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White Paper to Justify the Use of Strip Effluent in the Defense Waste Processing Facility During Slurry Mix Evaporator Processing

D. P. Lambert

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A special thanks to Ian Wright and Jeremiah Ledbetter for collecting and documenting the DWPF processing data. The Excel spreadsheet was very helpful and saved me a lot of time.

EXECUTIVE SUMMARY

At the request of Savannah River Remediation (SRR), a white paper was written to assess whether it would be acceptable to process Strip Effluent (SE) in both the Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) in the Defense Waste Processing Facility (DWPF) for the nitric-glycolic acid flowsheet. Savannah River National Laboratory (SRNL) experimental data and DWPF Sludge Batch 9 (SB9) process data was reviewed, looking at batches which included Precipitate Reactor Feed Tank (PRFT) and/or Strip Effluent Feed Tank (SEFT) feeds that led to long processing times. These batches were reviewed looking for processing problems such as melter feed trips, foamovers, heating rod or steam coil fouling, missed Reduction/Oxidation (REDOX) targets, and other process anomalies.

The nitric-glycolic acid flowsheet was very effective during SRNL testing in processing sludge, PRFT and SEFT feeds without the processing problems noted above. One of the advantages of this flowsheet is the stable pH of the SRAT and SME products, indicative of the anion chemistry being essentially complete by the end of the SRAT dewater cycle. In contrast, the nitric-formic acid flowsheet processing leads to a pH increase of up to 3 pH units during just the SME cycle in extended processing in DWPF. The longest of the SRNL experiments had a boiling time after acid addition of 92 hours.

A review of the DWPF batch history from SB9 identified three batches with extremely long processing times. Batches 784, 785 and 786 had up to 300 hours at boiling after acid addition. This led to large pH changes in the SME cycle, with SME product pH above 9 and significant deviations from the REDOX target. No significant foaming, coil fouling, or melter feed trips were noted. This processing was not used to extend maximum boiling time due to issues in these batches with pH changes and REDOX. The use of the nitric-glycolic flowsheet, at a pH of 6 or above, where issues with rheology and coil fouling were rarely noted in simulant runs, likely would have been successful in meeting all processing targets even during these extremely long processing times.

Based on the review, the following is recommended for the nitric-glycolic acid flowsheet:

1. Strip Effluent can be added post acid addition during either the SRAT or SME cycle during processing using the nitric-glycolic acid flowsheet. If boiling times beyond 92 hours are necessary, the facility should pay close attention to pH changes and REDOX which may lead to foaming, coil fouling, and/or melter trips. Further testing at extended boiling times could be performed to alleviate this potential concern. If the facility desires the flexibility to add SEFT during caustic boiling, additional laboratory testing is needed.
2. The volume of SEFT that can be added during a 92 hour SRAT and SME cycle depends on steam flowrate and the volume of canister blast water that requires evaporation. Assuming 5,000 gallons of canister blast water, the maximum volume of SEFT that can be processed is 20,000 gallons at typical steam flowrate (3,000 lb/hr SRAT, 2,400 lb/hr SME) and 44,000 gallons at design basis steam flowrate (5,000 lb/hr SRAT and SME).
3. In order to maximize the volume of SE and monosodium titanate/Sludge Solids (MST/SS) processed in a batch as well as reduce SRAT/SME cycle times, it is recommended that DWPF explore opportunities to restore design basis steam flow rates. However, it is recognized that realization of this opportunity would require facility modifications, purge reductions, and CHA changes.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CHA	Consolidated Hazard Analysis
CPC	Chemical Processing Cell
DSFE	DWPF/Saltstone Facility Engineering
DWPF	Defense Waste Processing Facility
KMA	Koopman Minimum Acid
MCU	Modular Caustic Side Solvent Extraction Unit
MFT	Melter Feed Tank
MST	monosodium titanate
MWWT	Mercury Water Wash Tank
PI	Process Information
PRFT	Precipitate Reactor Feed Tank
REDOX	Reduction/Oxidation
SAC	Specific Administrative Controls
SB	Sludge Batch
SE	Strip Effluent
SEFT	Strip Effluent Feed Tank
SME	Slurry Mix Evaporator
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SS	Sludge Solids
SWPF	Salt Waste Processing Facility
TTQAP	Task Technical and Quality Assurance Plan
TSR	Technical Safety Requirement
TTR	Task Technical Request

1.0 Introduction

DWPF processes two waste streams from the Salt Waste Processing Facility (SWPF): SE and MST/ SS. Both SE and MST/SS will be added in the SRAT at DWPF. In the current process, after the addition of SE to the SRAT, the SRAT product is transferred to the SME for further processing and to reduce the volume of the SME product. The volumes of SE and MST/SS that will be generated by SWPF are expected to be significantly larger than the streams previously processed by the DWPF. So, there is a desire to increase the operational flexibility of the DWPF process by allowing SE to be added to either the SRAT or SME to keep pace with SWPF processing rates.

DWPF/Saltstone Facility Engineering (DSFE) issued a Technical Task Request[1] (TTR) to SRNL to perform flowsheet studies for Sludge Batch 9 (SB9). In particular, the TTR requests SRNL to develop a white paper or technical report which discusses the following.

- The feasibility of adding SE to the SME (in place of or in addition to the SRAT).
    - This should include discussion of restrictions, if any, on where SE may be added in the SME cycle.
- The maximum volume of SE that should be added to a given SRAT/SME batch.

A Task Technical and Quality Assurance Plan (TTQAP) was written to cover this task.[2]

The addition of SE to the SME has been previously studied for the nitric-formic acid flowsheet.[3] This study found that due to the long processing time that results from processing increased volumes of SE may lead to higher hydrogen generation, higher ammonia production, higher formate destruction, lower REDOX ratio, higher potential for foaming and coil fouling, and higher yield stress and consistency. As a result of this testing, the addition of SE to SME was not implemented in DWPF.

For the nitric-glycolic acid flowsheet planned for implementation in the near future in DWPF, the replacement of formic acid with glycolic acid essentially eliminates hydrogen and ammonia generation and stabilizes the pH, which reduces the potential for foaming, coil fouling, REDOX and rheology changes. As a result, adding SE to the SME will be reconsidered as part of this task for the nitric-glycolic acid flowsheet. A number of nitric-glycolic acid flowsheet reports were reviewed for adding SE to the SME including the following reports (Table 1-1).

Table 1-1. List of Primary Reports Reviewed

Title	Report
Impact of Salt Waste Processing Facility Streams on the Nitric-Glycolic Flowsheet in the Chemical Processing Cell	SRNL-STI-2016-00665[4]
Sludge Batch 9 Simulant Runs Using the Nitric-Glycolic Acid Flowsheet	SRNL-STI-2016-00319[5]
Antifoam Development for Eliminating Flammability Hazards and Decreasing Cycle Time in the Defense Waste Processing Facility	SRNL-STI-2019-00677[6]
FY13 Glycolic-Nitric Acid Flowsheet Demonstrations of the DWPF Chemical Process Cell with Simulants	SRNL-STI-2013-00343[7]
Nitric-Glycolic Flowsheet Testing for Maximum Hydrogen Generation Rate	SRNL-STI-2015-00130[8]

One important aspect of this study is to review pertinent DWPF Process Information (PI) data for relevance to long processing times at elevated temperature such as long processing times in the SRAT due to large volumes of SEFT or long processing times in the SME resulting water from large numbers decontaminated canisters. Important data include hydrogen generation, heat transfer coefficients, foaming indications such as SME pH increases or foam detection alarms, excessive melter feed trips, and other indications related to hydrogen generation, foaming, fouling, and other process upsets due to long processing times.

No new testing was performed to support this study. Instead, prior reports were reviewed for their relevance to this study. In addition, DWPF data was reviewed to find SRAT and SME batches that had long processing times that might be pertinent to this study. This review focused on process chemistry.

1.1 Quality Assurance

The following data are considered Safety Class and will be used to evaluate DWPF Technical Safety Requirement (TSR) Specific Administrative Controls (SACs) 5.8.2.11 and 5.8.2.38:
Hydrogen Generation: Hydrogen generation rates for the SRAT and SME on a 6000-gallon basis.

The report review was completed to comply with the requirements for performing reviews of technical reports. The extent of the review is established in manual E7 2.60.[9] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[10]

2.0 Background

Extensive testing was completed in developing the nitric-glycolic acid flowsheet. To date 102 SRAT or SRAT and SME demonstrations with simulants have been completed and two radioactive waste tests have been completed. However, little of this testing was completed with both SE and MST/SS. Table 2-1 summarizes the nine experiments with SE and MST/SS and the four experiments with SE. No radioactive waste testing has been completed with SE or MST/SS.

Table 2-1. SRNL Simulant Testing with Large SEFT Additions

Experiments	DWPF Scaled Volume of SE, gallons	DWPF Scaled Volume of MST/SS, gallons	DWPF Scaled Can Blast Volume, gallons	Report
GN60-GN64	9,000	1,800	No SME Cycle	SRNL-STI-2013-00343[7]
GN80-GN83	18,000	0	6 000	SRNL-STI-2015-00130[8]
SB9NG61	00	00	6,000	SRNL-STI-2016-00319[5]
SB9NG62	12,000	1,000	0	
NGAY-17112	15,000	3,000	6,000	SRNL-STI-2019-00677[6]
NGA MD20				

The important processing parameter influencing DWPF SRAT and SME processing is time at elevated temperatures. This means that in processing SE, the extent of the reactions will be the same whether processing is completed at 2500 or 5000 lb/hr steam as the liquid temperature will be the same and the reaction rates are controlled by the reaction temperature. As a result, the time at elevated temperature and not the volume of SE and MST/SS is the important factor to consider.

