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Measurement Acceptance Region (MAR) Assessment Results Based on the October 2019 Sludge Batch 10 Projections

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EXECUTIVE SUMMARY

Recent analysis of a Tank 26 sample indicated a higher than anticipated sulfur concentration in the supernatant and a significant amount of insoluble sulfur. The final concentration in the glass product would be well above the sulfate limits of 0.6-0.65 weight percent (wt.%) determined for previous sludge batches. In preparation for Sludge Batch 10 (SB10) processing, washing of Tank 51 will be performed to reduce the sulfate concentration in the glass product to less than 0.65 wt.% at waste loadings (WLs) of interest. To evaluate the feasibility of two Tank 51 Na wash endpoints (0.8 and 1.2M), Savannah River Remediation requested that the Savannah River National Laboratory perform assessments using the Defense Waste Processing Facility Product Composition Control System (PCCS) models and their associated Measurement Acceptance Region (MAR) constraints.

The objective of this study was to determine whether a glass frit could be developed for these washing options to yield an acceptable glass product over 32-40% waste loading (WL) while incorporating sufficient volumes of streams from the Salt Waste Processing Facility (SWPF). These high activity streams from SWPF include monosodium titanate and sludge solids from the Sludge Solids Receipt Tank and Cs-containing strip effluent.

Based on these MAR assessment results, frits are available for both the 0.8M and 1.2M Na wash endpoint options for coupled operation with SWPF. Frit 418 was identified as a viable candidate and was previously used to process Sludge Batches 3-6, 7a, and 7b. For coupled operations, the operating window is at least 9 percentage points and it demonstrates the ability to maximize salt waste throughput. Due to the high concentrations of Al_2O_3 and Na_2O, a target WL of 36% may not be achievable, but evaluations of potential SME compositions with PCCS during blending evaluations will confirm the WL that can be targeted for a particular SME batch. For sludge only operations, there is an is operating window of 25-36% WL for the 1.2M Na wash endpoint; however, there is no operating window for the 0.8M Na wash endpoint.

Once washing has been completed and the Tank 40 blend projection has been updated, additional MAR assessments should be conducted to continue the frit development effort for SB10. It is expected that iterative MAR assessments will be performed as the SB10 Tank 40 blend projection matures. After the Tank 40 blend projection is finalized and a viable frit(s) has been identified for SB10 processing, these remaining tasks will be completed.

- Fabrication and viscosity testing to verify that the frit vendor will not have any manufacturing issues (if necessary)
- Assessments and potential experiments related to the variability study and sulfate solubility limit
- Assessment of glass density used for fissile mass loading calculations

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
EVs	extreme vertices
MAR	Measurement Acceptance Region
MST	monosodium titanate
NGS	Next Generation Solvent
PCCS	Product Composition Control System
SB10	Sludge Batch 10
SB9	Sludge Batch 9
SE	Strip Effluent
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
WL	waste loading
wt.%	weight percent

1.0 Introduction

Recent analysis of a Tank 26 sample indicated a higher than anticipated sulfur concentration in the supernatant and a significant amount of insoluble sulfur.[1] The final concentration in the glass product would be well above the sulfate limits of 0.6-0.65 weight percent (wt.%) determined for previous sludge batches.[2-11] In preparation for Sludge Batch 10 (SB10) processing, washing of Tank 51 will be performed to reduce the sulfate concentration in the glass product to less than 0.65 wt.% at waste loadings (WLs) of interest. To evaluate the feasibility of two Tank 51 Na wash endpoints (0.8 and 1.2M), Savannah River Remediation (SRR) requested that the Savannah River National Laboratory (SRNL) perform assessments using the Defense Waste Processing Facility (DWPF) Product Composition Control System (PCCS) models and their associated Measurement Acceptance Region (MAR) constraints.

