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Selection of Glasses to Confirm the 0.65 Weight Percent Sulfate Solubility Limit for Sludge Batch 10

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EXECUTIVE SUMMARY

In preparation for Sludge Batch 10 (SB10) processing, projections of sulfate (SO_4^{2-}) in glass at 36% waste loading (WL) were calculated in May 2020 for Tank 40 blend projections representing 0.7M and 0.85M Na wash endpoints. The projected SO_4^{2-} concentrations for either sludge-only (SO) or coupled processing with the Salt Waste Processing Facility (SWPF) were either near or exceeded the current Sludge Batch 9 (SB9) limit of 0.65 weight percent (wt.%). Four nominal glass compositions were selected based on SO and coupled processing for the 0.85M Na wash endpoint Tank 40 blend projection to conduct an initial evaluation of the SB10 sulfate solubility behavior. A sulfate salt phase was absent from each of the prepared glasses, which provided preliminary results that supported the 0.65 wt.% SO_4^{2-} limit for SB10.

In May 2021, Savannah River Remediation (SRR) reprojected SB10 based on the analytical results from the Tank 51 qualification sample that was washed in the Savannah River National Laboratory (SRNL) Shielded Cells Facility. Calculation-based frit assessments were performed on Tank 40 blend projections for 0.9M and 1M Na wash endpoints using the DWPF Product Composition Control System (PCCS) glass property models and their associated Measurement Acceptance Region (MAR) constraints. Based on these results, SRR finalized the decision to proceed with the 1M Na wash endpoint and Frit 473. These 1M SO_4^{2-} projections are lower than the previous 0.85M Na wash endpoint SO_4^{2-} projections, but still near the limit of 0.65 wt.%. This report documents the development of a supplementary test matrix of nine glasses to confirm that the 0.65 wt.% SO_4^{2-} limit is applicable to the SB10 glass composition region defined by the 1M Na wash endpoint and Frit 473.

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
ID	identification
MAR	Measurement Acceptance Region
MST/SS	monosodium titanate/sludge solids
NGS	Next Generation Solvent
PCCS	Product Composition Control System
SB10	Sludge Batch 10
SB3	Sludge Batch 3
SB4	Sludge Batch 4
SB7b	Sludge Batch 7b
SB8	Sludge Batch 8
SB9	Sludge Batch 9
SE	strip effluent
SEFT	Strip Effluent Feed Tank
SME	Slurry Mix Evaporator
SMRF	Slurry-Fed Melt Rate Furnace
SO	sludge-only
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SSRT	Sludge Solids Receipt Tank
SWPF	Salt Waste Processing Facility
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
WL	waste loading
wt.%	weight percent

1.0 Introduction

To support initial operations at the Defense Waste Processing Facility (DWPF), the original sulfate (SO_4^{2-}) solubility limit for Product Composition Control System (PCCS) Slurry Mix Evaporator (SME) acceptability was defined at 0.4 weight percent (wt.%) in glass based on pilot-scale melter testing.[1,2] This limit signified that 0.4 wt.% SO_4^{2-} could be retained in the glass without the formation of a sulfate phase. The utilization of a 0.4 wt.% SO_4^{2-} limit in glass for SME acceptability was challenged for Sludge Batch 3 (SB3), which included a neptunium (Np)-based stream projected to contain a significant fraction of ferrous sulfamate.[3] Laboratory-scale crucible testing with both batch chemicals and simulated Sludge Receipt and Adjustment Tank (SRAT) product was performed, and a new PCCS SME acceptability limit for SO_4^{2-} was established at 0.6 wt.% for SB3, which was confirmed by supplementary Slurry-Fed Melt Rate Furnace (SMRF) testing with simulated SME product.[3] While 0.6 wt.% SO_4^{2-} was allowed in the melter feed, it was anticipated that less than 0.6 wt.% would be retained in the glass based on SO_4^{2-} volatility during DWPF melter processing, which provides some conservatism with respect to the formation of a sulfate phase. PCCS was not revised to reflect the updated SO_4^{2-} limit and since then DWPF has imposed this constraint administratively outside of PCCS.[2] The 0.6 wt.% SO_4^{2-} limit was confirmed for Sludge Batch 4 (SB4) through Sludge Batch 7b (SB7b) by laboratory-scale crucible testing with batch chemicals.[4-9] For Sludge Batch 8 (SB8) and Sludge Batch 9 (SB9), the limit was defined at 0.65 wt.%.[10-13]