It should be noted that most of these experiments were completed at design basis boilup of 5,000 lb/hr scaled steam. SRNL experiments GN83 and SB9NG61 were processed at a scaled boilup rate of 2,500 lb/hr.

DWPF typically processes at peak steam flow of 3,000 lb/hr in the SRAT and 2,500 in the SME. A graph of the steam flowrate for DWPF SRAT batch 794 is summarized in Figure 2-1.

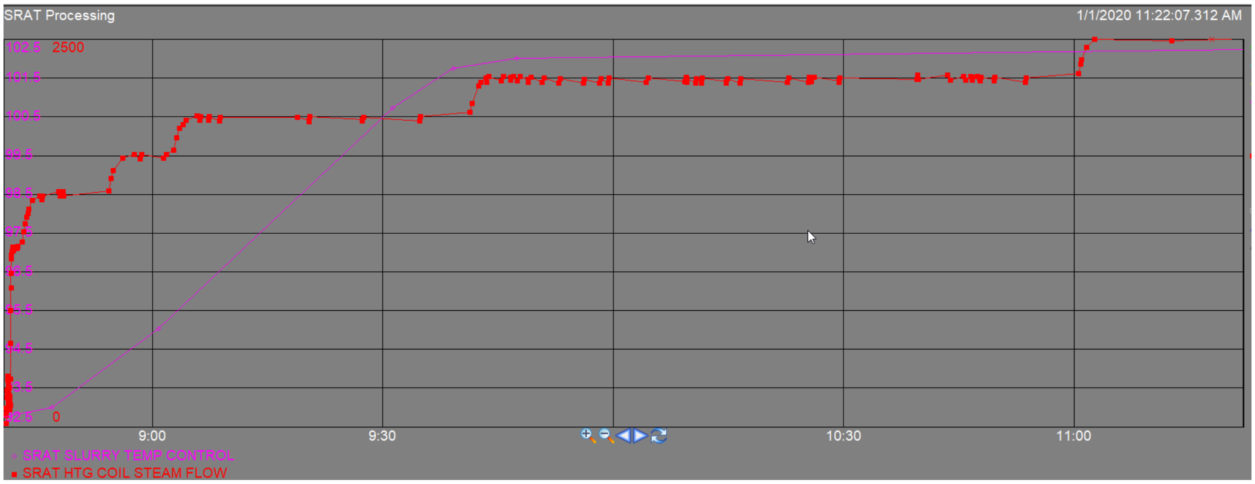

Figure 2-1. DWPF SRAT Steam Flowrate During Ramp-up to Maximum Steam Flow (2500 lb/hr)

Note that a full boil isn't reached until the SRAT temperature levels out, which was more than an hour after heatup to boiling was initiated. The DWPF practice of slowly ramping up to boiling, lowers the average boilup rate to well below the peak. The combination of lower steam flowrate than design basis and slow ramp-up of steam flow combine to significantly increase the time at temperature, essentially doubling it in typical processing.

One other note is that DWPF doesn't process each batch the same way. For instance, if there is an operational issue with the canister decontamination equipment, no can blast water will be added to one SME cycle while as many as 15 can blasts may be added to another SME batch. The evaporation of this water requires additional time at temperature, which factors into the total time at temperature for the SRAT and SME cycle. As a result, the volume of can blast water used in each experiment is summarized in Table 2-1.

Even though DWPF has not processed with nitric-glycolic acid flowsheet, there have been nitric-formic flowsheet batches with extended processing times due to large additions of Strip Effluent, large volumes of can blast water to evaporate, or long idle times at boiling or near boiling. These batches are evaluated in Section 3 to see whether any processing issues such foamovers, excessive melter feed pump trips, etc. are noted. If DWPF processed these batches with extremely long times at temperature, it is expected that processing for long times at temperature using the nitric-glycolic acid flowsheet would also be successful.

2.1 Review of Nitric-Glycolic Acid Flowsheet Experimental Data

Experimental data collected in development of the nitric-glycolic acid flowsheet was reviewed. Experiments with long processing times were usually due to large processing volumes of PRFT, SEFT, and decontamination canister water or lower boilup rates. The tests discussed in this section had longer time at boiling than typical processing tests.

2.1.1 GN60-GN64[7]

A series of five back to back SRAT-only experiments were performed during March 2013 to determine the influence of process heels and back to back operation on SRAT processing. The first operation was completed without a heel and the last four had a heel (a scaled 1,500 gallon heel was left from the previous

SRAT product). Experiments GN61-GN64 are the only nitric-glycolic acid tests that utilized a heel. These tests also included both 1,800 scaled gallons of PRFT and 9,000 scaled gallons of 0.01 M boric acid SEFT simulants. The PRFT simulant contained about 7 wt % total solids, containing monosodium titanate and sludge solids. The SEFT simulant had no added Next Generation Solvent simulating entrainment. The volumes of both PRFT and SEFT simulant were considered bounding for Actinide Removal Process (ARP) and Modular Caustic Side Solvent Extraction Unit (MCU) processing. Total time at boiling per test in the SRAT cycle was 20 hours.

2.1.2 GN80-GN83[8]

A series of four SRAT and SME tests were performed during January and February of 2015 to perform DWPF Chemical Processing Cell (CPC) testing at conditions that would bound the catalytic hydrogen production for the nitric-glycolic flowsheet. The simulant tests utilized various boil-up rate schemes (using SRAT and/or SME steam flows of 2,500 or 5,000 lb/hr). 18,000 scaled gallons of 0.015M boric acid SEFT and 6,000 gallons of canister decontamination water simulants were added but no PRFT simulant was added. The tests were designed to be conducive to catalytic hydrogen generation (i.e., using high concentration of noble metals, high stoichiometric excess acid) with the objective of producing a maximum process-representative hydrogen generation during the CPC processing with the glycolic acid flowsheet. Note that GN83, with 2,500 lb/hr steam flow in both the SRAT and SME cycle had the longest total time at boiling of about 92 hours.

2.1.3 SB9NG61 and SB9NG62[5]

During the development of the nitric-glycolic acid flowsheet for Sludge Batch 9 (SB9), one SRAT and SME simulation, SB9NG61, was completed with no PRFT or SEFT simulant, and 6,000 gallons of canister decontamination water simulant. One SRAT and SME simulation, SB9NG62, was completed with 1,000 scaled gallons of PRFT simulant, 12,000 scaled gallons of SEFT simulant and no canister decontamination water simulant. The SB9NG62 SRAT and SME cycle had a boilup rate of 5,000 lb/hr. Note that SB9NG61, with 2,500 lb/hr steam flow in both the SRAT and SME cycle had the longest total time at boiling of about 80 hours compared to 42 hours for SB9NG62.

2.1.4 NGA Y-17112 and NGA MD20[6]

During development of a new antifoam for DWPF, two identical SRAT and SME simulations were completed with the only variable being the antifoam used (Momentive™ Y-17112 or Evonik Surfynol® MD20). The SRAT and SME simulations were completed with 3,000 scaled gallons of PRFT simulant (MST only, no added sludge solids), 15,000 scaled gallons of 0.0015 M nitric acid SEFT simulant and 6,000 gallons of canister decontamination water simulant. The simulations, with 5,000 lb/hr steam flow in both the SRAT and SME cycle had a total time at boiling of about 54 hours.

2.2 Review of Nitric-Formic Acid Flowsheet Processing Data in DWPF

The data review for this report was limited to SB9 processing. SB9 processing in DWPF began with Batch 774 on 11/7/16 and continued until batch 795, when DWPF entered an outage to prepare for SWPF startup. Ian Wright and Jeremiah Ledbetter compiled data from PI along with analytical data in a Microsoft Excel Workbook and documented the data in a memo.[11] There was considerable variability in processing in the SB9 batches. The timeline for these operations is summarized in Table 2-2. Some of this variability is summarized in Table 2-3. Twenty SB9 batches have been completed. Three of the batches contained only sludge, one contained sludge and PRFT, eleven included sludge and SEFT and five consisted of sludge, PRFT and SEFT. The amount of time at boiling and the steam used during testing (post acid addition) are included in Table 2-2 because they lead to long times at boiling during the batch, as does the number of canisters blasted each batch. Batch 784, a batch with ~3,800 gallons of PRFT, ~5,300 gallons of SEFT and fifteen can blasts, had the longest time at boiling (almost 300 hours, more than twelve days). Two other batches (785 and 786) both had >100 hours of boiling due to high volume of SEFT addition and more than five canisters blasted. The processing from these operations will be discussed in more detail in Section 3.3.