The objective of this study was to determine whether a glass frit could be developed for these washing options to yield an acceptable glass product over 32-40% WL while incorporating sufficient volumes of streams from the Salt Waste Processing Facility (SWPF). These high activity streams from SWPF include monosodium titanate (MST) and sludge solids (SS) from the Sludge Solids Receipt Tank (SSRT) and Cs-containing strip effluent (SE).

2.0 Quality Assurance

This work was requested via a Technical Task Request (TTR)[12] and directed by a Task Technical and Quality Assurance Plan.[13] The functional classification of this task is Production Support. This task is not waste form affecting and does not need to follow the quality assurance requirements of RW-0333P.[14] Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.[15] This document, including all calculations, was reviewed by a Design Check. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[16] All calculations, document preparation, and reviews satisfy the quality requirements for Production Support.

3.0 Inputs and Assumptions

Two SB10 Tank 40 blend projections (calcine basis) representing a 0.8M and 1.2M Na wash endpoint were received from SRR in October 2019.[17] The elemental concentrations were converted to oxides and normalized to 100 wt.% as shown in Table 3-1.

Table 3-1. Normalized SB10 Tank 40 Blend Projection (wt.%)

Oxide	0.8M Na Wash Endpoint	1.2M Na Wash Endpoint	Oxide	0.8M Na Wash Endpoint	1.2M Na Wash Endpoint
Al_2O_3	34.48	32.42	MnO	4.38	4.05
B_2O_3	0.05	0.05	Na_2O	23.24	27.92
BaO	0.09	0.08	NiO	0.81	0.75
CaO	1.86	1.72	PbO	0.10	0.10
Ce_2O_3	0.10	0.09	SO_4^{2-}	1.74	2.10
Cr_2O_3	0.62	0.58	SiO_2	1.86	1.73
CuO	0.06	0.05	ThO_2	1.80	1.67
Fe_2O_3	22.22	20.58	TiO_2	0.03	0.03
K_2O	0.22	0.23	U_3O_8	5.42	5.02
La_2O_3	0.05	0.04	ZnO	0.04	0.04
Li_2O	0.05	0.05	ZrO_2	0.17	0.15
MgO	0.61	0.56			

SRNL performed subsequent calculations to estimate compositions of SE[18] and the SSRT effluent stream.[19,20] Of the five cases originally evaluated for Sludge Batch 9 (SB9), the following three cases were evaluated for SB10:

- Case 1: Single MST strike operation and no entrained insoluble sludge solids. This case represents the baseline for coupled processing with SWPF.
- Case 3: Single MST strike operating and 600 mg/L of entrained insoluble sludge solids, which were assumed to be SB9 sludge solids.
- Case 4: Double MST strike operation (i.e., two sequential contactings of waste in SWPF with 0.4 g MST/L of waste in each) and no entrained insoluble sludge solids.

Case 2 and Case 5 represented more conservative cases for SB9 where the aluminum concentration in the SSRT effluent stream was set to 0 mg/mL.[20,21] These cases were omitted from this SB10 evaluation since the Tank 40 blend Al_2O_3 concentration is almost two times higher than that of SB9 and there is no risk of failing a PCCS constraint[22] related to a low Al_2O_3 concentration.

Once the MAR assessments were completed for Cases 1, 3, and 4, an evaluation of the sludge-only case was requested by SRR; however, it was not included in the frit development effort described in this report.

Other pertinent inputs include:

- 0.7M Na (total Na) wash endpoint for the SSRT stream[21]
- SE has a Cs-137 concentration of 66 curies/gallon[21]
- DWPF receives 6000 gallons of sludge slurry from Tank 40 per Sludge Receipt and Adjustment Tank (SRAT) batch[17]
- DWPF receives 12,800 gallons of SE per SRAT batch based on the Next Generation Solvent (NGS)[a,21]
- Frit must be able to accommodate 2800 gallons of the SSRT effluent stream (MST/SS) per SRAT batch for MST single strike operation and 4200 gallons for MST double strike operation[21]

