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The Impact of the MCU Life Extension Solvent on Sludge Batch 8 Projected Operating Windows

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EXECUTIVE SUMMARY

As a part of the Actinide Removal Process (ARP)/Modular Caustic Side Solvent Extraction Unit (MCU) Life Extension Project, a next generation solvent (NGS) and a new strip acid will be deployed. The strip acid will be changed from dilute nitric acid to dilute boric acid (0.01 M). Because of these changes, experimental testing or evaluations with the next generation solvent are required to determine the impact of these changes (if any) to Chemical Process Cell (CPC) activities, glass formulation strategies, and melter operations at the Defense Waste Processing Facility (DWPF).

The introduction of the dilute (0.01M) boric acid stream into the DWPF flowsheet has a potential impact on glass formulation and frit development efforts since B_2O_3 is a major oxide in frits developed for DWPF. Prior knowledge of this stream can be accounted for during frit development efforts but that was not the case for Sludge Batch 8 (SB8). Frit 803 has already been recommended and procured for SB8 processing; altering the frit to account for the incoming boron from the strip effluent (SE) is not an option for SB8. Therefore, the operational robustness of Frit 803 to the introduction of SE including its compositional tolerances (i.e., up to 0.0125M boric acid) is of interest and was the focus of this study. The primary question to be addressed in the current study was: What is the impact (if any) on the projected operating windows for the Frit 803 – SB8 flowsheet to additions of B_2O_3 from the SE in the Sludge Receipt and Adjustment Tank (SRAT)? More specifically, will Frit 803 be robust to the potential compositional changes occurring in the SRAT due to sludge variation, varying additions of ARP and/or the introduction of SE by providing access to waste loadings (WLs) of interest to DWPF?

The Measurement Acceptability Region (MAR) results indicate there is very little, if any, impact on the projected operating windows for the Frit 803 – SB8 system regardless of the presence or absence of ARP and SE (up to 2 wt% B_2O_3 contained in the SRAT and up to 2000 gallons of ARP). It should be noted that 0.95 wt% B_2O_3 is the nominal projected concentration in the SRAT based on a 0.0125M boric acid flowsheet with 70,000 liters of SE being added to the SRAT.

The impact on CPC processing of a 0.01M boric acid solution for elution of cesium during Modular Caustic Side Solvent Extraction Unit (MCU) processing has previously been evaluated by the Savannah River National Laboratory (SRNL). Increasing the acid strength to 0.0125M boric acid to account for variations in the boric acid strength has been reviewed versus the previous evaluation. The amount of acid from the boric acid represented approximately 5% of the total acid during the previous evaluation. An increase from 0.01 to 0.0125M boric acid represents a change of approximately 1.3% which is well within the error of the acid calculation. Therefore, no significant changes to CPC processing (hydrogen generation, metal solubilities, rheological properties, REDOX control, etc.) are expected from an increase in allowable boric acid concentration from 0.01M to 0.0125M.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CPC	Chemical Process Cell
DWPF	Defense Waste Processing Facility
EVs	Extreme Vertices
MAR	Measurement Acceptability Region
MCU	Modular Caustic Side Solvent Extraction Unit
NGS	Next Generation Solvent
PCCS	Product Composition Control System
PRFT	Precipitate Reactor Feed Tank
SB	Sludge Batch
SE	Strip Effluent
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
T_L	Liquidus Temperature
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance Plan
η	Viscosity
WL	Waste Loading

1.0 Introduction

The Actinide Removal Process (ARP)/Modular Caustic Side Solvent Extraction Unit (MCU) Life Extension includes activities required to support ARP/MCU extended operations to treat dissolved salt cake waste (i.e., remove actinides, strontium, and cesium) and deliver a low-activity decontaminated salt solution waste stream to the Saltstone Processing Facility (SPF). The resulting cesium and actinide/strontium salt stream is processed in the Defense Waste Processing Facility (DWPF). As a part of the ARP/MCU Life Extension Project, a next generation solvent (NGS) and a new strip acid will be deployed. The strip acid will be changed from dilute nitric acid to dilute boric acid (0.01 M). Because of these changes, experimental testing with the next generation solvent is required to determine the impact of these changes (if any) to Chemical Process Cell (CPC) activities, glass formulation strategies, and melter operations at the DWPF.