Table 2-2. Timeline for DWPF SB9 SRAT and SME Cycles

	SRAT			SME	
Batch	Start Time	End Time	Time at Acid Addition	Start Time	End Time
774	11/7/16 1:30 AM	11/11/16 5:50 PM	11/9/16 3:30 PM	11/11/16 12:45 AM	11/17/16 5:10 AM
775	11/11/16 5:55 PM	11/19/16 12:15 AM	11/17/16 2:10 AM	11/17/16 5:15 AM	11/23/16 2:20 PM
776	11/19/16 12:20 AM	11/24/16 10:50 AM	11/22/16 5:20 AM	11/23/16 2:25 PM	11/30/16 5:40 AM
777	11/24/16 10:55 AM	11/30/16 11:10 PM	11/26/16 7:30 AM	11/30/16 5:45 AM	12/6/16 9:20 PM
778	11/30/16 11:15 PM	12/19/16 12:35 PM	12/3/16 1:45 AM	12/6/16 9:25 PM	12/27/16 12:45 AM
779	12/19/16 12:40 PM	12/27/16 1:55 AM	12/21/16 4:25 AM	12/27/16 12:50 AM	1/2/17 1:45 AM
780	12/27/16 2:00 AM	1/3/17 7:15 AM	12/31/16 1:55 PM	1/2/17 1:50 AM	1/8/17 5:25 AM
781	1/3/17 7:20 AM	1/8/17 12:35 PM	1/5/17 3:40 PM	1/8/17 5:30 AM	1/14/17 10:00 AM
782	1/8/17 12:40 PM	12/4/17 7:45 AM	11/5/17 4:45 PM	1/14/17 10:05 AM	12/8/17 5:55 PM
783	12/4/17 7:50 AM	5/24/18 5:50 PM	5/17/18 1:20 AM	12/8/17 6:00 PM	5/30/18 7:40 PM
784	5/24/18 5:55 PM	7/4/18 11:00 AM	7/2/18 3:40 AM	5/30/18 7:45 PM	8/27/18 5:35 PM
785	7/4/18 11:05 AM	9/7/18 9:10 PM	8/3/18 6:05 AM	8/27/18 5:40 PM	11/13/18 5:05 PM
786	9/7/18 9:15 PM	12/17/18 12:55 PM	10/4/18 12:40 AM	11/13/18 5:10 PM	1/11/19 5:05 PM
787	12/17/18 1:00 PM	2/18/19 3:00 AM	1/22/19 3:20 PM	1/11/19 5:10 PM	3/14/19 3:00 AM
788	2/18/19 3:05 AM	3/19/19 3:20 PM	2/20/19 5:50 AM	3/14/19 3:05 AM	4/2/19 12:25 PM
789	3/19/19 3:25 PM	4/5/19 5:45 AM	3/23/19 9:55 PM	4/2/19 12:30 PM	5/17/19 5:10 AM
790	4/5/19 5:50 AM	5/17/19 10:30 AM	4/14/19 8:15 AM	5/17/19 5:15 AM	7/2/19 10:15 AM
791	5/17/19 10:35 AM	6/9/19 12:00 AM	5/21/19 5:10 PM	6/8/19 10:05 PM	6/28/19 1:40 AM
792	6/9/19 12:05 AM	7/3/19 1:05 AM	6/28/19 7:30 PM	6/28/19 1:45 AM	7/15/19 1:00 PM
793	7/3/19 1:10 AM	7/15/19 3:20 PM	7/10/19 4:05 PM	7/15/19 1:05 PM	7/15/19 3:20 PM

Table 2-3. DWPF SB9 Processing Summary

	SRAT				SME			SRAT+SME	
Batch	PRFT Added [gal]	Time > 99°C After Acid [hr]	SEFT Added [gal]	Steam Added After Acid [lbs]	Time >99°C [hr]	Cans Blasted	Steam added [lbs]	Time >99°C [hr]	Steam added [lbs]
774	-	26.9	-	78,772	35.8	4	114,304	62.7	193,076
775	-	22.6	-	75,124	43.2	5	128,909	65.8	204,033
776	-	26.8	4,343	75,975	50.2	5	142,451	76.9	218,427
777	-	30.9	-	80,184	35.3	3	111,574	66.3	191,757
778	-	26.3	4,246	75,086	18.0	2	72,839	44.3	147,925
779	-	42.9	8,663	119,667	39.8	4	135,360	82.7	255,027
780	-	26.6	3,939	75,053	58.4	6	165,734	85.0	240,787
781	-	27.3	2,885	75,066	59.8	6	158,218	87.1	233,285
782	1,551	37.2	-	75,083	29.6	6	147,870	66.8	222,953
783	-	42.6	6,622	121,168	15.3	1	75,438	57.9	196,605
784	3,803	29.4	5,299	82,896	266.3	15	476,679	295.8	559,576
785	841	63.2	7,410	161,970	112.8	7	284,855	175.9	446,826
786	-	73.2	8,873	202,462	58.2	6	201,241	131.3	403,702
787	4,100	40.8	7,499	113,038	19.1	0	74,760	59.9	187,798
788	-	57.5	7,800	164,532	22.8	1	96,095	80.3	260,627
789	-	47.7	8,415	135,375	27.0	6	232,745	74.7	368,120
790	-	42.9	7,900	119,326	11.2	5	147,705	54.1	267,031
791	-	49.9	7,024	129,834	27.5	8	237,451	77.4	367,285
792	3,745	29.3	5,168	88,308	12.3	4	200,947	41.5	289,255
793	4,410	25.8	261	85,468	48.0	9	219,306	73.8	304,774

Due to the startup of SWPF, future processing in DWPF will include the addition of sludge, PRFT and SEFT during SRAT processing and the addition of canister blast water and the process frit/water slurry during SME processing. Since SWPF volumes of PRFT and SEFT will be higher than was typical during ARP/MCU processing, the processing time for each SRAT batch will be longer. It should be noted that the processing time is also longer as the result of the facility throttling the steam flow to control foaming during processing with typical SRAT steam flowrate of 3,000 lb/hr and the typical SME steam flowrate of 2,400 lb/hr. To decrease the processing time in the SRAT and SME, it is desirable to increase the steam flowrate to design basis or 5,000 lb/hr. An improved antifoam[6] or less foaming due to the nitric-glycolic acid flowsheet may allow the facility to increase the SRAT and SME steam flowrate and decrease processing time.

An estimate was made for the boiling time needed to process the various streams added during SRAT and SME processing. Four estimates were completed, assuming typical facility steam flow rate and design basis flowrate combined with using typical process feeds and 3x process feeds. The results of these calculations are summarized in Table 2-4. If design basis steam flow can be achieved in the SRAT and SME, the typical boiling time post acid addition should be <35 hours and would be <65 hours even if the SEFT, PRFT and Decon canister water added is 3x the typical expected for SWPF processing. If typical facility nitric-formic acid flowsheet steam flows are used in the SRAT and SME, the typical boiling time post acid addition should be <80 hours and would be <150 hours even if the SEFT, PRFT and Decon canister water added is 3x the typical expected for SWPF processing.

Table 2-4. Calculated Post Acid Boiling Times for DWPF Processing Scenarios

		Facility Norms			Design Basis		
Step	Addition Volume, gal	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	NA	3,000	8.3	NA	5,000	5.0
SEFT Addition	9,000	6.0	3,000	25.0	10	5,000	15.0
Hg Stripping	NA	NA	3,000	3.3	NA	5,000	2.0
Decon Canisters	5,000	NA	2,400	17.4	NA	5,000	8.3
Process Frit	2,700	NA	2,400	9.4	NA	5,000	4.5
Total	**22,600**	**NA**	**NA**	**63.5**	**NA**	**NA**	**34.9**
		Worst Case Facility Norms			Worst Case Design Basis		
Step	Addition Volume, gal	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	NA	3,000	8.3	NA	5,000	5.0
SEFT Addition	27,000	6.0	3,000	75.1	10	5,000	45.1
Hg Stripping		NA	3,000	0.0	NA	5,000	0.0
Decon Canisters	15,000	NA	2,400	52.2	NA	5,000	25.0
Process Frit	2,700	NA	2,400	9.4	NA	5,000	4.5
Total	**55,600**	**NA**	**NA**	**145.0**	**NA**	**NA**	**79.6**

3.0 Discussion

The nitric-glycolic acid flowsheet has been recommended to DWPF to eliminate the production of hydrogen due to catalytic decomposition of formic acid. One benefit of the nitric-glycolic acid flowsheet is that the pH remains essentially constant after the chemical reactions are complete, usually during the SRAT dewater. In similar operations with the nitric-formic acid flowsheet, not only is significant hydrogen generated but the pH may increase from a pH of about 6 to a pH of 10 or 11 by the time SME processing is complete. Increasing the pH above 7 leads to rheologically thicker slurries and the release of ammonia to the offgas.[5,12]

DWPF would like the flexibility to add Strip Effluent during either the SRAT or SME cycle. This would give DWPF the capacity to handle more Strip Effluent in a batch or wait and add the SEFT to the SME if that flexibility is helpful to processing the batch.

The SEFT solution, is a very dilute nitric or boric acid solution, so it does not significantly influence the chemistry in the SRAT (and isn't expected to influence the SME chemistry either). The SEFT solution may increase the SRAT boiling time (or SME boiling time), especially when processing sludge with lower mercury concentrations (if mercury is high, the SRAT batch duration is determined by the time need to steam strip enough mercury to reach the SRAT product mercury target).