4.0 Methodology for the Variation Stage MAR assessments

The approach taken for the Variation Stage MAR assessments[23] was to evaluate how robust candidate frit compositions were relative to expected variation in the composition of the SB10 SRAT material and the uncertainty in targeting the desired waste loading (WL). These uncertainties take effect as DWPF (i) conducts the blending process[b] to target the desired WL for the next Slurry Mix Evaporator (SME) batch, and (ii) subsequently judges the new SME batch for MAR acceptability via the PCCS process, which is driven by the analysis of samples of the new SME batch.

Cases 1 and 3 (single strike operation) were evaluated at 2400-3600 gallons of MST/SS and Case 4 (double strike operation) was evaluated at 3800-4200 gallons of MST/SS in increments of 400 gallons. Compositional variation (±) was applied to SRAT compositions representing each volume addition of the MST/SS stream to account for likely, but not necessarily bounding, differences that may be seen in the material that is transferred from Tank 40 and SWPF into the SRAT during the processing of SB10. The compositional variation for the individually-tracked oxides was represented by the larger of 0.5 wt.% or 7.5% of the nominal concentration. Those oxides not tracked individually were grouped into an "Others"

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[a] NGS contains the extractant MaxCalix (1,3-alt-25,27-bis(3,7- dimethyloctyl-1-oxy)calix[4]arene-benzocrown-6), which uses a boric acid strip solution.
[b] Combining SRAT material with frit and the heel of the SME.

component.[c] The resulting SRAT oxide intervals are summarized in Table 4-1 and Table 4-2, which represent the minimum and maximum oxide concentrations for the various cases of sludge-only, and MST/SS and SE additions. Extreme vertices (EVs) were generated for the SB10 SRAT compositions representing sludge-only and at each volume of the MST/SS evaluated in these assessments.

Table 4-1. SRAT Oxide Intervals (wt.%) for the 0.8M Na Wash Endpoint

Oxide	Sludge Only	Case 1 Single Strike	Case 3 Single Strike 600 mg/L solids	Case 4 Double Strike
Al_2O_3	31.90-37.07	25.82-31.68	25.50-31.20	24.28-28.78
B_2O_3	0-0.55	0.16-1.21	0.05-1.12	0.12-1.14
CaO	1.36-2.36	0.96-2.05	1.09-2.14	0.87-1.90
Cs_2O	0-0.50	1.03-2.13	0.78-1.93	0.94-1.97
Fe_2O_3	20.56-23.89	16.11-19.98	19.51-22.77	15.11-17.97
MnO	3.88-4.88	2.93-4.16	4.49-5.75	2.72-3.79
Na_2O	21.49-24.98	24.23-29.52	21.15-24.64	24.82-29.17
NiO	0.31-1.31	0.13-1.17	0.28-1.28	0.09-1.11
SiO_2	1.36-2.36	0.96-2.06	1.58-2.67	0.87-1.90
ThO_2	1.30-2.30	0.91-2.01	0.91-1.99	0.83-1.86
TiO_2	0-0.53	6.00-9.80	5.18-8.16	11.39-14.29
U_3O_8	4.92-5.92	3.75-5.03	3.98-5.17	3.48-4.58
Others	3.35-4.35	2.76-3.93	2.76-3.91	2.57-3.63