Bricker issued a Technical Task Request (TTR) to support the assessments of the impact of the next generation solvent on the downstream DWPF flowsheet unit operations (i.e., CPC, glass formulation, and melter operations).¹ Newell and Peeler issued a Task Technical and Quality Assurance Plan (TTQAP) in response to the TTR which outlined the technical approach to be used to meet programmatic objectives.²

The downstream impacts of the boric acid strip effluent (SE) to the Chemical Process Cell (CPC) processing have been previously evaluated. No impacts were noted for the CPC processing using boric acid on hydrogen generation, rheological properties, metal solubilities, foaming, or other processing parameters. The downstream impact of the boric acid SE on glass formulation activities and melter operations using the baseline flowsheet (0.01M or 10mM boric acid concentration) have also previously been evaluated.³ The results of that paper study assessment indicated that Frit 418 was robust to the implementation of the baseline 0.01M boric acid SE into the Sludge Batch 7b (SB7b) flowsheet (sludge-only or ARP-added)^a. More specifically, the projected operating windows for the nominal SB7b projections remained essentially constant (i.e., 25-43 or 25-44% waste loading (WL)) regardless of the flowsheet options (sludge-only, ARP added, and/or the presence of the SE). These results indicated that even if SE is not transferred to the Sludge Receipt and Adjustment Tank (SRAT), there would be no need to add boric acid (from a trim tank) to compositionally compensate for the absence of the boric acid SE in either a sludge-only or ARP-added SB7b flowsheet.

Since that assessment, the specifications of the incoming boric acid have been proposed (or defined) as 0.01M \pm 0.0025M. In addition, DWPF has completed processing of SB7b and is currently processing Sludge Batch 8 (SB8). Therefore, a second request has been made for SRNL to assess the impact of imposing the boric acid molarity specifications on the SB8 flowsheet.^b It should be noted that Peeler and Edwards recommended Frit 803 for SB8 processing and thus its composition will be used to support this assessment.⁴ The compositional bases for this assessment (sludge, ARP, and frit) will be discussed in detail in Section 4.0.

^a In the 2011 assessment, introduction of the Actinide Removal Process (ARP) stream was based on Appendix J from S.G. Subosits, "Actinide Removal Process Material Balance Calculation with Low Curie Salt Feed," *X-CLC-S-00113 Rev 0*, Appendix J, September 24, 2004. To support the current assessment, the measured composition reported by DWPF of the Precipitate Reactor Feed Tank (PRFT) was used.

^b Personal communication from A. Samadi to D. Peeler, June 6, 2013 via email.

As outlined in the TTQAP, the introduction of the dilute (0.01M) boric acid stream into the DWPF flowsheet has a potential impact on glass formulation and frit development efforts since B_2O_3 is a major oxide in frits developed for DWPF. Introduction of the boric acid in an upstream unit operation may require compositional adjustments to the frit to ensure both process and product performance properties are maintained during production. Given Frit 803 has already been recommended and procured for SB8 processing, the option of altering the frit to account for the incoming boron from the SE is not a preferred option. However, the response of Frit 803 to the introduction of the baseline SE including its compositional tolerances (i.e., up to 0.0125M boric acid) is of interest and the focus of this study. Therefore, the primary question to be addressed in the current study for impact of the boric acid on glass formulation and melter processing is: What is the impact (if any) on the projected operating windows for the Frit 803 – SB8 flowsheet to additions of B_2O_3 from the SE in the SRAT? More specifically, will Frit 803 be robust to the potential compositional changes occurring in the SRAT due to sludge variation, varying additions of ARP and/or the introduction of SE by providing access to waste loadings of interest to DWPF? For the CPC evaluation, the primary question is whether the previous testing remains valid at the revised boric acid concentration.