The important factor for the chemical reactions that occur in the SRAT process is the time at elevated temperature (usually boiling), since the boiling temperatures are essentially constant in the SRAT and SME. The rate of the chemical reaction doesn't depend on the boilup rate as the boiling temperature is the same with 2,500 or 5,000 lb/hr steam flow. However, since at 2,500 lb/hr steam, the batch time is at least twice as long, there is more time for the chemical reactions to go further towards completion. Since no nitric-glycolic acid flowsheet testing has been completed adding SEFT to the SME, it is conservatively assumed that there is little expected chemistry difference in the SRAT or the SME other than the reactions may be further along in the SME since the slurry has had more time at temperature. As a result, the time at temperature post acid addition should be limited to the maximum duration that has been tested experimentally, until longer processing in DWPF or longer testing has been completed without issues. This also means that DWPF should attempt to increase steam flow to minimize processing time as twice as much SEFT can be processed in the same time at design basis or 5,000 lb/hr as can be processed at 2,500 lb/hr.

3.1 Nitric-Formic Acid Flowsheet Experimental Results

A study was completed by SRNL to evaluate whether the addition of SEFT in the SME cycle would have any influence on nitric-formic Acid Flowsheet CPC processing.[3] This testing was designed to bound all future DWPF processing, so it included high noble metal concentration (HM or H Modified) and high SEFT addition volume (38,000 gallons, 8,000 in SRAT, 30,000 in SME). The report recommended "Based on the testing completed, an endorsement of the flowsheet change for adding strip effluent to the SME is not currently warranted." Processing issues noted in the report included high formate destruction leading to missing the REDOX target, high SRAT and SME product pH, rod fouling, high SRAT and SME product rheology, and high ammonia generation.

The nitric-glycolic acid flowsheet has been successful in improving processing that influences each of the issues noted above. This flowsheet has very low hydrogen and ammonia generation, stable pH, more stable rheology and less fouling. As a result, the addition of large volumes of Strip Effluent in the SRAT and SME cycle using the nitric-glycolic acid flowsheet could be acceptable.

3.2 Nitric-Glycolic Acid Flowsheet Experimental Results

Since only two of the nitric-glycolic acid flowsheet simulations, GN83 and SB9NG61, were completed with prototypic boilup rates (2,500 lb/hr scaled boilup rate) leading to long times at temperature, these two will be reviewed in detail to look for process upsets such as foaming, scaling, etc. that might impact DWPF processing using the nitric-glycolic acid flowsheet.

3.2.1 GN83[8]

CPC simulation GN83 was the last of a series of four SRAT and SME tests to determine bounding catalytic hydrogen production for the nitric-glycolic flowsheet. GN83 was the longest test with a total SRAT and SME boiling time of approximately 92 hours (~4 days) post acid addition. The boil-up rate was a scaled 2,500 lb/hr. 18,000 scaled gallons of SEFT, and 6,000 gallons of canister decontamination water simulants were added but no PRFT simulant was added. A graph showing the temperature profile of the test is in Figure 3-1.

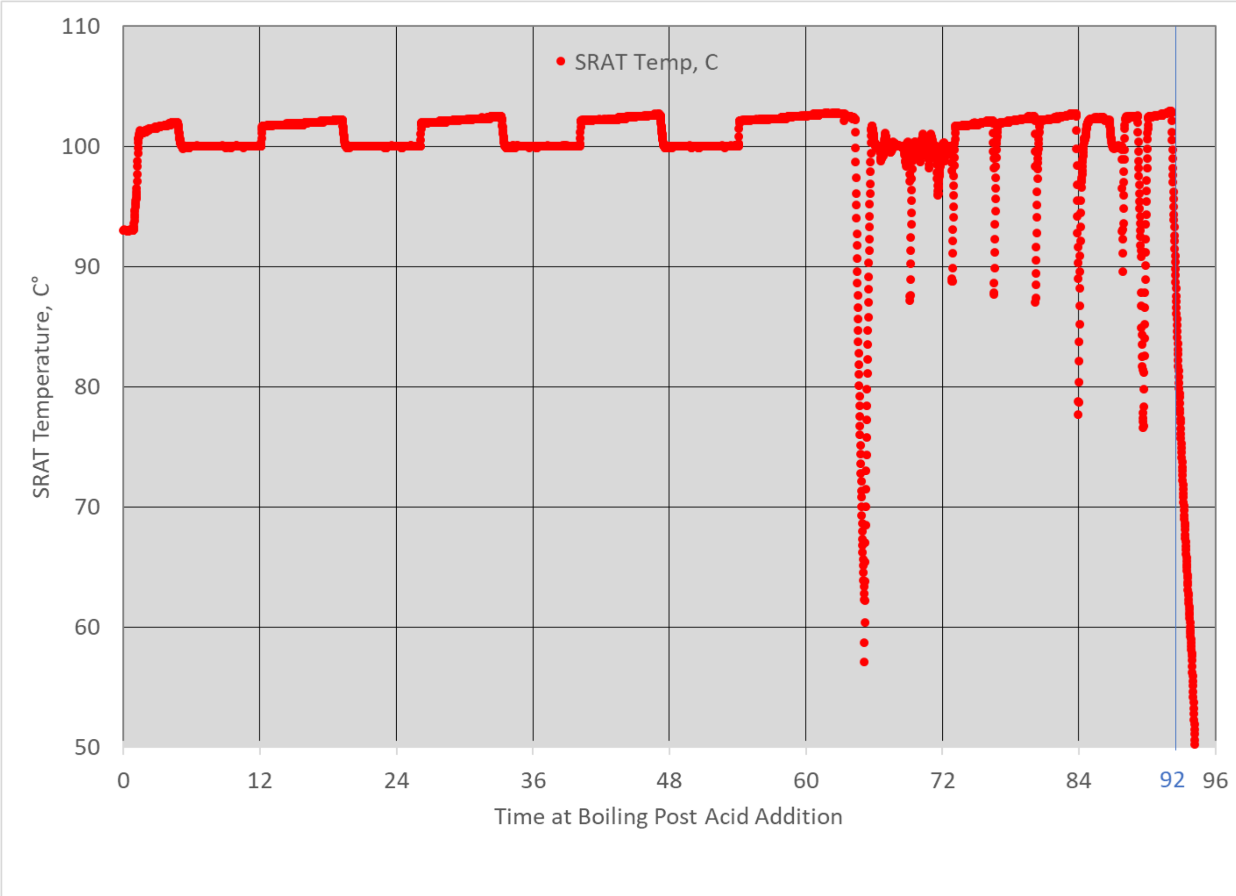

Figure 3-1. GN83 Temperature Profile post Acid Addition

The Koopman Minimum Acid (KMA) stoichiometry[13] was 110% for Run GN83 (118% Hsu[14]). Note that high noble metals concentrations were used. The Rh concentration was 3.56 times higher than SB9NG51 and the Ru concentration was 3.04 times higher than SB9NG51 (see Table 3-1). The noble metals in this test would have been very challenging for the nitric-formic acid flowsheet. Even though the acid stoichiometry was on the high side of efficient processing and the noble metals were also high, the pH was stable throughout the 92 hours of boiling at ~4 (Figure 3-2). Note that the increases in temperature following by drops in temperature were caused by boiling at half the time at 5,000 lb/hr boilup followed by half the time at "simmer" to average a boilup of 2,500 lb/hr. The laboratory measured pH at room temperature was 4.60 for the SRAT product and 4.66 for the SME product. Throughout the experiment, a pH meter continuously monitors pH. The laboratory pH is more accurate than the online pH measurement, which must be stable for up to 4 days without calibration,

Table 3-1. GN83 Trimmed Sludge Noble Metal and Mercury Targets, wt % total solids basis

