

Contract No:

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Evaluation of the 0.65 Weight Percent Sulfate Solubility Limit for Sludge Batch 10 Based on November 2022 Projections

F.C. Johnson

January 2023

SRNL-STI-2023-00013, Revision 0

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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *SB10, sulfate solubility, glass, Frit 473, Frit 625*

Retention: *Lifetime*

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Savannah River National Laboratory is operated by
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EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) is currently preparing to initiate processing of Sludge Batch 10 (SB10), which is comprised of material from Tanks 11H, 13H, 15H, 26F, 40H (heel only), and Alternate Feed Stock-2 and Sodium Reactor Experiment material from H-Canyon. Frit 473 was recommended for sludge-only (SO) and coupled processing with the Salt Waste Processing Facility (SWPF) based on previous assessments of SB10 projections with the DWPF Product Composition Control System glass property models and their associated Measurement Acceptance Region constraints. Savannah River Mission Completion (SRMC) subsequently pursued Wash Cycle Y to further reduce the total sulfur in the sludge batch and increase processing flexibility at DWPF. In October 2022, SRMC System Planning provided an updated SB10 Tank 40H blend baseline projection based on the Tank 51 confirmation sample results. Due to the reduced Na concentration relative to previous SB10 projections, two additional projections based on the addition of 7,000 gallons of caustic were provided in November 2022.

Previous laboratory-scale crucible testing with batch chemicals confirmed that the sulfate (SO_4^{2-}) limit for SB10 was 0.65 weight percent (wt.%) in glass. This limit signifies that 0.65 wt.% SO_4^{2-} can be retained in the glass without the formation of a sulfate phase. Since this work was conducted in 2021 and 2022, the objective of this evaluation is to determine whether the 0.65 wt.% SO_4^{2-} limit is still appropriate for the SB10 reprojections received in November 2022.

The reprojected SB10 glass composition region is based on Frit 625 and Frit 473 at 32-40% waste loading for SO and single strike coupled operation, which generally overlaps those regions studied in previous SB10 and Sludge Batch 6 sulfate solubility studies. The minor shift of the reprojected SB10 projection will not adversely influence the experimentally observed sulfate solubility behavior. It is recommended that DWPF continue using the 0.65 wt.% SO_4^{2-} limit in glass for SB10 processing.

A task is currently underway to determine whether a sulfate solubility limit greater than 0.65 wt.% is feasible for SB10, which could allow for higher transfer volumes of the monosodium titanate and sludge solids stream from SWPF. These results will be documented in a subsequent report.

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
MST/SS	monosodium titanate/sludge solids
SB10	Sludge Batch 10
SB6	Sludge Batch 6
SB9	Sludge Batch 9
SE	strip effluent
SO	sludge-only
SRAT	Sludge Receipt and Adjustment Tank
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
SWPF	Salt Waste Processing Facility
WL	waste loading
wt.%	weight percent

1.0 Introduction

The Defense Waste Processing Facility (DWPF) is currently preparing to initiate processing of Sludge Batch 10 (SB10), which is comprised of material from Tanks 11H, 13H, 15H, 26F, 40H (heel only), and Alternate Feed Stock-2 and Sodium Reactor Experiment material from H-Canyon. Frit 473^a was recommended for sludge-only (SO) and coupled processing with the Salt Waste Processing Facility (SWPF) based on previous assessments of SB10 projections with the DWPF Product Composition Control System glass property models and their associated Measurement Acceptance Region constraints.^{1,2} Savannah River Mission Completion (SRMC) subsequently pursued Wash Cycle Y to further reduce the total sulfur in the sludge batch and increase processing flexibility at DWPF.³ In October 2022, SRMC System Planning provided an updated SB10 Tank 40H blend baseline projection based on the Tank 51 confirmation sample results.^{4,5} Due to the reduced Na concentration relative to previous SB10 projections, two additional projections based on the addition of 7,000 gallons of caustic were provided in November 2022.⁶

Previous laboratory-scale crucible testing with batch chemicals confirmed that the sulfate (SO_4^{2-}) limit for SB10 was 0.65 weight percent (wt.%) in glass.⁷⁻¹⁰ This limit signifies that 0.65 wt.% SO_4^{2-} can be retained in the glass without the formation of a sulfate phase. Since this work was conducted in 2021 and 2022, the objective of this evaluation is to determine whether the 0.65 wt.% SO_4^{2-} limit is still appropriate for the SB10 reprojections received in November 2022.