To assess the impact of the introduction of the new SE on SB8 Slurry Mix Evaporator (SME) acceptability decisions, Variation Stage Measurement Acceptability Region (MAR) assessments developed by SRNL will be used.⁵

2.0 Objective

The objective of this report is to provide supplemental information on the downstream impacts of the new SE (0.01M boric acid) on the projected operating windows for the Frit 803-SB8 flowsheet given implementation of the new SE (0.0125M accounting for the proposed compositional specifications or tolerances). Peeler and Edwards provided an initial assessment on the impact of the 0.01M boric acid flowsheet.³ Newell, et al., addressed the impacts to the CPC processing.⁶

3.0 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

4.0 Compositional Information

Given the introduction of the new SE is anticipated to occur during SB8 processing, the latest projections of SB8 were used to support this assessment. Table 4-1 summarizes the SB8 projection (sludge-only) from Savannah River Remediation (SRR) received on April 30, 2013.^a Table 4-1 also shows the addition of the ARP stream at two volumes (1000 and 2000 gallons) to the nominal sludge-only projection.^b It should be noted that the MAR assessment performed on the coupled operations flowsheet used increments of 250 gallons of ARP product addition from 0 to 2000 gallons. Only the 1000 and 2000 gallons projections are shown in Table 4-1. As

^a SB8 projections were received via email from D.W. McIlmoyle to D.K. Peeler on 4-30-13 (see SRNL-NB-2012-00070, page 123 for more details).

^b Nominal coupled operations projections are based on the measured PRFT materials as reported by DWPF.

previously mentioned, Peeler and Edwards recommended Frit 803, whose nominal composition is provided in Table 4-2, for processing SB8.⁴

Table 4-1. SB8 Nominal Projections (Sludge-Only, 1000- and 2000-Gallons of ARP Added) (wt%, calcined oxides).

Sludge ID	SB8 Sludge-Only	SB8 1000 Gallons of ARP	SB8 2000 Gallons of ARP
Al ₂ O ₃	17.923	16.901	16.009
BaO	0.129	0.120	0.112
CaO	2.003	1.874	1.761
Ce ₂ O ₃	0.337	0.315	0.295
Cr ₂ O ₃	0.152	0.147	0.144
CuO	0.056	0.054	0.051
Fe ₂ O ₃	31.575	29.464	27.621
K ₂ O	0.136	0.194	0.244
La ₂ O ₃	0.082	0.076	0.071
MgO	0.510	0.477	0.448
MnO	9.247	8.621	8.075
Na ₂ O	23.374	25.943	28.185
NiO	2.769	2.583	2.421
PbO	0.046	0.043	0.040
SO ₄	1.629	1.629	1.629
SiO ₂	2.905	2.814	2.735
ThO ₂	1.276	1.189	1.113
TiO ₂	0.033	2.107	3.918
U ₃ O ₈	5.591	5.234	4.923
ZnO	0.053	0.050	0.046
ZrO ₂	0.175	0.167	0.159

Table 4-2. Nominal Composition of Frit 803.

Oxide	Frit 803
B ₂ O ₃	8
Li ₂ O	6
Na ₂ O	8
SiO ₂	78

With respect to the contribution of boron oxide from the strip effluent, calculations were performed by SRNL for a 0.0125M boric acid ($0.01\text{M} \pm 0.0025\text{M}$ tolerance) upper bound as a function of the volume of SE added to the SRAT. The assumptions and inputs used by SRNL to perform these calculations are provided in Table 4-3 with the resulting output shown in Table 4-4.

Table 4-3. Assumptions Used To Calculate the Kg of Boron Added to the SRAT.

Amount of SRAT product transferred per batch	4,500	Gallons from SRAT
Amount of SRAT product transferred per batch	17,033	liters from SRAT
SRAT Product Density	1.25	kg/L
SRAT Nitric Acid Amount	50	gallons
SRAT Formic Acid Amount	350	gallons
Nitric Acid Concentration	10	Molar
Formic Acid Concentration	23.6	Molar
SRAT Product Calcine Solids	0.15	g oxide/g sludge
Waste Loading	42	%
Frit Boron Oxide Concentration	8	wt%
Boron Oxide Molecular Weight	69.62	g/mol
Boron Elemental Weight	10.811	g/mol
Amount of SRAT product transferred per batch	21,291	kg
SRAT Nitric Acid Amount	189	Liters
SRAT Formic Acid Amount	1,325	Liters
SRAT Nitric Acid Amount	1,893	Moles
SRAT Formic Acid Amount	31,264	Moles
Total Acid Amount	33,157	Moles
Amount of SRAT product oxides	3,194	kg sludge oxide
Amount of Glass Produced	7,604	kg glass

Table 4-4. Concentration of B₂O₃ Added to the SRAT as a Function of SE Volume Added.

