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Residual Insoluble Solids Expected from Saltcake Dissolution

C. J. Martino

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EXECUTIVE SUMMARY

A review of previous SRS saltcake dissolution and characterization studies was performed to inform the analysis of the amount of residual insoluble solids that may result from saltcake dissolution. A realistic value covering most cases for the maximum volume of a settled layer of residual insoluble solids that would result from dissolution of a volume of saltcake was calculated as 0.08 mL/mL (8 vol%). Applying factors to account for dissolution test uncertainty and differences between in-tank settling and laboratory testing, the recommended value for a settled layer of residual insoluble solids that would result from dissolution of a volume of saltcake is 0.16 mL/mL (16 vol%). This should apply to dissolution of saltcake with typical minimal amounts of entrained sludge but may not apply to saltcake with significant sludge. As it is composed of primarily non-radioactive isotopes, the precipitated aluminum hydroxide material does not contribute to the heat load.

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LIST OF ABBREVIATIONS

HLW	High-Level Waste
IW	Inhibited Water
IS	Insoluble Solids
n.m.	not measured
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
VDS	Variable Depth Sample
XRD	X-Ray Diffraction

1.0 Introduction

1.1 Background

Tank 9H Variable Depth Sample (VDS) HTF-09-21-42, taken on May 17, 2021 during the preparation of Batch 1B of the Tank 9H salt dissolution campaign, showed an unexpected high quantity of solids following salt dissolution.¹ These undissolved solids must be assessed for potential flammability concerns related to gas retention and release to ensure safe operations. This report provides an evaluation of the historic evidence for insoluble solids remaining from salt dissolution. Savannah River Remediation (SRR) requested that Savannah River National Laboratory (SRNL) perform this review to provide a basis for quantifying undissolved solids resulting from salt dissolution activities. The objective of this evaluation is to support current salt tank project scopes.

Several topics for potential future evaluations are outside of the scope of this analysis. First, this evaluation does not address how much gas is retained by saltcake insoluble solids such as aluminum hydroxide and sludge solids. Second, this analysis does not employ equilibrium modelling to evaluate different salt dissolution and retrieval methods that could minimize the formation of aluminum hydroxide. Third, this analysis does not provide additional characterization of insoluble solids resulting from salt dissolution.

2.0 Methodology

2.1 Approach

The approach used in this evaluation was to examine the history of samples and field observations and look at cases with high aluminum concentration or precipitated aluminum hydroxide. Information on dissolution tests and in-tank dissolution were favored, particularly when identification and quantification of a volume of aluminum hydroxide (gibbsite or bayerite isomers of $\text{Al}(\text{OH})_3$)² remaining per volume of saltcake dissolved was given. The goal of this evaluation is to conservatively estimate the residual insoluble solids in the absence of any additional saltcake characterization information. Outside of this analysis but possible future scope, other types of sample information or representative data could be used along with OLI modeling to determine maximum mass and volume of aluminum hydroxide that can be formed for different materials during dissolution (similar to what was done for Tank 41H).³

The approach for conservatism in this analysis is not to absolutely bound any layer that could possibly be encountered in a portion of a Savannah River Site (SRS) High-Level Waste (HLW) salt dissolution campaign. Rather, this evaluation aims to overpredict the amount of aluminum hydroxide that would be precipitated from salt dissolution and bound most of the salt dissolution batches. The intent of this approach is for this prediction to be applicable generally to SRS saltcake dissolution regardless of the specific dissolution method employed.

2.2 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.

3.0 Discussion

3.1 Categories of Saltcake Insoluble Solids

It is useful to classify types of suspended material that may result from saltcake dissolution. In this analysis, three categories of suspended material are proposed: loose salt crystals, sludge solids, and precipitated aluminum hydroxide.

First, loose salt crystals are either suspended or reprecipitated during saltcake dissolution. These crystals would tend to be either from the major crystalline salt component, such as sodium nitrate, or from sparingly soluble salts, such as sodium sulfate, sodium carbonate, or sodium oxalate, that are displaced during dissolution of surrounding salts. Although there is some uncertainty, once the loose salt crystals settle, they are assumed to have similar gas retention and release characteristics to that of the bulk saltcake. The heat load for loose salt crystals would already be considered by the tank inventory. Cesium-137 activity in the interstitial liquid is expected to be gradually lowered throughout subsequent salt dissolution batches if some flow through the saltcake occurs. Strontium-90 may be present as an insoluble strontium carbonate salt that preferentially dissolves later in the process,⁴ with these less soluble salts contributing to the heat load in the saltcake until near the late stages of dissolution, where it then will contribute to the heat load in the dissolved salt.