2.0 Quality Assurance

This work was requested via a Technical Task Request and directed by a Task Technical and Quality Assurance Plan.^{11,12} The functional classification of this task is Safety Class. Microsoft Excel and JMP Version 16.0.0 were used to support this work.^{13,14} Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.¹⁵ This document, including calculations, was reviewed by a Design Verification. The Savannah River National Laboratory (SRNL) documents the Design Verification using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.¹⁶ The Design Checklists for this report are stored in electronic laboratory notebook experiment C7592-00599-01.

3.0 Inputs and Assumptions

The elemental concentrations of each SO Tank 40H blend projection provided by SRMC System Planning (on a calcine basis) were converted to oxides and normalized to 100 wt.%. Two Tank 40H blend projections with 7,000 gallons of added caustic^b are shown in Appendix A Table A-1; one with a Tank 51 heel of 10 inches and the other with a heel of 30 inches.^{5,6}

3.1.1 SRNL-Developed Inputs for Coupled Processing with SWPF

SRNL performed subsequent calculations with the SO projections in Appendix A Table A-1 to estimate the composition in the Sludge Receipt and Adjustment Tank (SRAT) during coupled operation with SWPF. These calculations were based on projected compositions for strip effluent (SE) and the monosodium titanate and sludge solids (MST/SS) in the Sludge Solids Receipt Tank effluent that were originally developed for Sludge Batch 9 (SB9).¹⁷⁻²⁰ This evaluation focuses on the following cases for coupled operation:

- Case 1: Single strike operation with no entrained insoluble sludge solids. This case represents the baseline.
- Sludge and SE

^a 8B₂O₃-8Li₂O-5Na₂O-79SiO₂ in weight percent.

^b 50 weight percent NaOH.

Other pertinent inputs include:

- DWPF receives 5700 gallons of sludge slurry from Tank 40H per SRAT batch²¹
- Nominal single strike operation results in 2800 gallons of the MST/SS stream¹⁸
 - Incremental variations in these transfer volumes were initially requested for SB9,¹⁸ which SRNL assumed to be ± 400 gallons for these assessments; however, DWPF also requested that 4500 gallons be evaluated for single strike operation.²²
- 0.4 g/L of MST for a single strike¹⁸
- DWPF receives 15,000 gallons of SE per SRAT batch (Next Generation Solvent)^{e,18,21}
- Cs-137 concentration in SE is 66 Ci/gallon²³

The nominal SRAT compositions representing the coupled operation scenarios are shown in Appendix Table A-2. Due to the glass composition similarities, SE based on BOBCalixC6 solvent^d was not evaluated.

3.2 Development of the Reprojected SB10 Glass Composition Region

The reprojected SB10 glass composition region is based on Frit 625 and Frit 473 at 32-40% waste loading (WL) for the SO and coupled operation SRAT compositions shown in Appendix A Table A-1 and Table A-2. The SRAT compositions were renormalized without ThO₂ and U₃O₈ since these components are not expected to have an impact on the sulfate solubility behavior and were not included in the glasses used in the previous SB10 sulfate solubility experiments.⁷⁻¹⁰ Frit 625^e was evaluated for use during the SB9 to SB10 transition. The overall minimum and maximum concentrations of the glass oxides were determined.