Strip Effluent Impacts	0.0125 M Boric Acid				
	5,000	20,000	50,000	70,000	liters
Strip Effluent Added per SRAT Batch	5,000	20,000	50,000	70,000	liters
Strip Effluent Added per SRAT Batch	1321	5284	13210	18494	gallons
Amount of Boron in Strip Effluent	62.5	250	625	875	moles
Amount of Boron in Strip Effluent	0.7	2.7	6.8	9.5	kg
Percentage of Acid Added to SRAT in SE	0.6	2.3	5.7	7.9	%
<i>Amount of Boron Oxide per SRAT Batch</i>	<i>2.2</i>	<i>8.7</i>	<i>21.8</i>	<i>30.5</i>	<i>kg</i>
<i>Concentration of Boron Oxide in SRAT Product Calcined Solids</i>	<i>0.07</i>	<i>0.27</i>	<i>0.68</i>	<i>0.95</i>	<i>wt%</i>

Using a bounding 70,000 liters of SE added per SRAT batch and assuming a 0.0125M (or 12.5mM) SE boric acid concentration, 9.5 kg of boron (elemental) (or 30.5 kg of B₂O₃ on a calcined oxide basis) would be added to the SRAT. Therefore, the total mass of sludge oxides in the SRAT including the B₂O₃ contribution would increase to 3224.5 kg of calcined sludge oxides (3194 kg of sludge oxides + 30.5 kg of B₂O₃ from SE). The percent of boron in the SRAT can

then be calculated as $(30.5 \div 3224.5) * 100$ which yields 0.95 wt% (calcined solids) of B₂O₃ in the SRAT.

In the previous assessment,³ the B₂O₃ contribution was calculated to be 0.84 wt% for the nominal baseline flowsheet of 0.01M boric acid. It should be noted that the use of 70,000 liters of SE per SRAT could be considered a bounding case given current limitations of approximately 55,000 liters (or 15,000 gallons) to the SRAT. For example, if DWPF were to operate using 20,000 liters of SE per SRAT batch, then the B₂O₃ contribution to the SRAT would be 0.27 wt% - a factor of ~3X lower than that being carried forward in the assessment.

5.0 Results and Discussion

5.1 Chemical Processing Cell Evaluations

A review was conducted of the previous testing to determine if the change from a 0.010M boric acid concentration in the strip effluent to a 0.0125M concentration would have a noticeable impact on the CPC process. A review of the acid calculations from the test runs indicated that boric acid accounted for approximately 5% of the total acid added during the testing. A change from 0.010 to 0.0125M for the boric acid concentration reflects a 25% change in the amount of boric acid, but only a 1.3% change in the acid added during the tests. A change in acid concentration of this magnitude is insufficient to alter the outcome of the testing given the amount of uncertainty in the acid calculation. The previous test results are not invalidated by the change in boric acid concentration and additional testing or evaluations are not considered necessary to conclude that the boric acid will not impact CPC processing.

5.2 Variation Stage MAR Results

5.2.1 *MAR Results Without SE*

A Variation Stage assessment of the sludge projections of Table 4-1 (without boric acid) has been reported previously.^a That Variation Stage MAR assessment was following the same approach as described by Peeler and Edwards.⁵ Specifically, the standard variation approach was applied to each column of sludge projections in Table 4-1 (i.e., $\pm 7.5\%$ around the major oxides, and ± 0.5 wt% around the minor oxides). These sludge composition intervals were then used to generate extreme vertices (EVs) for each of the sludge projections of Table 4-1. The EVs were then coupled with Frit 803 over a WL interval of 25 – 50% WL to determine the WL interval over which all of the EVs were classified as acceptable for both process and product performance constraints as defined by DWPF's Product composition Control System (PCCS).

Table 5-1 summarizes the results of the MAR assessments for the Frit 803 – SB8 system with and without ARP (SE was not accounted for in this previous assessment).