Metal	Concentration
Ag	0.0164
Pd	0.0034
Rh	0.0475
Ru	0.2713
Hg	1.00

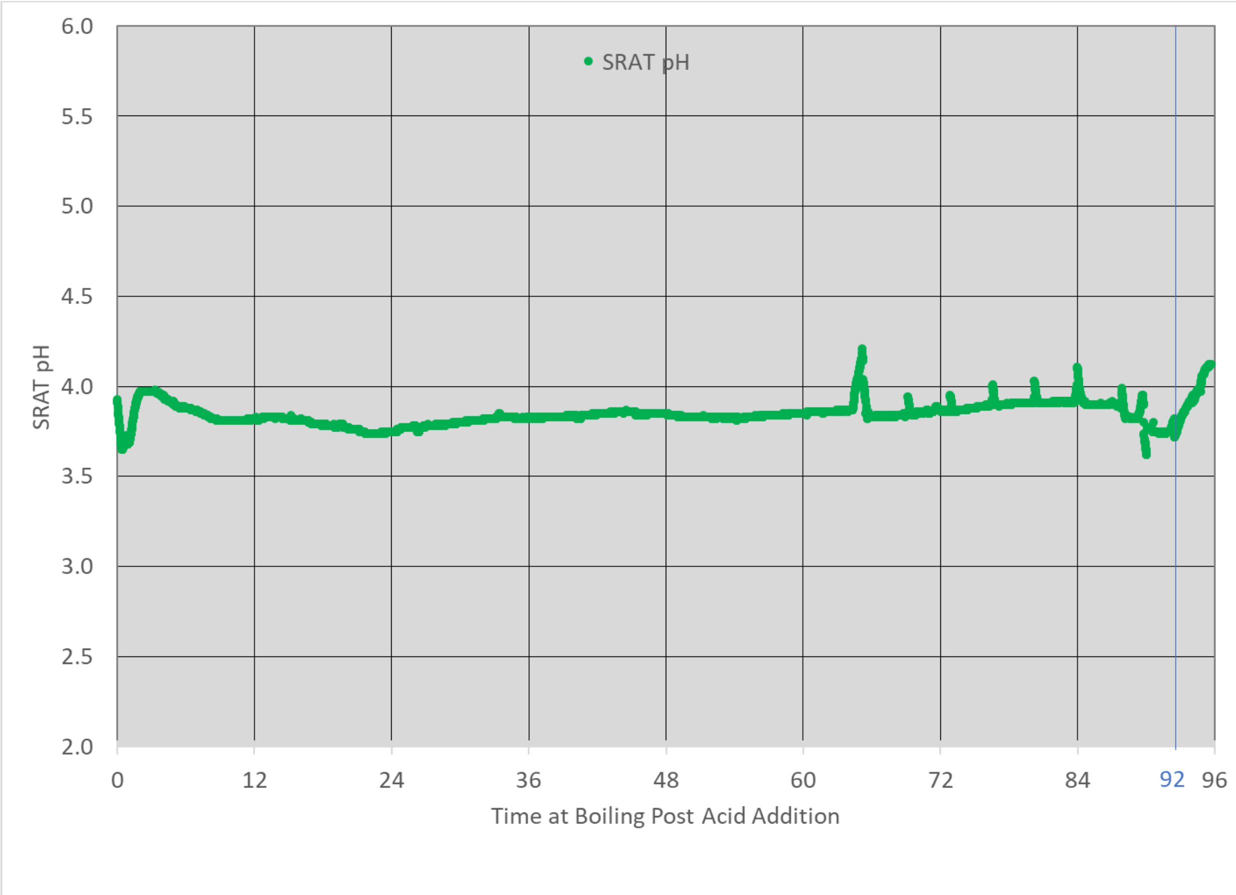

Figure 3-2. GN83 pH Profile post Acid Addition

Records for Run GN83 were reviewed to search for processing issues, including the ones identified in the nitric-formic acid flowsheet SEFT to SME report. None were identified.

3.2.2 SB9NG61[5]

During the development of the nitric-glycolic acid flowsheet for Sludge Batch 9 (SB9), one SRAT and SME simulation, SB9NG61, was completed with no PRFT or SEFT simulant, and 6,000 gallons of canister decontamination water simulant, with a scaled 2,500 lb/hr boilup in both the SRAT and SME cycle. The total time at boiling was 80 hours. A graph showing the temperature profile of the test is below in Figure 3-3.

The KMA stoichiometry was 100 % (Hsu 105%) for Run SB9NG61. SB9 levels of noble metals and mercury (Table 3-2) were low compared to those added to the sludge for Run GN83 but the mercury was higher. Even though the acid stoichiometry was on target for efficient processing and the noble metals were lower than Run GN83, the pH was stable throughout the 92 hours of boiling at ~4 (Figure 3-4). Note that the short drops in temperature each hour were caused by collecting condensate in the Mercury Water Wash Tank (MWWT) for the measurement of the boilup rate. The laboratory measured pH at room temperature was 4.93 for the SRAT product and 4.94 for the SME product.

Table 3-2. SB9 Trimmed Sludge Noble Metal and Mercury Targets, wt % total solids basis