4.0 Results and Discussion

Table 4-1 presents a comparison of the minimum and maximum nominal oxide concentrations of the reprojected SB10 glass composition region described in Section 3.2 and the glasses tested for the SB10 sulfate solubility studies. A difference is shown when the SB10 reprojected oxide concentration falls outside of the oxide interval evaluated for the SB10 sulfate solubility studies.⁷⁻¹⁰ Note that the differences were calculated using more significant figures than shown and were rounded to one decimal place. The oxide intervals for the reprojected SB10 glass region are generally within those oxide intervals evaluated in the SB10 sulfate solubility studies. The difference is generally less than 0.5 wt.% for a given oxide, except for the following:

- Li₂O is 0.6 wt.% below the lowest SB10 sulfate solubility study Li₂O concentration
- Fe₂O₃ is 1.1 wt.% above the highest SB10 sulfate solubility study Fe₂O₃ concentration

The reduced Li₂O concentration is a result of compositions based on Frit 625 at 40% WL and causes a minor shift in composition, which is not expected to impact the current sulfate solubility limit of 0.65 wt.%. The increased Fe₂O₃ concentration is a result of SO compositions at 40% WL. Previous work has shown that Fe₂O₃ increases sulfate solubility;^{24,25} however, more recent DWPF sludge batch data was of interest to further strengthen this justification. To determine whether glass compositions from previous sulfate solubility studies are already within the SB10 glass composition region of interest, compositions were compiled from Sludge Batch 2 through SB9.²⁶⁻³⁴ This data review focused on the SO glass compositions based on Appendix A Table A-1 at 40% WL with both Frit 473 and Frit 625. To enhance the potential for identifying glasses, the intervals for the major oxides (concentrations greater than 0.5 wt.%) were expanded by ± 1 wt.%, except for SiO₂, which was expanded by ± 2 wt.% since it has the highest concentration in glass (Appendix A Table A-3).

^c Next Generation Solvent 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.

^d BOBCalixC6 is calix[4]arene-bis(tert-octylbenzo-crown-6), which uses a nitric acid strip solution.

^e 1Al₂O₃-8B₂O₃-7Li₂O-6Na₂O-78SiO₂ in weight percent.

Ten Sludge Batch 6 (SB6) sulfate solubility study glasses were found to have compositions that simultaneously satisfy the oxide intervals of the search criteria.²⁹ Glasses that had a sulfate concentration less than 0.65 wt.% or formed a sulfate phase were excluded from further evaluation. Two compositions, LAL-15 and LFL-05, remained after applying these additional screening parameters. These compositions were incorporated into the “SB10 Sulfate Study Glasses” intervals of Table 4-1. As shown in Table 4-2, the reprojected SB10 Fe₂O₃ is within the revised interval that includes the two SB6 glass compositions. Based on these results, the increased Fe₂O₃ concentration is not expected to impact the current sulfate solubility limit of 0.65 wt.%.

Table 4-1. Comparison of the Oxide Intervals (wt.%) for the Reprojected SB10 Glass Region and the SB10 Sulfate Study Glasses

Oxide	Reprojected SB10 Glass Composition Region		SB10 Sulfate Study Glasses		Difference*	
	minimum	maximum	minimum	maximum	minimum	maximum
Al ₂ O ₃	7.25	12.60	7.62	12.70	-0.4	----
B ₂ O ₃	4.82	5.76	4.81	6.32	----	----
BaO	0.02	0.04	0.02	0.03	----	----
CaO	0.34	0.58	0.30	0.52	----	0.1
Ce ₂ O ₃	0.06	0.10	0.05	0.08	----	----
Cr ₂ O ₃	0.06	0.10	0.07	0.18	----	----
Cs ₂ O	0.00	0.91	0.00	0.86	----	0.1
CuO	0.02	0.03	0.01	0.03	----	----
Fe ₂ O ₃	5.55	9.50	4.79	8.39	----	1.1
Gd ₂ O ₃	0.02	0.04	0.00	0.00	----	----
K ₂ O	0.06	0.15	0.03	0.14	----	----
La ₂ O ₃	0.02	0.03	0.01	0.02	----	----
Li ₂ O	4.23	5.47	4.82	5.54	-0.6	----
MgO	0.12	0.21	0.12	0.21	----	----
MnO	1.56	2.68	1.37	2.40	----	0.3
Na ₂ O	13.61	17.79	13.60	17.52	----	0.3
NiO	0.23	0.40	0.18	0.32	----	0.1
PbO	0.01	0.02	0.01	0.02	----	----
SO ₄ ²⁻	0.24	0.37	0.61	0.71	-0.4	----
SiO ₂	47.43	54.42	47.40	54.38	----	----
TiO ₂	0.01	4.47	0.01	4.69	----	----
ZnO	0.01	0.02	0.01	0.01	----	----
ZrO ₂	0.02	0.04	0.04	0.09	----	----