^a The results of the Variation Stage assessment (i.e., projected operating windows) were transmitted to SRR on 5-1-13 via personal communication (email) – see page 124 of SRNL-NB-2012-00070 for more details.

Table 5-1. Projected Operating windows for the Frit 803 - SB8 Sludge Only and Coupled Operations Systems (No Strip Effluent Added)

Sludge/ARP (gallons)	Projected Operating Window	# of EVs failed at next highest WL
0	30 – 40 (T_L)	14 out of 4202
1000	28 – 43 (low η)	165 out of 4440
1250	27 – 42 (low η)	12 out of 4440
1500	27 – 42 (low η)	150 out of 4440
1750	26 – 42 (low η)	297 out of 4440
2000	29 – 41 (low η)	113 out of 4440

The Frit 803 – SB8 sludge-only operating window is 30-40% WL with predictions of liquidus temperature (T_L) limiting access to higher WLs. At 41% WL for the sludge-only flowsheet, 14 out of the 4202 EVs fail T_L . As the ARP product is added to the SRAT, the projected operating windows initially increase (up to 1000 gallons of ARP product) to 28 – 43% WL and transition from a T_L limited system to a low viscosity (low η) limited system due to the additional Na_2O being added to the glass from ARP coupled with higher targeted WLs. With the addition of 1250 gallons of ARP product, the maximum WL that can be attained is reduced to 42% given the continual increase in Na_2O content which drives viscosity predictions to lower values and thus cuts off access to higher WLs. At 43% WL, 12 out of the 4440 EVs fail low η . A gradual reduction (albeit it slight and still very acceptable) in the upper WL that can be achieved continues with ARP additions up to 2000 gallons. ARP product additions greater than 2000 gallons were not assessed given the known impact of TiO_2 concentration on the projected operating window. That is, with ARP product additions greater than 2000 gallons, the TiO_2 content in the glass at 40% WL exceeds the 2 wt% TiO_2 (in glass) PCCS limit. Hence the current restrictions placed on the amount of ARP product that can be added to the SRAT until the TiO_2 solubility limit and the T_L model are revised.

The results of this assessment indicate that Frit 803 is a viable option for the 4-30-13 SB8 projection (with variation applied) for both sludge-only and coupled operations up to 2000 gallons of ARP. Viable in this context means that the projected operating windows range from at least as low as 32% WL to as least as high as 40% WL with sludge variation accounted for. This projected window will allow DWPF to target a nominal 36% WL and provide some robustness to WL variation (± 4 WL points) that has been observed during normal facility operations.

5.2.2 MAR Results with SE

So given this baseline, what is the impact of the addition of 70,000 liters of SE to the SRAT on the projected operating windows? As previously mentioned, the 70,000 liters of SE translates into 0.95 wt% B_2O_3 in the SRAT. An enhanced Variation Stage assessment was performed as part of this study in which the EVs of the sludge components were based on the minimum and maximum values of the 4-30-13 projection and, to account for the SE addition, a B_2O_3 component was added with a range of 0 to 2 wt%. The use of 2 wt% is almost twice the oxide content of the 0.95 wt% calculated based on the assumptions and inputs shown previously. It should be noted that the B_2O_3 range of 0 – 2 wt% is larger than applying either a $\pm 7.5\%$ or a ± 0.5 wt% value around the nominal 0.94 wt%.

The results of the SE-based Variation Stage assessment are shown in Table 5-2. A comparison of Table 5-1 and Table 5-2 shows very little difference in the projected operating windows with and without SE added to the SRAT. The sludge-only flowsheet (no ARP) yields the identical projected operating window of 30-40% WL. The same general trends are observed with the SE-based coupled operations flowsheet as shown in Table 5-1. With initial additions of ARP and accounting for SE (up to 2 wt%), the projected operating windows increase to 28-43% WL and transition to a low η limited system. With further additions of ARP (while still accounted for 2 wt% SE), the upper achievable WL gradually decreases due to the additional Na₂O being added to a low η system. The two differences observed between Table 5-1 and Table 5-2 are the upper WL for the 1750 gallon addition of ARP (41 and 42% WL with and without SE accounted for, respectively) and the dual constraint limitation with 2000 gallons of ARP added for the SE-based assessments. More specifically, both low η and durability (ΔG_p) limit access to WLs of 42% and higher for the SE-based coupled operations flowsheet.