Metal	Concentration
Ag	0.0139
Pd	0.0037
Rh	0.0156
Ru	0.0762
Hg	2.48

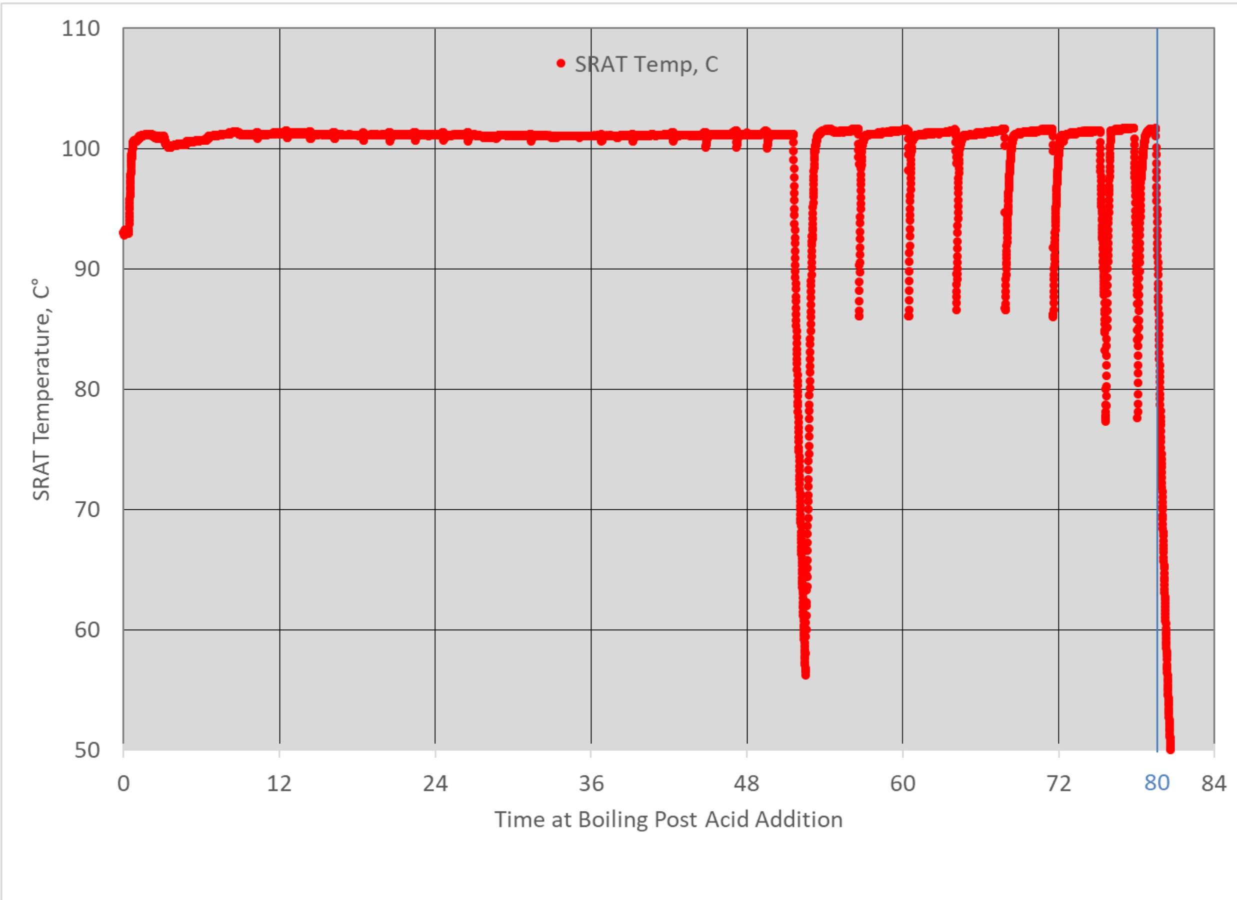

Figure 3-3. SB9NG61 Temperature Profile post Acid Addition

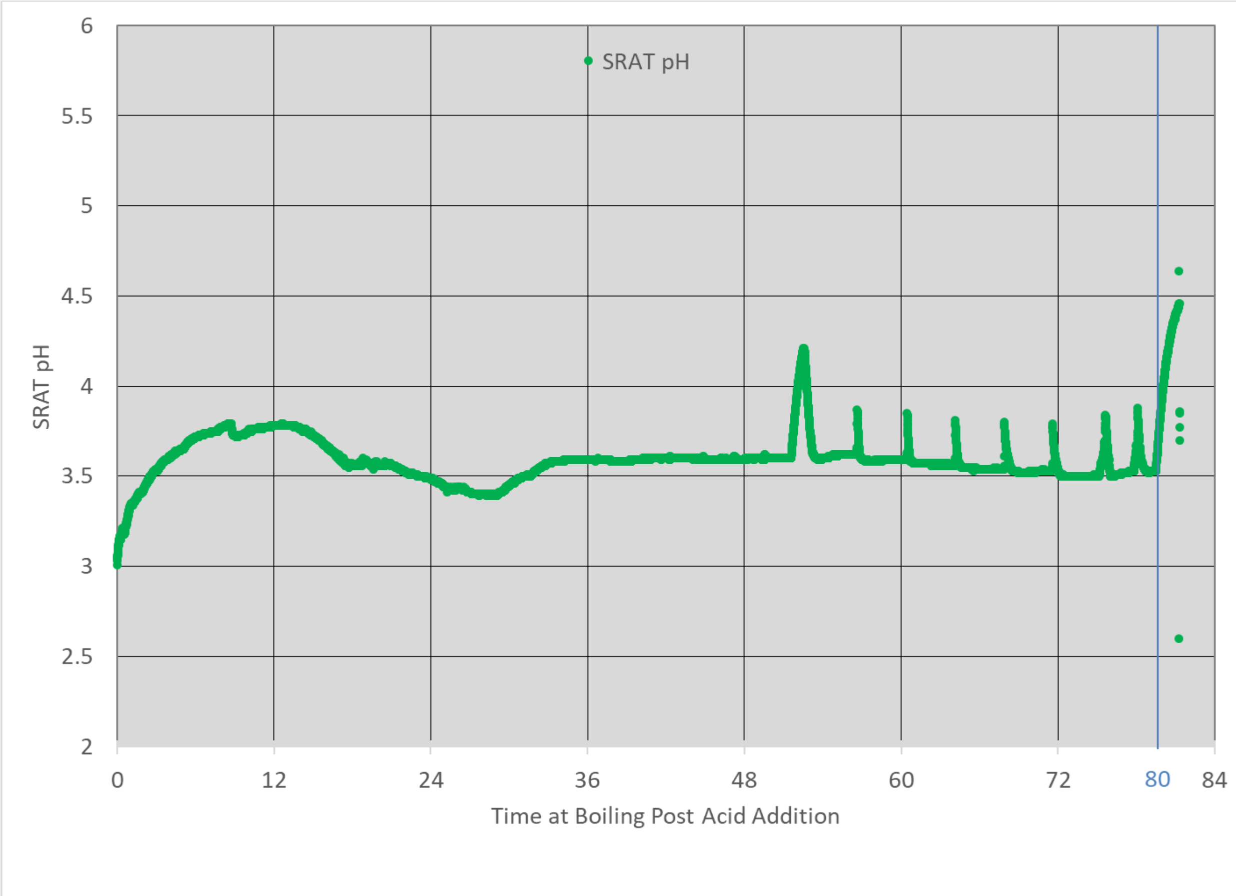

Figure 3-4. SB9GN61 pH Profile post Acid Addition

3.2.3 SB9NG58 and SB9NG60 pH profile[5]

At higher acid stoichiometry (100% KMA and above), the pH essentially remains constant throughout the SRAT and SME processing. A few tests were performed at lower acid stoichiometries (below 80% KMA), including NG58 and the SB9 shielded cells demonstration. The pH in NG58 (76.9% KMA) slowly increased during the 25 hours of boiling during the sludge-only SRAT cycle (Figure 3-5). It is likely that the pH would have continued to increase through a long SRAT and SME cycle. The final pH of the SRAT product was 7.75 for NG58 and 6.96 for SC-18 compared to 4.43 for the NG60 SME product. For SB9, the lower acid stoichiometries likely will lead to a processable SME product, but this might not be true for future sludge batches.

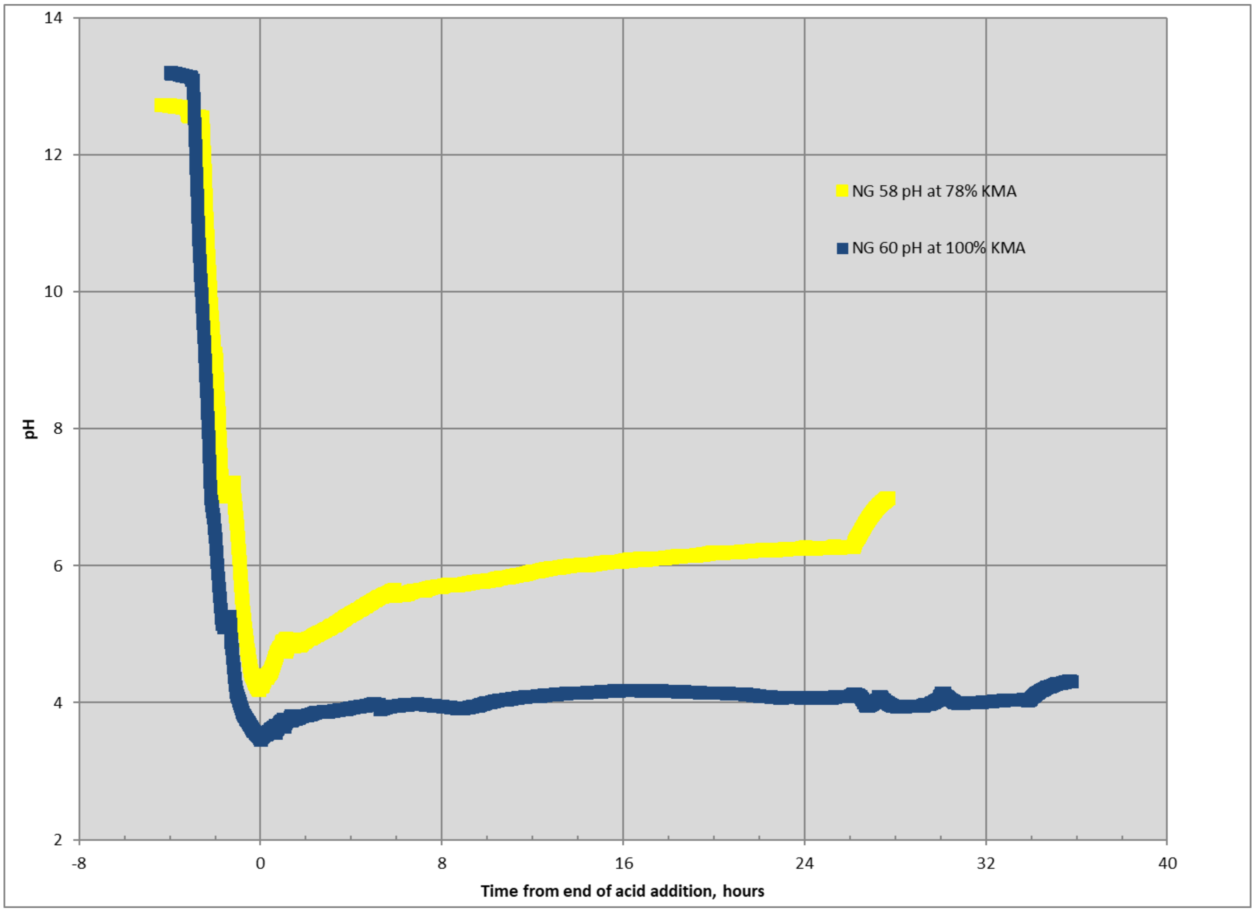

Figure 3-5. SB9NG58 and SB9NG60 pH Profile

3.3 Review of Nitric-Formic Acid Flowsheet Processing Data in DWPF

A review of DWPF SRAT and SME cycles was completed for SB9. Almost 800 SRAT and SME cycles have been completed in DWPF so limiting the review to SB9 still covered the years 2016-2019 and includes processing with PRFT and SEFT additions.

3.3.1 Long Duration SB9 Batches in DWPF

If the long processing times in SB9 batches led to process upsets, the review looked for evidence of processing problems such as high pH SME products. Longer boiling times led to high pH SME products as shown in Figure 3-6. The three operations with the longest processing times, 1.5-3.8 times the typical SB9 batch, were SB9 batches 784, 785 and 786. These batches also had the highest SME product pH.