In preparation for Sludge Batch 10 (SB10) processing, projected SO_4^{2-} concentrations in glass were calculated for Tank 40 blend projections representing 0.7M and 0.85M Na wash endpoints.[14,15] As shown in Table 1-1, the projected SO_4^{2-} concentrations in glass at 36% waste loading (WL) for either sludge-only (SO) or coupled processing with the Salt Waste Processing Facility (SWPF) are either near or exceed the current SB9 limit of 0.65 wt.%.

Table 1-1. May 2020 Projected SO_4^{2-} Concentrations in Glass at 36% WL for Sludge-Only and Coupled Processing

Na Wash Endpoint	0.7M	0.85M
Sludge-only Projection	0.69 wt.%	0.72 wt.%
Coupled Projection*	0.58 wt.%	0.61 wt.%

*As-received coupled projection from Sludge Batch Planning.

To support the decision for the Na wash endpoint at that time, Savannah River Remediation (SRR) requested that a small set of glasses be fabricated to conduct a preliminary evaluation of the SB10 sulfate solubility behavior.[16] Four nominal glass compositions were selected based on SO and coupled processing for the 0.85M Na wash endpoint Tank 40 blend projection. The waste loadings were allowed to vary from 31-40% to achieve SO_4^{2-} concentrations in glass that were at the current limit of 0.65 wt.%, and above and below this limit. A sulfate salt phase was absent from each of the prepared glasses, which provided preliminary results that supported the 0.65 wt.% SO_4^{2-} limit for SB10.[17]

In May 2021, SRR Sludge Batch Planning reprojected SB10 based on the analytical results from the Tank 51 qualification sample that was washed in the Savannah River National Laboratory (SRNL) Shielded Cells Facility.[18] Calculation-based frit assessments were performed on Tank 40 blend projections for 0.9M and 1M Na wash endpoints using the DWPF PCCS glass property models and their associated Measurement Acceptance Region (MAR) constraints.[19] Based on these results, SRR finalized the decision to proceed with the 1M Na wash endpoint and Frit 473. Table 1-2 presents the projected SO_4^{2-} concentrations in glass at 36% WL for SO and coupled processing based on the Tank 40 blend projections for the 1M Na wash endpoint.[18] These SO_4^{2-} projections are lower than the 0.85M Na wash endpoint SO_4^{2-} projections in Table 1-1, but still near the limit of 0.65 wt.%.

Table 1-2. May 2021 Projected SO_4^{2-} Concentrations in Glass at 36% WL for Sludge-Only and Coupled Processing

Na Wash Endpoint	1M
Sludge-only Projection	0.59 wt.%
Coupled Projection*	0.57 wt.%

*As-received coupled projection from Sludge Batch Planning.

This report documents the development of a supplementary test matrix to confirm that the 0.65 wt.% SO_4^{2-} limit is applicable to the SB10 glass composition region defined by the 1M Na wash endpoint and Frit 473.

2.0 Quality Assurance

This work was requested via a Technical Task Request (TTR)[20] and directed by a Task Technical and Quality Assurance Plan (TTQAP).[21] The TTR indicated the portion of the work scope covered by this report (TTR Task 3) is classified as Safety Class and not subject to RW-0333P requirements. Microsoft Excel was used to support this work. Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.[22] This document, including calculations, was reviewed by a Design Verification. SRNL documents the Design Verification using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[23] The Design Checklist for this report is stored in electronic laboratory notebook experiment C7592-00311-39.

