1A	100	37.7	0.132	0.348	0.611	0.379
T4V1-1B	100	35.6	0.134	0.334	0.670	0.401
T4V1-2A	100	33.2	0.112	0.284	0.651	0.394
T4V1-2B	100	34.6	0.127	0.335	0.611	0.379
T4V2-1A	100	30.5	0.116	0.280	0.707	0.414
T4V2-1B	100	31.0	0.114	0.286	0.663	0.399
T4V2-2A	100	31.7	0.106	0.288	0.582	0.368
T4V2-2B	100	33.2	0.107	0.307	0.535	0.349
T4V3-1A	100	35.6	0.099	0.339	0.413	0.292
T4V3-1B	100	34.0	0.095	0.319	0.424	0.298
T4V3-2A	100	38.1	0.112	0.337	0.498	0.332

The following data supports the reported PCT results.

Table C- 5

Normalized Glass Compositions

Oxide	T1V	T2V	T3V	T4V
Al ₂ O ₃	6.13%	5.90%	6.11%	5.65%
B ₂ O ₃	8.03%	8.40%	8.19%	8.26%
CaO	1.58%	1.58%	1.54%	1.54%
CuO	0.01%	0.01%	0.01%	0.01%
Fe ₂ O ₃	13.06%	13.25%	12.98%	12.21%
K ₂ O	0.00%	0.00%	0.00%	0.00%
Li ₂ O	3.20%	3.26%	3.22%	3.35%
MgO	2.09%	2.03%	2.09%	2.08%
MnO	1.48%	1.58%	1.58%	1.46%
Na ₂ O	11.24%	10.71%	11.30%	11.44%
NiO	0.22%	0.24%	0.23%	0.22%
P ₂ O ₅	0.28%	0.29%	0.33%	0.26%
PbO	0.06%	0.05%	0.06%	0.05%
SiO ₂	52.48%	52.55%	52.23%	53.32%
TiO ₂	0.04%	0.05%	0.04%	0.04%
ZnO	0.06%	0.06%	0.06%	0.06%
ZrO ₂	0.03%	0.03%	0.03%	0.03%
	100.00%	100.00%	100.00%	100.00%

Table C- 6 Raw Analysis of PCT Leachates and Solution Standards

Elemental Analysis (µg/ml)					
sample	Na	Li	K	B	Si
Blank-1	<0.530	<0.020	<0.600	<0.180	<0.180
Blank-2	<0.530	<0.020	<0.600	<0.180	<0.180
Arm1	10.2	9.81	<0.600	13	43.1
Arm2	9048	9.34	<0.600	11.7	41.3
Arm3	9071	9.75	<0.600	12.8	43
Std1	■	9.41	9.82	19.7	50.2
chkstd	4.92	4.92	5.04	5.28	5.13
ea1	382	98.8	1.23	360	560
ea2	334	93.2	0.992	305	538
ea3	320	89.9	0.975	285	524
t1v-1	10.4	5.82	<0.600	8.69	50.2
t1v-2	10.1	5.57	<0.600	8.41	47.2
Std2	■	9.74	9.72	19.6	51.2
chkstd	3.17	4.92	5.06	5.29	4.92
t1v-3	10.4	5.59	<0.600	8.46	48.9
t2v-1	11.5	5.95	<0.600	10	55.6
t2v-2	11.9	6.07	<0.600	9.77	57
t2v-3	11.7	6.11	<0.600	9.8	56.2
t3v-1	11	5.68	<0.600	9.12	50.8
Std3	■	9.64	9.88	20.5	50.7
chkstd	5	5	5.1	5.39	5.07
t3v-2	11.2	5.9	<0.600	9.27	50.5
t3v-3	11.3	6.05	<0.600	9.91	51.2
t4v-1	12.9	6.48	<0.600	10.5	56.6
t4v-2	13.2	6.47	<0.600	10.3	56.5
t4v-3	13.4	6.48	<0.600	10.9	60.4
Std4	■	9.38	9.87	20.1	49.2
chkstd	4.93	4.77	5.07	5.23	5.06

Where:

chkstd is the Mobile Lab standard to check instrument

Std1, Std2, Std3, and Std4 are multi-element solution standards submitted to verify analysis

Multi-Element ICP Standard Lot # 691120 ± 0.5%

Al 4 µg/ml K 10 µg/ml

B 20 µg/ml Si 50 µg/ml

Fe 4 µg/ml Na 81 µg/ml

Li 10 µg/ml

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Sludge

Retention: Permanent

September 2, 1998

**M. N. BROSEE, MANAGER
DEFENSE WASTE PROCESSING FACILITY**

**TANK 42 SLUDGE-ONLY PROCESS DEVELOPMENT FOR THE DEFENSE WASTE
PROCESSING FACILITY (DWPF) (U)**

In response to the Technical Task Request #HLW/DWPF/TTR-970134, a series of laboratory scale process simulations were performed to ensure the current sludge-only flowsheet is acceptable for processing sludge batch 1B in the DWPF Chemical Processing Cell (CPC). Sludge batch 1B is the next sludge macrobatch that will be processed in DWPF, once the current Tank 51 sludge is exhausted (expected August 1998).

One processing change is that 10% more acid is required than is currently used to ensure the destruction of nitrite in a twelve-hour aqueous boil time in the SRAT cycle. The generation of hydrogen and nitrous oxide is well within the current DWPF safety envelop.

The report is being reissued to clarify the Product Consistency Test (PCT) results.

If you have additional questions regarding the attached report, please contact D. P. Lambert, 7-7680.

A handwritten signature in cursive script, reading 'E. W. Holtzscheiter'.

E. W. Holtzscheiter, Manager
SRTC - Immobilization Technology Section