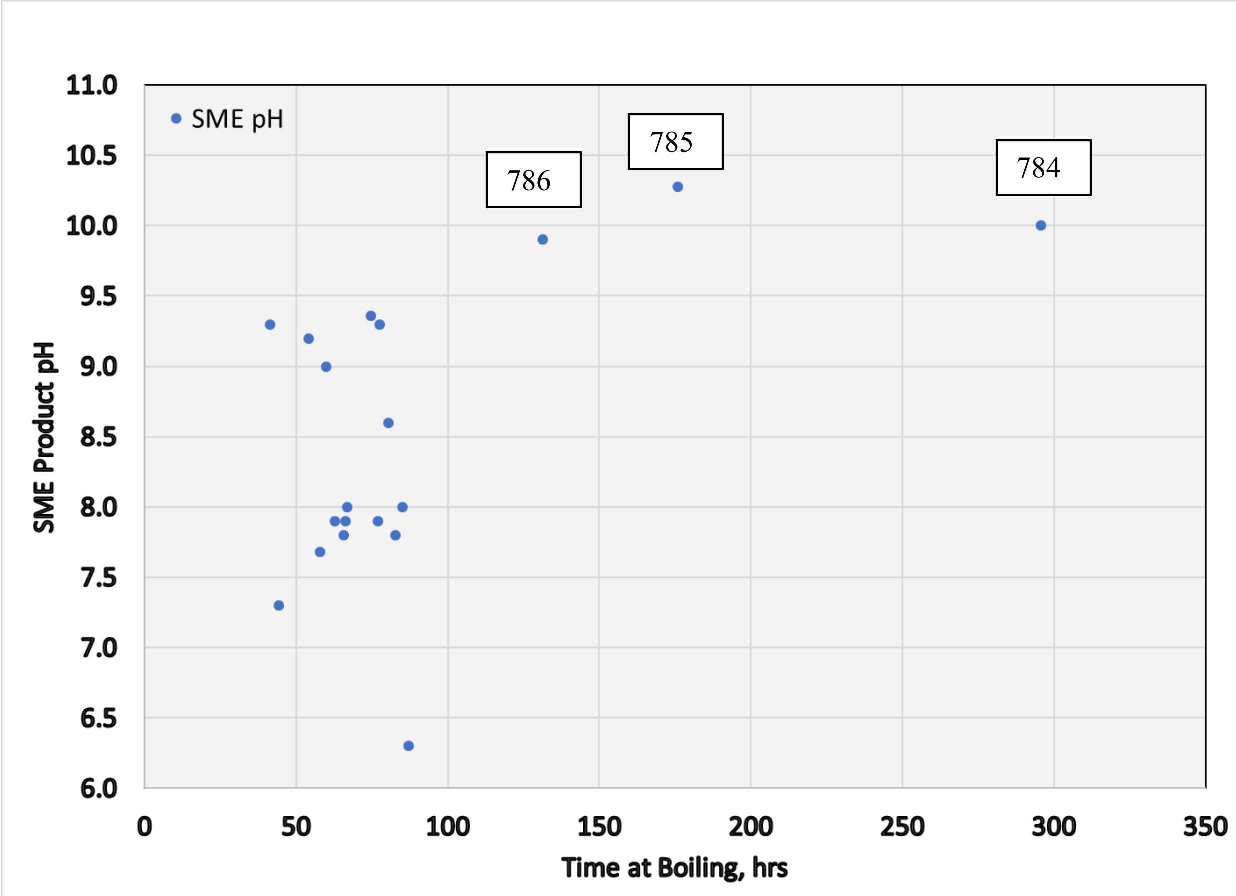

Figure 3-6. Impact of Boiling Time after Acid Addition on SME Product pH

High pH SME products can lead to thick slurry rheology, which may lead to processing problems including excessive melter trips caused by air entrainment in the Melter Feed Tank (MFT), foaming during boiling in the SRAT or SME and steam coil fouling. The long processing times can lead to significant anion destruction, which will result in missing the REDOX target. A graph (Figure 3-7) shows the influence of total steam flow on SME product pH. The processing data that was measured is summarized in Table 3-3.

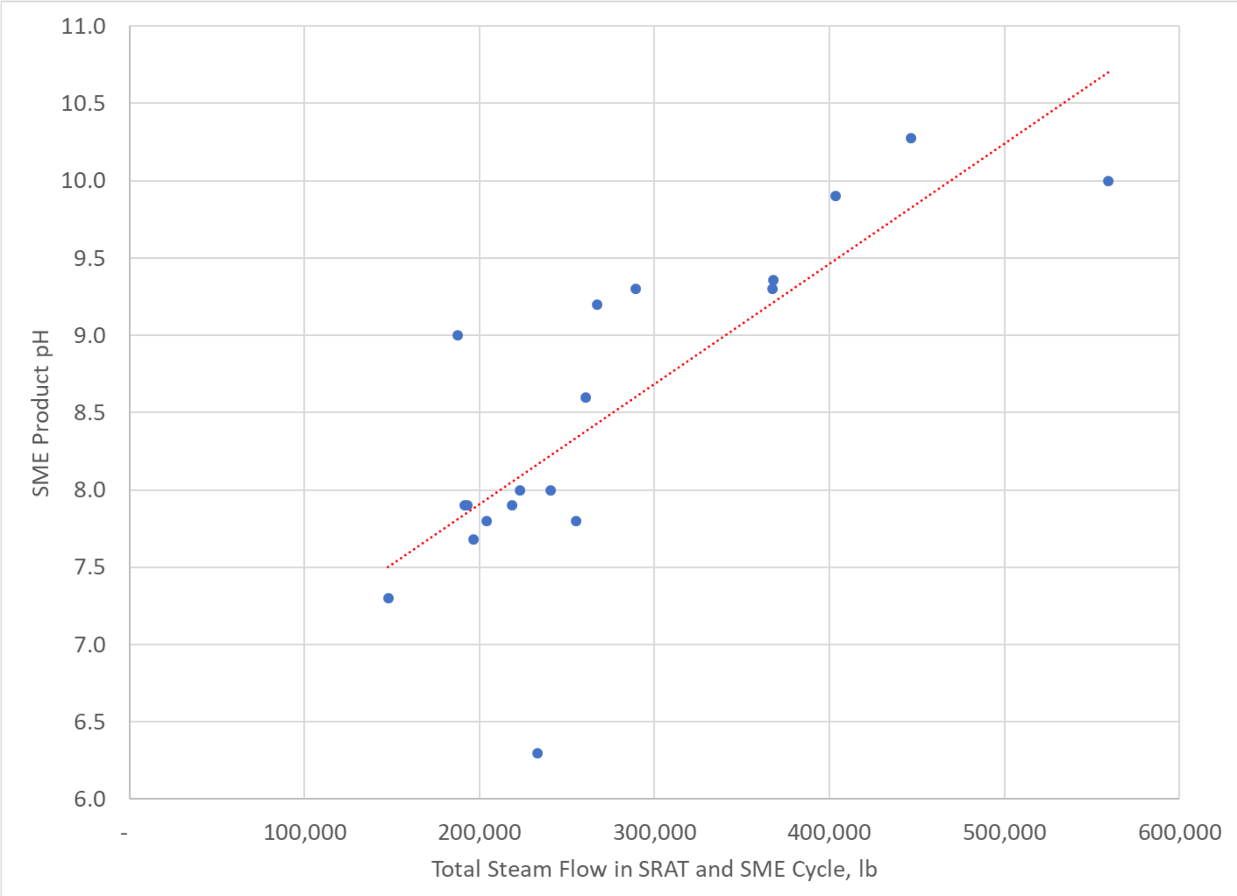

Figure 3-7. Impact of Total SRAT and SME Steam Mass on SME Product pH

Table 3-3. SB9 Processing Issues s

Batch	SRAT pH	SME pH	pH Delta	Melter Trips	cans blasted	Redox target	glass redox*	Delta Redox	Foam overs
774	7.6	7.9	0.3	0	4	0.15	0.16	0.01	0
775	7.2	7.8	0.6	3	5	0.15	0.14	-0.01	0
776	7.6	7.9	0.3	3	5	0.15	0.15	0.00	0
777	7.0	7.9	0.9	2	3	0.14	0.16	0.02	0
778	7.5	7.3	-0.2	0	2	0.15	0.13	-0.02	0
779	7.8	7.8	0.0	0	4	0.14	0.12	-0.03	0
780	7.4	8.0	0.6	1	6	0.16	0.16	0.00	0
781	7.5	6.3	-1.2	3	6	0.15	0.16	0.01	0
782	7.4	8.0	0.6	2	6	0.10	0.11	0.01	0
783	7.8	7.7	-0.1	0	1	0.11	0.09	-0.02	0
784	7.6	10.0	2.4	0	15	0.10	0.19	0.09	0
785	7.4	10.3	2.9	0	7	0.10	0.16	0.06	0
786	8.0	9.9	1.9	0	6	0.20	0.05	-0.15	0
787	7.3	9.0	1.7	0	0	0.15	0.09	-0.06	0
788	7.5	8.6	1.1	1	1	0.10	0.09	-0.01	0
789	7.3	9.4	2.0	0	6	0.10	0.07	-0.03	0
790	7.6	9.2	1.6	0	5	0.10	0.09	-0.01	0
791	7.6	9.3	1.7	0	8	0.10	0.09	-0.01	0
792	7.5	9.3	1.8	0	4	0.05	0.09	0.04	2
793	7.5	10.1	2.6	0	9	0.10	0.09	-0.01	1
794	7.9	10.8	2.9	Beyond Data Collected					

* Calculated from SME product analytical results

No foamovers or melter feed trips were noted during batches 784, 785 or 786. There was a large increase in pH during the SME cycle and there was a large discrepancy between the planned REDOX and the REDOX estimate based on SME product composition.

3.3.2 Estimation of Boiling Times post Acid Addition

Based on nitric-glycolic acid flowsheet simulant testing, nitrite destruction and mercury reduction are essentially complete by the end of SRAT dewater. Post dewater, the reactions with slow kinetics, including reduction reactions and nitrate and glycolate destruction have the potential to slowly change the REDOX. So, the best measure of the extent of the reactions is the boiling time after acid addition. As a result, the batch processing time was estimated using batch volumes of SEFT and canister blast water added. The time of boiling was estimated based on the mass of water added and the steam flowrate. Estimates of the boiling times were completed at both facility norm steam flowrate and design basis steam flowrate. The data is summarized in Table 3-4 for nominal processing volumes and in Table 3-5 for processing volumes that are three times nominal.