These results indicate that the 0.0125M (or 12.5mM) boric acid upper limit (based on anticipated compositional tolerances) will have very little, if any, impact on the projected operating windows for the Frit 803 – SB8 system regardless of the presence or absence of ARP and SE.

Table 5-2. Projected Operating windows for the Frit 803 - SB8 Sludge Only and Coupled Operations (ARP and SE) Systems (Strip Effluent Added – 0 to 2 wt%)

Sludge/ARP/SE (gallons)	Projected Operating Window	# of EVs failed at next highest WL
0	30 – 40 (T _L)	14 out of 8806
1000	28 – 43 (low η)	279 out of 9179
1250	27 – 42 (low η)	37 out of 9221
1500	27 – 42 (low η)	237 out of 9221
1750	26 – 41 (low η)	5 out of 9221
2000	29 – 41 (low η / ΔG_p)	195 out of 9221

6.0 Conclusions

The introduction of the dilute (0.01M) boric acid stream into the DWPF flowsheet has potential impact on glass formulation and frit development efforts since B₂O₃ is a major oxide in frits developed for DWPF. Prior knowledge of this stream can be accounted for during frit development efforts but that was not the case for SB8. Frit 803 has already been recommended and procured for SB8 processing; altering the frit to account for the incoming boron from the SE is not an option. Therefore, the response of Frit 803 to the introduction of the baseline SE including its compositional tolerances (i.e., up to 0.0125M boric acid) is of interest and was the focus of this study. The primary question to be addressed in the current study was: What is the impact (if any) on the projected operating windows for the Frit 803 – SB8 flowsheet to additions of B₂O₃ from the SE in the SRAT? More specifically, will Frit 803 be robust to the potential compositional changes occurring in the SRAT due to sludge variation, varying additions of ARP and/or the introduction of SE by providing access to waste loadings (WLs) of interest to DWPF?

To support this assessment, SRNL performed Variation Stage MAR assessments for Frit 803 – SB8 potential flowsheets involving sludge-only and coupled (with and without ARP and SE) operations. The metric to gage the impact of the addition of SE was based on the projected operating windows which are defined as the WL interval over which glasses are classified as acceptable based on current DWPF PCCS models. Calculations were made based on an assumed 70,000 liters of SE added to the SRAT which translated into 30.5 kg of B₂O₃ (calcined oxide basis) being added to the SRAT or ultimately 0.95 wt% B₂O₃ in the calcined SRAT product. Although the volumes used to support this calculation are considered bounding, SRNL utilized a maximum B₂O₃ content of 2 wt% in the SRAT to support this assessment (i.e., a 2x increase).

These MAR results indicate that there is very little, if any, impact on the projected operating windows for the Frit 803 – SB8 system regardless of the presence or absence of ARP (up to 2000 gallons) and SE (up to 2 wt% B₂O₃ contained in the SRAT).

The impact on CPC processing of a 0.01M boric acid solution for elution of cesium during Modular Caustic Side Solvent Extraction Unit (MCU) processing has previously been evaluated by the Savannah River National Laboratory (SRNL). Increasing the acid strength to 0.0125M boric acid to account for variations in the boric acid strength has been reviewed versus the previous evaluation. The amount of acid from the boric acid represented approximately 5% of the total acid during the previous evaluation. An increase from 0.01 to 0.0125M boric acid represents a change of approximately 1.3% which is well within the error of the acid calculation. Therefore, no significant changes to CPC processing (hydrogen generation, metal solubilities, rheological properties, REDOX control, etc.) are expected from an increase in allowable boric acid concentration from 0.01M to 0.0125M.

7.0 Recommendations, Path Forward or Future Work

The following recommendation is made based on the results of this study:

- If the molarity of the boric acid flowsheet is increased above that corresponding to 2 wt% B₂O₃ in the SRAT (on a calcined oxide basis), the ramifications on predicted properties and SME acceptability decisions could become more serious warranting additional evaluations.

8.0 References

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