3.0 Glass Selection

A Tank 40 blend projection for the 1M Na wash endpoint for SO processing was received from SRR System Planning (on a calcine basis) in May 2021.[18] The elemental concentrations were converted to oxides and normalized to 100 weight percent (wt.%) as shown in Appendix Table A-1. Per a previous request from SRR,[24] SRNL also performed subsequent calculations with the SO projection in Appendix Table A-1 to estimate the composition in the SRAT during coupled operation with SWPF. These calculations involved developing compositions for strip effluent (SE) and the Sludge Solids Receipt Tank (SSRT) effluent stream, which consists of monosodium titanate and sludge solids (MST/SS). This evaluation focuses on the following case for the SSRT effluent stream:

- Case 1: Single MST strike operation with no entrained insoluble sludge solids. This case represents the baseline for coupled operation with SWPF.

Other pertinent inputs include:

- DWPF receives 5700 gallons of sludge slurry from Tank 40 per SRAT batch[25]
- Single strike operation results in 2800 gallons of the SSRT effluent stream (MST/SS) per SRAT batch[26]
- DWPF receives 15,000 of SE per SRAT batch (Next Generation Solvent (NGS)[a])[25,26]
- Cs-137 concentration in SE is 66 Ci/gallon[27]

The methodology used for these calculations was originally developed for SB9.[26,28-30] The nominal SRAT composition representing the coupled operation scenario for Case 1 is shown in Appendix Table A-2. A second SRAT composition was developed for an SSRT volume of 4200 gallons (Case 1A), which would allow for operational flexibility if necessary during SB10 processing.[31] As compared to the nominal Case 1, the TiO_2 concentration of Case 1A is increased by ~ 3 wt.% due to the increased concentration of MST. Using the SO, Case 1 and Case 1A compositions, the SO_4^{2-} concentrations were fixed such that the resulting SO_4^{2-} concentrations in glass would be 0.65 wt.% at 32%, 36% and 40% WL. Similar to previous sulfate solubility studies,[4,7-10,13] the SRAT compositions were renormalized without U_3O_8 and ThO_2 since these

[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.

components are not expected to have an impact on the sulfate solubility behavior. Glass compositions were developed by combining the renormalized SRAT compositions for SO, Case 1 and Case 1A with Frit 473 ($8B_2O_3$-$8Li_2O$-$5Na_2O$-$79SiO_2$, wt.%) at 32%, 36% and 40% WL. Target compositions for the nine recommended glass compositions are shown in Appendix Table A-3. These compositions provide supplementary compositional variation to the preliminary four glasses that were previously evaluated for the 0.85M Na wash endpoint.[16,17]

4.0 Recommendation

A supplementary test matrix of nine glasses is recommended to confirm that the 0.65 wt.% SO_4^{2-} limit is applicable to the SB10 glass region defined by the 1M Na wash endpoint and Frit 473. The prepared glasses will be visually inspected for the presence of a yellow sulfate salt phase.

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Appendix A. Composition Information

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

Na Wash Endpoint	**1M**
Projection	**SO**
Al_2O_3	29.72
B_2O_3	0.03
BaO	0.08
CaO	1.22
Ce_2O_3	0.19
Cr_2O_3	0.30
CuO	0.06
Fe_2O_3	19.64
K_2O	0.11
La_2O_3	0.04
Li_2O	0.05
MgO	0.48
MnO	5.61
Na_2O	31.83
NiO	0.74
PbO	0.03
SO_4^{2-}	1.64
SiO_2	1.72
ThO_2	2.45
TiO_2	0.03
U_3O_8	3.82
ZnO	0.03
ZrO_2	0.18

Table A-2. SRNL-Developed SRAT Compositions for Coupled Operation for the 1M Na Wash Endpoint