Table 4-2. SRAT Oxide Intervals (wt.%) for the 1.2M Na Wash Endpoint

Oxide	Sludge Only	Case 1 Single Strike	Case 3 Single Strike 600 mg/L solids	Case 4 Double Strike
Al_2O_3	29.98-34.85	24.72-30.20	24.59-29.92	23.34-27.63
B_2O_3	0-0.55	0.12-1.16	0.02-1.08	0.08-1.09
CaO	1.22-2.22	0.87-1.96	1.01-2.05	0.80-1.82
Cs_2O	0-0.50	0.93-2.01	0.70-1.84	0.84-1.87
Fe_2O_3	19.04-22.13	15.21-18.77	18.52-21.56	14.33-17.01
MnO	3.55-4.55	2.74-3.94	4.21-5.48	2.55-3.62
Na_2O	25.82-30.01	27.70-33.22	24.09-28.43	27.93-32.68
NiO	0.25-1.25	0.10-1.13	0.24-1.24	0.06-1.07
SiO_2	1.23-2.23	0.88-1.96	1.46-2.56	0.80-1.83
ThO_2	1.17-2.17	0.83-1.92	0.84-1.91	0.76-1.78
TiO_2	0-0.53	5.51-9.12	4.81-7.68	10.62-13.35
U_3O_8	4.52-5.52	3.51-4.76	3.76-4.92	3.28-4.36
Others	3.57-4.57	2.98-4.15	2.94-4.10	2.79-3.85

To support these assessments, SRNL utilized a method to allow for a rapid screening and an assessment of a large array of candidate frits to simultaneously satisfy the operating scenarios represented by Cases 1, 3, and 4. Note that the sludge-only case is solely for reference and was not included in the frit development effort. For each of the scenarios represented by Table 4-1 and Table 4-2, all the EVs for Cases 1, 3, and 4 were combined with an array of over 10,000 frits covering the B_2O_3-Li_2O-MgO-Na_2O-SiO_2 region at WLs in the interval of 24 – 40%. Each of the resulting glass compositions was evaluated against the PCCS MAR criteria to determine whether the composition would pass the SME acceptability process. An overall

[c] The "Others" components include BaO, Ce_2O_3, Cr_2O_3, CuO, K_2O, La_2O_3, Li_2O, MgO, PbO, SO_4^{2-}, ZnO, and ZrO_2.

operating window of at least 9 percentage points across Cases 1, 3, and 4 (simultaneously) was the primary success metric used to select a frit for SB10 processing.

5.0 Variation Stage MAR Assessment Results

Of the 10,000+ frits evaluated, Frit 418 ($8B_2O_3$-$8Li_2O$-$8Na_2O$-$76SiO_2$, wt.%) was identified as a viable candidate for SB10 processing with both the 0.8M and 1.2M Na wash endpoints for coupled operation with SWPF. Frit 418 is already included in the DWPF frit specification[24] as it was used to process Sludge Batches 3-6, 7a, and 7b.

Table A-1 and Table A-2 in Appendix A provide the results of the MAR assessments with Frit 418 for the 0.8M and 1.2M Na wash endpoint options, respectively. The Al_2O_3, Na_2O, and TiO_2 concentrations in the SRAT (wt.%) are provided for reference. Consider Case 1 in Table A-1 as an example for the interpretation of the information provided. At 2400 gallons of MST/SS per SRAT batch, the operating window (WL interval over which all EVs pass the SME acceptability process) is 24-38% WL (shaded green). The number of EVs evaluated was 9408. At 39% WL, 7.2% of the EVs fail the nepheline constraint (shaded red).

Excluding the sludge-only case, the target WL is generally 33-34% to ensure a ±4 percentage point buffer around the target. Blending calculations and evaluations of potential SME compositions with PCCS may allow higher WLs to be targeted for a particular SME batch. For sludge-only operation, the total Na_2O concentration is too low for the 0.8M Na wash endpoint option, which results in no operating window. The 1.2M Na wash endpoint option exhibits minimal differences between the operating windows for sludge-only and coupled operation.

The failed nepheline constraint at higher waste loadings for some of the EVs is waste form affecting as it reduces the chemical durability of the waste form. Nepheline ($NaAlSiO_4$) is a crystalline phase that is prone to form in glass during slow cooling when higher concentrations of Al_2O_3 and Na_2O are present.[25] The formation of nepheline is controlled in the DWPF PCCS by the following expression:[22,26]

$$\frac{SiO_2}{Al_2O_3+Na_2O+SiO_2} > 0.62$$

where SiO_2, Na_2O, and Al_2O_3 are the concentrations in the glass as mass fractions. Nepheline is *not* predicted to form when the value is greater than 0.62.