* Differences were calculated using more significant figures than shown and were rounded to one decimal place, which may result in slight variation due to rounding.

Table 4-2. Comparison of the Oxide Intervals (wt.%) for the Reprojected SB10 Glass Region and the SB6 and SB10 Sulfate Study Glasses

Oxide	Reprojected SB10 Glass Composition Region		SB6 & SB10 Sulfate Study Glasses		Difference*	
	minimum	maximum	minimum	maximum	minimum	maximum
Al ₂ O ₃	7.25	12.60	7.62	12.70	-0.4	----
B ₂ O ₃	4.82	5.76	4.80	6.32	----	----
BaO	0.02	0.04	0.02	0.06	----	----
CaO	0.34	0.58	0.30	0.58	----	----
CdO	0.00	0.00	0.00	0.01	----	----
Ce ₂ O ₃	0.06	0.10	0.05	0.09	----	----
Cr ₂ O ₃	0.06	0.10	0.05	0.18	----	----
Cs ₂ O	0.00	0.91	0.00	0.86	----	0.1
CuO	0.02	0.03	0.01	0.05	----	----
Fe ₂ O ₃	5.55	9.50	4.79	10.23	----	----
Gd ₂ O ₃	0.02	0.04	0.00	0.05	----	----
K ₂ O	0.06	0.15	0.03	0.14	----	----
La ₂ O ₃	0.02	0.03	0.01	0.05	----	----
Li ₂ O	4.23	5.47	4.80	5.54	-0.6	----
MgO	0.12	0.21	0.12	0.32	----	----
MnO	1.56	2.68	1.37	2.92	----	----
Na ₂ O	13.61	17.79	13.60	17.52	----	0.3
NiO	0.23	0.40	0.18	1.25	----	----
P ₂ O ₅	0.00	0.00	0.00	0.19	----	----
PbO	0.01	0.02	0.01	0.02	----	----
SO ₄ ²⁻	0.24	0.37	0.61	0.75	-0.4	----
SiO ₂	47.43	54.42	46.52	54.38	----	----
SrO	0.00	0.00	0.00	0.03	----	----
TiO ₂	0.01	4.47	0.01	4.69	----	----
ZnO	0.01	0.02	0.01	0.03	----	----
ZrO ₂	0.02	0.04	0.04	0.10	----	----

* Differences were calculated using more significant figures than shown and were rounded to one decimal place, which may result in slight variation due to rounding.

5.0 Conclusions and Recommendations

The reprojected SB10 glass composition region is based on Frit 625 and Frit 473 at 32-40% for SO and single strike coupled operation, which generally overlaps those regions studied in previous SB10 and SB6 sulfate solubility studies. The minor shift of the reprojected SB10 projection will not adversely influence the experimentally observed sulfate solubility behavior. It is recommended that DWPF continue using the 0.65 wt.% SO₄²⁻ limit in glass for SB10 processing.

6.0 Future Work

A task is currently underway to determine whether a sulfate solubility limit greater than 0.65 wt.% is feasible for SB10, which could allow for higher transfer volumes of the MST/SS stream from SWPF.^{35,36} These results will be documented in a subsequent report.