Case	Case 1 Single Strike	Case 1A Single Strike
Tank 40 Volume (gal)	5700	5700
SSRT Volume (gal)	2800	4200
SE Volume[a] (gal)	15,000	15,000
Al_2O_3	24.42	22.89
B_2O_3	0.86	0.80
BaO	0.06	0.06
CaO	0.97	0.89
Ce_2O_3	0.15	0.14
Cr_2O_3	0.23	0.22
Cs_2O	2.05	1.89
CuO	0.05	0.04
Fe_2O_3	15.61	14.40
K_2O	0.31	0.33
La_2O_3	0.03	0.03
Li_2O	0.04	0.04
MgO	0.38	0.35
MnO	4.46	4.11
Na_2O	33.77	34.74
NiO	0.59	0.54
PbO	0.02	0.02
SO_4^{2-}	1.34	1.26
SiO_2	1.37	1.26
ThO_2	1.95	1.80
TiO_2	8.12	11.23
U_3O_8	3.04	2.80
ZnO	0.03	0.03
ZrO_2	0.14	0.13

[a] Under typical processing conditions, 15,000 gallons represents 2 transfers from the Strip Effluent Feed Tank (SEFT), whereas 22,000 gallons represents 3 transfers from the SEFT and allows for flexibility if a processing upset occurs. The compositional difference on a glass basis between 15,000 and 22,000 gallons of SE is minimal, which results in little or no difference in the operating windows when evaluating 15,000 versus 22,000 gallons of SE with MAR assessments.[19] Thus, for the purpose of the sulfate solubility evaluation, 15,000 gallons is a representative volume compositionally.

Table A-3. Targeted Glass Compositions

Glass ID	SB10S-05	SB10S-06	SB10S-07	SB10S-08	SB10S-09	SB10S-10	SB10S-11	SB10S-12	SB10S-13
Case	SO	Coupled Case 1	Coupled Case 1A	SO	Coupled Case 1	Coupled Case 1A	SO	Coupled Case 1	Coupled Case 1A
WL	32%	32%	32%	36%	36%	36%	40%	40%	40%
Al_2O_3	10.117	8.172	7.623	11.408	9.215	8.596	12.699	10.257	9.568
B_2O_3	5.452	5.729	5.705	5.133	5.446	5.419	4.815	5.163	5.133
BaO	0.027	0.021	0.019	0.030	0.023	0.022	0.033	0.026	0.024
CaO	0.414	0.323	0.297	0.466	0.364	0.335	0.519	0.406	0.372
Ce_2O_3	0.063	0.049	0.045	0.071	0.056	0.051	0.079	0.062	0.057
Cr_2O_3	0.101	0.079	0.072	0.113	0.089	0.081	0.126	0.099	0.090
Cs_2O	0.000	0.687	0.631	0.000	0.774	0.711	0.000	0.862	0.792
CuO	0.020	0.016	0.015	0.023	0.018	0.016	0.025	0.020	0.018
Fe_2O_3	6.685	5.223	4.794	7.538	5.890	5.406	8.391	6.556	6.017
K_2O	0.038	0.105	0.109	0.043	0.118	0.123	0.048	0.132	0.137
La_2O_3	0.014	0.011	0.010	0.016	0.012	0.011	0.018	0.014	0.013
Li_2O	5.457	5.453	5.452	5.139	5.135	5.133	4.821	4.816	4.815
MgO	0.164	0.128	0.118	0.185	0.144	0.133	0.206	0.161	0.148
MnO	1.910	1.492	1.370	2.154	1.683	1.545	2.397	1.873	1.719
Na_2O	14.235	14.702	14.969	15.418	15.944	16.245	16.600	17.186	17.521
NiO	0.253	0.198	0.181	0.285	0.223	0.204	0.317	0.248	0.228
PbO	0.011	0.008	0.008	0.012	0.009	0.009	0.013	0.010	0.010
SO_4^{2-}	0.650	0.650	0.650	0.650	0.650	0.650	0.650	0.650	0.650
SiO_2	54.306	54.178	54.140	51.221	51.077	51.034	48.136	47.975	47.928
TiO_2	0.010	2.719	3.739	0.012	3.066	4.216	0.013	3.412	4.693
ZnO	0.012	0.009	0.008	0.013	0.010	0.009	0.015	0.011	0.011
ZrO_2	0.062	0.048	0.044	0.070	0.055	0.050	0.078	0.061	0.056

Note that glass identification (ID) numbering is continued from the preliminary evaluation.[16,17]

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