Due to the Cr_2O_3 concentration in the sludge, the solubility constraint for Cr_2O_3 fails for some of the EVs at higher waste loadings. The PCCS limit for Cr_2O_3 is 0.3 wt.% minus measurement uncertainty.[22]

6.0 Conclusions

Based on these MAR assessment results, frits are available for both the 0.8M and 1.2M Na wash endpoint options for coupled operation with SWPF. Frit 418 was identified as a viable candidate and was previously used to process Sludge Batches 3-6, 7a, and 7b. For coupled operations, the operating window is at least 9 percentage points and demonstrates the ability to maximize salt waste throughput. Due to the high concentrations of Al_2O_3 and Na_2O, a target WL of 36% may not be achievable, but evaluations of potential SME compositions with PCCS during blending evaluations will confirm the WL that can be targeted for a particular SME batch. For sludge only operations, there is an is operating window of 25-36% WL for the 1.2M Na wash endpoint; however, there is no operating window for the 0.8M Na wash endpoint.

7.0 Recommendations

Once washing has been completed and the Tank 40 blend projection has been updated, additional MAR assessments should be conducted to continue the frit development effort for SB10. It is expected that iterative MAR assessments will be performed as the SB10 Tank 40 blend projection matures. After the Tank 40 blend projection is finalized and a viable frit(s) has been identified for SB10 processing, these remaining tasks will be completed.

- Fabrication and viscosity testing to verify that the frit vendor will not have any manufacturing issues (if necessary)
- Assessments and potential experiments related to the variability study and sulfate solubility limit
- Assessments of glass density used for fissile mass loading calculations[27]

8.0 References

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Appendix A. MAR Assessment Results

Table A-1. MAR Assessment Results for the SB10 0.8 Na Wash Endpoint

Case	Sludge Only	Case 1 Single Strike - No Sludge Solids				Case 3 Single MST Strike - 600 mg/L Sludge Solids				Case 4 Double MST Strike - No Sludge Solids	
MST/SS Volume (gallons)	0	2400	2800	3200	3600	2400	2800	3200	3600	3800	4200
SRAT Al_2O_3 (wt.%)	34.48	29.47	28.93	28.41	27.92	29.02	28.50	28.02	27.57	26.77	26.25
SRAT Na_2O (wt.%)	23.24	26.20	26.64	27.06	27.46	22.92	22.90	22.88	22.87	26.83	27.13
SRAT TiO_2 (wt.%)	0.03	6.50	7.41	8.28	9.12	5.68	6.37	7.00	7.59	12.31	13.29
Number of EVs	8317	9408	9408	9408	9318	9048	9048	9048	9300	9836	9836
% WL, 24	highv(54%)					highv(0.17%)					
25	highv(46%)										
26	highv(42%)										
27	highv(39%)										
28	highv(37%)										
29	highv(35%)										
30	highv(33%)										
31	highv(31%)										
32	highv(27%)										
33	highv(24%)										
34	highv(16%)										
35	highv(7.9%)										
36	Cr_2O_3(47%) highv(1.4%) highv Cr_2O_3(0.48%)										
37	Cr_2O_3(48%)										
38	Cr_2O_3(49%) Cr_2O_3 Neph(0.28%) Neph(1.5%)					TL(0.01%)	TL(0.17%)	TL(0.32%)	TL(0.71%)		
39	Cr_2O_3(39%) Cr_2O_3 Neph(10%) Neph(14%)	Neph(7.2%)	Neph(7.2%)	Neph(7.0%)	Neph(6.6%)	TL(3.6%)	TL(4.9%)	TL(6.1%)	TL(7.0%)		
40	Cr_2O_3(13%) Cr_2O_3 Neph(35%) Neph(42%) TL Cr_2O_3(2.1%) TL Cr_2O_3 Neph(0.06%)	Cr_2O_3(34%) Cr_2O_3 Neph(14%) Neph(20%)	Cr_2O_3(40%) Cr_2O_3 Neph(8.1%) Neph(13%)	Neph(20%)	Neph(20%)	Cr_2O_3(38%) TL(0.01%) TL Cr_2O_3(11%)	Cr_2O_3(36%) TL(0.09%) TL Cr_2O_3(12%)	TL(13%)	TL(14%)	lowv(0.19%) Neph(8.5%)	lowv(7.8%) Neph(7.7%)