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Appendix A. Supplementary Information

Table A-1. Normalized SB10 Confirmation Sample Tank 40H Blend Projections (wt.%)

Projection	7000 gal Caustic 10" Heel November 2022	7000 gal Caustic 30" Heel November 2022
Al ₂ O ₃	28.10	27.42
B ₂ O ₃	0.05	0.05
BaO	0.09	0.09
CaO	1.33	1.35
Ce ₂ O ₃	0.24	0.24
Cr ₂ O ₃	0.23	0.23
CuO	0.07	0.07
Fe ₂ O ₃	22.03	22.27
Gd ₂ O ₃	0.09	0.09
K ₂ O	0.17	0.17
La ₂ O ₃	0.06	0.06
Li ₂ O	0.09	0.09
MgO	0.49	0.48
MnO	6.20	6.29
Na ₂ O	30.43	30.74
NiO	0.90	0.93
PbO	0.05	0.05
SO ₄ ²⁻	0.88	0.88
SiO ₂	2.00	2.06
ThO ₂	2.26	2.21
TiO ₂	0.04	0.04
U ₃ O ₈	4.08	4.08
ZnO	0.04	0.04
ZrO ₂	0.09	0.09

Table A-2. SRNL-Developed SRAT Compositions for Coupled Operation (wt.%)

Case	Sludge and SE 10" Heel	Sludge and SE 30" Heel	Case 1 Single Strike 10" Heel	Case 1A Single Strike 30" Heel
Tank 40 Volume (gal)	5700	5700	5700	5700
MST/SS Volume (gal)	0	0	4500	4500
SE Volume (gal)	15,000	15,000	15,000	15,000
Al ₂ O ₃	27.01	26.35	22.10	21.59
B ₂ O ₃	0.92	0.93	0.72	0.72
BaO	0.08	0.08	0.07	0.07
CaO	1.28	1.30	1.00	1.01
Ce ₂ O ₃	0.23	0.23	0.18	0.18
Cr ₂ O ₃	0.22	0.22	0.17	0.17
Cs ₂ O	2.14	2.14	1.68	1.67
CuO	0.07	0.07	0.05	0.05
Fe ₂ O ₃	21.17	21.40	16.51	16.69
Gd ₂ O ₃	0.09	0.09	0.07	0.07
K ₂ O	0.31	0.30	0.35	0.35
La ₂ O ₃	0.06	0.06	0.05	0.05
Li ₂ O	0.08	0.08	0.07	0.07
MgO	0.47	0.47	0.37	0.36
MnO	5.96	6.04	4.65	4.71
Na ₂ O	29.96	30.27	33.57	33.80
NiO	0.86	0.89	0.67	0.70
PbO	0.05	0.05	0.04	0.04
SO ₄ ²⁻	0.84	0.84	0.71	0.71
SiO ₂	1.92	1.98	1.50	1.54
ThO ₂	2.17	2.12	1.70	1.66
TiO ₂	0.03	0.03	10.64	10.64
U ₃ O ₈	3.92	3.92	3.06	3.06
ZnO	0.04	0.04	0.03	0.03
ZrO ₂	0.08	0.08	0.07	0.06

Table A-3. Nominal and Expanded Oxide Intervals for the SB10 SO Glass Composition Region at 40% WL (wt.%)

Oxide	SB10 SO Glass Composition Region (40% WL)		Search Criteria	
	minimum	maximum	minimum	maximum
Al ₂ O ₃	11.70	12.60	10.70	13.60
B ₂ O ₃	4.82	4.82	3.82	5.82
CaO	0.57	0.58	0	1.58
Fe ₂ O ₃	9.41	9.50	8.41	10.50
Li ₂ O	4.24	4.84	3.24	5.84
MnO	2.65	2.68	1.65	3.68
Na ₂ O	16.00	16.72	15.00	17.72
SiO ₂	47.65	48.28	45.65	50.28