Second, sludge solids that are similar in composition to settled sludge in the SRS sludge tanks are expected to be contained in small quantities within the saltcake.⁵ Some of the material may be from insoluble metal oxides and hydroxides that were entrained through the HLW evaporators, and other portions of the material may have been precipitated due to the concentration in the evaporator and subsequent cooling that results in solubility changes in the evaporator drop tanks. As much of the strontium and actinide content tends to be in this category of solids, the heat load of the saltcake is significantly impacted by the sludge solid material. Some of the insoluble solids in salt tanks that are less typical of sludge solids are zeolites, sodium aluminosilicates, and other silicon containing phases.

Third, aluminum hydroxide solid phases such as gibbsite and bayerite can be considered as a separate category because they are solids that have a high propensity to be created during saltcake dissolution. The other two categories of residual insoluble solids will either remain unchanged, decrease, or may be altered in minor ways during salt dissolution. However, the aluminum hydroxide solids have the potential to be precipitated in bulk as the chemistry changes during salt dissolution. The estimation of aluminum hydroxide precipitation is the main focus of this evaluation. By nature, aluminum hydroxide contains only aluminum, oxygen, and hydrogen, which are primarily non-radioactive elements in SRS waste. Thus, the heat load associated with layers of primarily non-radioactive aluminum hydroxide solids such as gibbsite and bayerite would be dependent on the other soluble (cesium-137) and insoluble (sludge) components in the overall mixture. At SRS, aluminum hydroxide (gibbsite and bayerite) and aluminum oxide hydroxide (boehmite, γ -AlO(OH)) are typically considered part of the sludge rather than the saltcake, but are being considered separately here.

The insoluble solids in the Tank 9H VDS HTF-09-21-42 were caught on a filter and analyzed by powder X-Ray Diffraction (XRD).¹ The major components were gibbsite, $\text{Al}(\text{OH})_3$, and sodium nitrate, NaNO_3 . Other minor library matches were for α - Al_2O_3 ,^a Fe_2O_3 , and an aluminosilicate phase.^b The sodium nitrate falls into the category of the loose salt crystals and would be expected to behave like the original saltcake. The sample HTF-09-21-42 slurry had 5.0 wt% aluminum, which corresponds to 14.6 wt% of slurry if all aluminum were to be considered as aluminum hydroxide. By comparison, sludge, as estimated with the iron oxide and other elements excluding aluminum hydroxide, only contributed to 0.22 wt% of the slurry.¹ From this, it is clear that the majority of the non-salt-like slurried solid material that needs to be considered for its potential to retain gas is the aluminum hydroxide phase gibbsite. When allowed to settle, the insoluble solids layer was approximately 60 vol% of the sample after 2 hours and approximately 15 vol% of the sample after 24 hours.⁶

^a It is unlikely that corundum, an aluminum oxide crystalline phase, would be formed by the HLW evaporation conditions or the saltcake dissolution conditions, as a much higher temperature would typically be required.²

^b The identification of the specific aluminosilicate phase should be considered tentative.

The mechanism of gibbsite and bayerite formation requires that the dilution of liquid phase aluminate (and potentially the dissolution of solid sodium aluminate) results in chemistry favorable for precipitation of aluminum hydroxide. The solubility of gibbsite and bayerite are very dependent on the pH (or hydroxide concentration).^{2, 7-8} During saltcake dissolution, the aqueous hydroxide concentration is lowered from its relatively high levels in the saltcake interstitial liquid, causing the solubilities of these aluminum hydroxide phases to decrease.⁸ The temperature does not greatly impact the equilibrium solubility of gibbsite and bayerite over the range applicable to saltcake dissolution.⁸ In systems with sodium hydroxide, aluminate, nitrate, nitrite, carbonate, and sulfate, the solubility of the aluminum hydroxide phases are greatly enhanced above 1 M hydroxide concentration versus systems with only sodium hydroxide and aluminate.⁷ Similarly, in systems with only sodium hydroxide, aluminate, and nitrate, dependence of the gibbsite solubility on the aqueous nitrate concentration has been demonstrated.⁹⁻¹² In practical situations during saltcake dissolution, the kinetics of aluminum hydroxide precipitation can be very slow (several days to multiple weeks)¹⁰⁻¹² when compared to the relatively fast kinetics of sodium nitrate salt dissolution. Gibbsite and bayerite crystals have densities of 2.42 g/mL and 2.53 g/mL, respectively,² so both of these solid phases would be expected to settle in dissolved saltcake solutions with nominal supernatant densities, which are typically 1.4 g/mL or lower.