(green)	No limiting constraints	highv/lowv	high viscosity/low viscosity
(yellow)	Limiting constraint is viscosity.	Neph	nepheline
(red)	At least one of the limiting constraints is nepheline (durability impact).	TL	liquidus temperature
(orange)	Limiting constraint is TL.	Cr_2O_3	Occurs when Cr_2O_3 > 0.3 wt.% minus measurement uncertainty.
(dark red)	At least one of the limiting constraints is maximum Cr_2O_3.		

Table A-2. MAR Assessment Results for the 1.2M Na Wash Endpoint

Case	Sludge Only	Case 1 Single Strike - No Sludge Solids				Case 3 Single MST Strike - 600 mg/L Sludge Solids				Case 4 Double MST Strike - No Sludge Solids	
MST/SS Volume (gallons)	0	2400	2800	3200	3600	2400	2800	3200	3600	3800	4200
SRAT Al_2O_3 (wt.%)	32.42	28.09	27.62	27.16	26.72	27.83	27.38	26.97	26.59	25.70	25.24
SRAT Na_2O (wt.%)	27.92	29.95	30.28	30.60	30.90	26.45	26.30	26.17	26.04	30.19	30.40
SRAT TiO_2 (wt.%)	0.03	6.01	6.87	7.69	8.48	5.31	5.97	6.58	7.15	11.48	12.42
Number of EVs	8605	8958	8958	9570	9570	9408	9408	9408	9408	9570	9584
% WL, 24	highv(0.23%)										
25											
26											
27											
28											
29											
30											
31											
32											
33											
34											
35											
36											
37	Neph(1.1%)									lowv(0.06%)	lowv(3.6%)
38	Neph(13%)	Neph(5.0%)	Neph(4.0%)	Neph(5.7%)	lowv(1.8%) Neph(5.7%)					lowv(12%)	lowv(27%)
39	Cr_2O_3(12%) Cr_2O_3 Neph(36%) Neph(44%)	Neph(32%)	lowv(0.22%) Neph(20%)	lowv(7.5%) lowv Neph(0.41%) Neph(19%)	lowv(21%) lowv Neph(1.4%) Neph(18%)					lowv(34%) lowv Neph(0.31%) Neph(8.3%)	lowv(39%) lowv Neph(0.76%) Neph(7.2%)
40	Cr_2O_3(4.9%) Cr_2O_3 Neph(44%) Neph(49%)	lowv Neph(2.3%) Neph(88%)	lowv Neph(16%) Neph(74%)	lowv Neph(30%) Neph(58%)	lowv(0.90%) lowv Neph(38%) Neph(49%)	Neph(12%)	Neph(9.9%)	Neph(7.0%)	Neph(5.4%)	lowv(19%) lowv Neph(23%) Neph(15%)	lowv(22%) lowv Neph(25%) Neph(11%)

No limiting constraints
Limiting constraint is viscosity.
At least one of the limiting constraints is nepheline (durability impact).

highv/lowv	high viscosity/low viscosity
Neph	nepheline
TL	liquidus temperature
Cr_2O_3	Occurs when Cr_2O_3 > 0.3 wt.% minus measurement uncertainty.