Table 3-4. Calculated Boiling Times - Nominal SEFT and Canister Decon Water Addition

			Facility Norms			Design Basis		
Step	Addition Volume, gal	Addition Mass, lb	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	25,035	NA	3,000	8.3	NA	5,000	5.0
SEFT Addition	9,000	75,105	6.0	3,000	25.0	10	5,000	15.0
Hg Stripping		9,895	NA	3,000	3.3	NA	5,000	2.0
Decon Canisters	5,000	41,725	NA	2,400	17.4	NA	5,000	8.3
Process Frit	2,700	22,532	NA	2,400	9.4	NA	5,000	4.5
Total Boiling Time	19,700	164,397	NA	NA	63.5	NA	NA	34.9

Table 3-5. Calculated Boiling Times – 3X SEFT and Canister Decon Water Addition

			Facility Norms			Design Basis		
Step	Addition Volume, gal	Addition Mass, lb	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	25,035	NA	3,000	8.3	NA	5,000	5.0
SEFT Addition	9,000	75,105	6.0	3,000	25.0	10	5,000	15.0
Hg Stripping		9,895	NA	3,000	3.3	NA	5,000	2.0
Decon Canisters	5,000	41,725	NA	2,400	17.4	NA	5,000	8.3
Process Frit	2,700	22,532	NA	2,400	9.4	NA	5,000	4.5
Total Boiling Time	19,700	164,397	NA	NA	63.5	NA	NA	34.9

3.3.3 Estimation of Maximum SEFT Volume

A calculation was completed to estimate the volume of SE that could be processed with a boiling time post acid addition of <92 hours. Estimates were made for both facility norm steam flowrate and design basis. The results are summarized in Table 3-6 for facility norm steam flowrates and Table 3-7 for design basis steam flowrates in the SRAT and SME. Based on these calculations, the maximum SEFT volume for Facility Norm steam flowrate is 20,000 gallons and the maximum SEFT volume for Design Basis steam flowrate is 44,000 gallons (3.4 times the projected volume each six days). Note this assumes only 5,000 gallons of canister blast water is added to the SME. If DWPF wants to process more canister blast water, then the volume of SEFT is recommended to be decreased to keep from exceeding the 92 hours of boiling.

Table 3-6. Calculated Maximum SEFT Volume for Facility Norm Steam Flowrate

Step	Addition Volume, gal	Addition Mass, lb	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	25,035	NA	3,000	8.3
SEFT Addition	20,500	171,073	5.5	3,000	57.0
Decon Canisters	5,000	41,725	NA	2,400	17.4
Process Frit	2,700	22,532	NA	2,400	9.4
Total Boiling Time	31,200	260,364	NA	NA	92.1

Table 3-7. Calculated Maximum SEFT Volume for Design Basis Steam Flowrate

Step	Addition Volume, gal	Addition Mass, lb	Addition rate, gpm	Boilup Rate, lb/hr	Boiling Time, hr
Dewater	3,000	25,035	NA	5,000	5.0
SEFT Addition	44,500	371,353	0	5,000	74.3
Decon Canisters	5,000	41,725	NA	5,000	8.3
Process Frit	2,700	22,532	NA	5,000	4.5
Total Boiling Time	55,200	460,644	NA	NA	92.1

3.3.4 Restrictions for When SEFT Can Be Added in SRAT and SME

The SEFT liquid includes entrained organic, primarily Isopar. As a result, the SE must be added at boiling so that the Isopar flashes off as it is added and does not accumulate. SE has never been added during SRNL testing under caustic conditions, so the SE should be added in the SRAT post acid addition. During SME processing, the SEFT liquid should not be added after SME product samples have been pulled in case a foamover or a fouled coil could lead to a composition change. The SE can be added any time prior to pulling the SME product sample.

3.4 Recommendations for DWPF Processing

The startup of SWPF will be a significant challenge for DWPF. To keep up with the expected SEFT and PRFT volumes (SWPF plans to produce 12,800 gallons of SEFT and 2,800 gal of PRFT every six days[15]), DWPF will need to process more efficiently. This will likely require increased steam flowrate in the SRAT and the SME. A new antifoam and/or implementation of the nitric-glycolic acid flowsheet should allow the facility to return to design basis steam flowrate and result in significant reductions in boiling time, without facility interruptions caused by foamover remediations.

It should be noted that the SRAT and SME condensers are undersized for the nitric-formic acid air purge rates. As a result, the ammonia scrubbers and formic acid vent condenser utilize chilled water to serve as secondary condensers. Much of the condenser load is used to cool the offgas. Increasing the boilup rate to design basis will increase the load on the condensers. To handle this increased load, the SRAT and SME air purge rates will have to be reduced and the condensers may need to be cleaned to ensure the condensers can effectively cool the offgas.

DWPF would also like the capability to process SEFT in either the SRAT or SME cycle while processing with the nitric-glycolic acid flowsheet. It is recommended that the boiling time after acid addition in both the SRAT and SME be limited to the maximum time tested in SRNL experiments or 92 hours. The SEFT addition can be split as needed between SRAT and SME processing. During typical SB9 processing, the SEFT addition time and the mercury stripping time are very similar so it wouldn't be practical to add SEFT to the SME. However, if the SEFT volume in a batch is increased to 3x nominal, the addition of SEFT to SME would offer flexibility in making the processing time in the SRAT and SME equal. If design basis steam flowrate can be achieved in DWPF, at least 3x the SEFT, and canister blast water could be processed without exceeding the 92 hours of boiling after acid addition.

Although DWPF may be capable of processing larger volumes of PRFT and SEFT, it is still recommended that DWPF focus on constant processing of batches as this is the most effective way to process in the CPC and melter. Constant processing would consist of adding the same volume of sludge, PRFT, and SEFT in each SRAT cycle and processing the same volume of water from canister blasts each SME cycle. It is especially important that the PRFT volume in each batch is essentially uniform, as the PRFT volume has a strong influence on the melter feed composition, since uniform melter feed composition is good for melter processing.

During the review of the SRAT and SME processing data from SB9, it was noted that during some of the batches, the time at temperatures greater than 90 °C is significantly longer than the time at boiling. Since nitric and formic acid are both added at 93 °C the time >90 °C should be up to twelve hours longer than at boiling. During Runs 789 and 792, the time >90 °C was more than 100 hours (4 days) longer than the time at boiling. Since reaction rates decrease by roughly half for every 10 °C drop in temperature, there may be more total reactions occurring at 90 °C than at boiling. It is recommended that the SRAT and SME are not simmered for long periods at high temperatures, especially after acid addition is complete for the nitric-formic acid flowsheet.

It should be noted that the PRFT and the SEFT addition flows are limited to 10 gallons per minute (gpm) by the Consolidated Hazard Analysis[16] (CHA) for potential organics flammability concerns. Due to additional conservatism in the jumper and orifice design, the actual addition rate is 6-7 gpm. Since the PRFT and SEFT addition rate is limited by the system design, increasing the steam addition rate to 5,000 lb/hr during PRFT and SEFT addition would lead to over concentration of the SRAT. In batches with a high mercury concentration where the Hg strip time is longer than the SEFT addition time, increasing the steam addition rate would shorten the time for Hg removal during the post SEFT addition reflux period. This potential benefit would be different for each sludge batch and would not shorten processing during SB9 sludge processing since it is low in Hg. Additional SRAT cycle time reduction may be achieved for future sludge batches that are higher in Hg, but would require facility modifications, purge reductions, and CHA changes to allow for SRAT and SME processing at design basis steam addition flowrate.

4.0 Conclusions

At the request of SRR, a white paper was written to assess whether it would be acceptable to process SE in both the SRAT and SME in DWPF for the nitric-glycolic acid flowsheet. SRNL experimental data and DWPF SB9 process data was reviewed, looking at batches which included PRFT and/or SEFT feeds that led to long processing times. These batches were reviewed looking for processing problems such as melter feed trips, foamovers, heating rod or steam coil fouling, missed REDOX targets, and other process anomalies.

The nitric-glycolic acid flowsheet was very effective during SRNL testing in processing sludge, PRFT and SEFT feeds without the processing problems noted above. One of the advantages of this flowsheet is the stable pH of the SRAT and SME products, indicative of the anion chemistry being essentially complete by the end of the SRAT dewater. In contrast, the nitric-formic acid flowsheet processing leads to a pH increase of up to 3 pH units during just the SME cycle in extended processing in DWPF. The longest of the SRNL experiments had a boiling time after acid addition of 92 hours.

A review of the DWPF batch history from SB9 identified three batches with extremely long processing times. Batches 784, 785 and 786 had up to 300 hours at boiling after acid addition. This led to large pH changes in the SME cycle, with SME product pH above 9 and significant deviations from the REDOX target. No significant foaming, coil fouling, or melter feed trips were noted. This processing was not used to extend maximum boiling time due to issues in these batches with pH changes and REDOX The use of the nitric-glycolic flowsheet, at a pH of 6 or above, where issues with rheology and coil fouling were rarely noted in simulant runs, likely would have been successful in meeting all processing targets even during these extremely long processing times.

5.0 Recommendations

Based on the review, the following is recommended for the nitric-glycolic acid flowsheet.

1. Strip Effluent can be added post acid addition during either the SRAT or SME cycle during processing using the nitric-glycolic acid flowsheet. If boiling times beyond 92 hours are necessary, the facility should pay close attention to pH changes and REDOX which may lead to foaming, coil fouling, and/or melter trips. Further testing at extended boiling times could be performed to alleviate this potential concern. If the facility desires the flexibility to add SEFT during caustic boiling, additional laboratory testing is needed.
2. The maximum volume of SEFT that can be added during a 92 hour SRAT and SME cycle depends on steam flowrate and the volume of canister blast water that requires evaporation. Assuming 5,000 gallons of canister blast water, the maximum volume of SEFT that can be processed is 20,000 gallons at typical steam flowrate (3,000 lb/hr SRAT, 2,400 lb/hr SME) and 44,000 gallons at design basis steam flowrate (5,000 lb/hr SRAT and SME).
3. In order to maximize the volume of SE and MST/SS processed in a batch as well as reduce SRAT/SME cycle times, it is recommended that DWPF explore opportunities to restore design basis steam flow rates. However, it is recognized that realization of this opportunity would require facility modifications, purge reductions, and CHA changes.

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