3.2 SRS Salt Dissolution History

There have been several summaries of SRS saltcake dissolution history through 2004.¹³⁻¹⁵ The summaries detail salt removal from Tanks 10H, 16H (annulus), 17F, 19F, 20F, 22H, 24H, 33H, 37H, 41H, and 49H. Additional salt dissolution campaigns since 2005 include salt from Tanks 10H, 25F, 37H, and 41H.¹⁶ Observations of residual insoluble solids were noted for Tank 19F and Tank 41H dissolution campaigns, and these are discussed below.

Tank 19F dissolution appeared to have residual sludge and zeolite roughly matching the amount of sludge and zeolite in the tank prior to salt dissolution.^c In total, 1050 kgal of salt was dissolved without appreciable gibbsite formation.¹⁷

A sample of the saltcake surface was taken after Tank 41H dissolution in 2003 (addition of 244 kgal of inhibited water (IW)). The top 3-inches of the saltcake sample was dissolved in the laboratory, resulting in residual insoluble solids that were primarily aluminum hydroxide (gibbsite and bayerite) and a smaller amount of iron and sludge components.¹⁸ Dissolution of the aluminum-rich saltcake material Supernate samples taken in 2020 at late stages of Tank 41H dissolution showed insoluble gibbsite, as well as insoluble sodium aluminosilicates, sodium carbonate, and other minor components.¹⁹⁻²⁰

3.3 Salt Samples and Dissolution Tests

SRS has a history of localized saltcake samples. Early salt retrieval campaigns in the 1980s typically involved localized salt samples prior to the in-tank dissolution, which were analyzed for water soluble and water insoluble components. Some of the actual campaigns also involved samples of the dissolved solutions of the residual insoluble solids. From these tests, the residual insoluble solids analysis is most important for our use if they can be related back to the original saltcake volume.

Subsequently, in the 1990s and early 2000s, localized samples were taken for saltcake criticality analyses. These samples were collected from either the surface of the saltcake or from the bottom of wells mined out of the bulk saltcake by the addition of water. The focus of these analyses was showing that the neutron poisons remaining in the saltcake insoluble solids were sufficient to poison the uranium-235 and other fissiles that would also primarily remain insoluble during saltcake dissolution.

^c The zeolite in Tank 19F was previously dispositioned to the tank rather than formed in the tank waste.

Starting in 2002, efforts were made to increase the subsurface characterization information. A core sampling system was implemented where a series of thin walled 1-inch inner diameter tubes would be pounded into the surface of the saltcake in a manner that minimized disturbance of the salt material being sampled when compared to the previous methods. Up to three successive 12-inch segments of saltcake would be collected, proceeding downward in the tank with each successive sample. Use of these relatively undisturbed samples allowed for improved saltcake physical property measurement (density, saturation, permeability).

In 2005 and 2006, samples from Tanks 25F, 28F, and 44F were collected in Hanford-style universal samplers using a Boart-LongyearTM LMTM75 core sampling rig. Use of this drill rig core sampler allowed for proceeding deeper into the saltcake to collect samples. The increased volume of the samples over the previous sampling method allowed for segment characterization and saltcake draining and dissolution testing, and provided ample dissolved saltcake for use in other research programs.

This section examines the saltcake samples and dissolution tests with a primary focus on the saltcake aluminum content and the observation of aluminum hydroxide formation, and a secondary focus on sludge contained in saltcake. In general, the samples are discussed in reverse chronological order (the reverse of the order in which they were described above) because the most useful information for this evaluation tends to be from the enhanced analyses of the most recent saltcake samples. The tabulated data presented in this section typically contains the aluminum, sodium, and water content of the saltcake. The aluminum and sodium content are useful in approximating the propensity for gibbsite formation upon dissolution. Water content is useful in comparing how much interstitial liquid would be present in the saltcake samples. Because hydroxide is contained in the interstitial liquid, the saltcake water content is generally directly proportional to the hydroxide per volume of saltcake.

3.3.1 LMTM75 Saltcake Core Samples

The first attempt to sample Tank 25 saltcake with the LMTM75 system was mostly unsuccessful due to drill string fluid leakage and incomplete samples.²¹ The second attempt to sample Tank 25 with this system provided a core for segment characterization and draining/dissolution testing.²² Table 3-1 contains aluminum, sodium, and moisture information for the Tank 25 saltcake segments. FTF-504 is closest to the tank surface while FTF-513 is closest to the tank bottom. Three of the core sample segments from near the middle of the tank had relatively high aluminum concentration for saltcake (4 to 6.5 wt%), while the upper and lower portions of the tank had more typical aluminum concentrations for saltcake (0.13 to 1.6 wt%). A composite with 1.7 wt% aluminum was drained, then dissolved with a series of three IW contacts, and the resulting aluminum and sodium concentrations are shown in Table 3-2. The dissolved salt solutions from the first and second contacts with IW subsequently precipitated gibbsite when observed after two weeks. The precipitated gibbsite was greatest in both total quantity and percent of solution volume for the first dissolution contact. When the solutions were agitated and allowed to settle (for a short but unspecified time), the gibbsite layers corresponded to approximately 20 vol% and <5 vol% of the first and second dissolution solutions, respectively. The corresponding volume of the gibbsite layer in the first dissolution solution to the volume of saltcake dissolved cannot be determined precisely due to the rewetting of the drained saltcake, but is likely greater than 20 vol% of the saltcake dissolved in the first dissolution contact. Dividing the solids layer volume from the first two dissolution steps by the overall volume of salt dissolved, the layer of gibbsite formed would correspond to approximately 16 vol% of the original saltcake by the end of Tank 25F salt dissolution. It is unknown whether this solids layer with minimal settling would be concentrated enough to retain gas.

Table 3-1. Concentration of Al and Na in Tank 25F Core Sample Segments on a Wet Basis.²²

Segment	Al (wt%)	Na (wt%)	Al/Na	water (wt%)
FTF-504	0.594	22.9	0.026	13.3
FTF-505	1.50	21.5	0.070	n.m
FTF-506	1.09	22.8	0.048	29.1
FTF-507	5.05	21.5	0.235	24.4
FTF-508	6.54	22.0	0.297	18.9
FTF-509	3.89	21.9	0.178	13.3
FTF-510	1.55	24.2	0.064	7.6
FTF-511	0.395	23.5	0.017	14.9
FTF-512	0.145	24.3	0.006	6.2
FTF-513	0.139	22.6	0.006	12.3
composite	1.71	25.0	0.068	12.2

Table 3-2. Concentration of Al and Na from Dissolution Test of Tank 25F Core Sample Composite.²²

dissolution test	Al (mg/L)	Na (mg/L)	Al/Na
interstitial liquid	32300	262000	0.123
1st dissolution	44900	213000	0.211
2nd dissolution	11100	193000	0.058
3rd dissolution	2000	131000	0.015
solution composite	14200	175000	0.081

Because the Tank 25F dissolution test was performed on a drained saltcake composite with about 1.7% aluminum, and some segments had higher aluminum concentrations (4 to 6.5 wt% Al), the volume of gibbsite formed per volume of original saltcake would be expected to have been higher for those individual high aluminum concentration saltcake segments. No dissolution tests were performed on the individual segments. From the draining and dissolution results, the composite of the segments show evidence of containing some insoluble sodium aluminate. The draining of the saltcake removes a portion of both hydroxide and the aluminate from the saltcake, thus changing the chemistry that relates to the precipitation of aluminum hydroxide. Draining of interstitial liquid prior to dissolution may partially explain differences in the aluminum hydroxide precipitation from the Tank 25F saltcake dissolution test when compared with saltcake dissolution tests with undrained saltcake.^d

^d The following is a hypothesis explaining why aluminum hydroxide precipitated from the first dissolution step of drained Tank 25F saltcake is apparently greater than the value (0.16 mL of residual insoluble solids per mL of dissolved saltcake) developed in this report. Based on Tank 25 characterization and dissolution test results, it is probable that some of the segments contained solid sodium aluminate salt. When these segments were combined in the composite, the solid sodium aluminate likely remained intact even though the overall aluminum content of the composite was much lower than that of the individual segments. Draining of the saltcake prior to dissolution removed a non-trivial amount of the interstitial liquid. Due to the presence of solid sodium aluminate and the hydroxide being present in the interstitial liquid, draining removes proportionately more of the hydroxide than the aluminate. When the sodium aluminate is dissolved (preferentially in the early dissolution contacts for this case where saltcake is mixed during dissolution) the resulting solution is deficient in hydroxide, leading to a supersaturated solution with respect to aluminum hydroxide. This supersaturated solution ultimately precipitates aluminum hydroxide. Thus, per this hypothesis, the dissolution of drained saltcake that contains solid-phase sodium aluminate salt has increased potential for gibbsite precipitation.

Table 3-3 contains aluminum and sodium data for a set of Tank 28F core samples, which were very wet.²³ It is unknown whether the relatively high water contents were representative of the entire saltcake or a localized phenomenon to right where the sample was collected. A hypothesis at the time was that the salt near the cooling coils might be drier and consolidated while the salt away from the coils where the sampling is performed might be wetter and looser. These wet samples had some segments with relatively high aluminum content (up to 10.8 wt% aluminum) and likewise were expected to be very susceptible to precipitation of aluminum hydroxide upon salt dissolution. A single-stage dissolution test of the undrained composite of the Tank 28F saltcake (with 2.38 wt% aluminum) was performed with a relatively large 4:1 mass ratio of water to salt. The resulting insoluble solids corresponded to only 0.0048 grams of dry insoluble solids per gram of saltcake dissolved. The XRD of the unwashed insoluble solids indicated sodium nitrate, bayerite, and gibbsite. The quantity of insoluble solids from the Tank 28F dissolution is consistent with the amounts that are typically seen in historic saltcake samples (see Table 3-6). On a blended composition basis, Tank 28F dissolution did not appear to generate more aluminum hydroxide than Tank 25F dissolution.

Table 3-3. Concentration of Al and Na in Tank 28F Core Sample Segments on a Wet Basis.²³

Segment	Al (wt%)	Na (wt%)	Al/Na	water (wt%)
FTF-456	0.281	21.7	0.013	17.3
FTF-457	2.57	21.5	0.120	13.6
FTF-459	5.88	18.1	0.325	39.5
FTF-460	0.655	22.1	0.030	21.9
FTF-461	3.03	23.4	0.129	32.2
FTF-462	3.8	20.7	0.184	24.1
FTF-463	10.8	20.3	0.532	30.7
FTF-464	6.17	19	0.325	37.9
FTF-465	3.79	18.7	0.203	24.8
composite	2.38	22.7	0.105	15.4

Thirteen years after the samples were collected, the water-soluble portion of the Tank 44F saltcake core samples were used in thermolytic hydrogen generation testing.²⁴ Previously, the individual segments were emptied, photographed, and measured for water content.²⁵ Table 3-4 contains the applicable aluminum, sodium, and water content data for the Tank 44F samples, with the composites receiving most of the analyses rather than the individual segments. Analytes in tables are listed as “n.m.” when they were not measured.

Tank 44F segments from the 171 to 285 inch tank level tended to be lighter in color and drier, and were used in an “upper composite.” Tank 44F segments from the 76 to 114 inch tank level were darker in color and wetter, and were used in a “lower composite.” The characterization of the upper composite indicated a more typical saltcake composition of primarily sodium nitrate with approximately 1 wt% aluminum. The characterization of the lower composite revealed the saltcake to be approximately half sodium carbonate with an aluminum content of approximately 3 wt%. The lower composite also showed higher concentrations of quantities of more typical sludge components, such as iron (4.3 wt%), uranium (1.6%), and manganese (1.1%). When oxides of the sludge metals are considered, the sludge content may contribute to a significant slurried sludge volume that would need to be considered in addition to any aluminum hydroxide present. Tank 44F history does not indicate that it has received sludge solids and thus the sludge solids evident in the sample would have been entrained through the evaporator.²⁶

Table 3-4. Tank 44F Saltcake Core Sample Analysis on a Wet Basis.²⁴

Segment	Al (wt%)	Na (wt%)	Al/Na	water (wt%)
FTF-44-06-48-1	n.m.	n.m.	n.m.	14
FTF-44-06-48-2	n.m.	n.m.	n.m.	12.6
FTF-44-06-48-3	n.m.	n.m.	n.m.	5.9
FTF-44-06-48-4	n.m.	n.m.	n.m.	7.6
FTF-44-06-48-5 bot	n.m.	n.m.	n.m.	19.5
FTF-44-06-48-5 top	n.m.	n.m.	n.m.	5.4
FTF-44-06-48-6	n.m.	n.m.	n.m.	7.4
FTF-44-06-48-10	n.m.	n.m.	n.m.	31.6
FTF-44-06-48-11	n.m.	n.m.	n.m.	30.5
upper composite (1 to 8)	1.07	25.3	0.042	n.m.
lower composite (10 and 11)	2.97	26	0.114	n.m.

3.3.2 Earlier Saltcake Core and Surface Samples

Table 3-5 contains information from saltcake segments from thin-walled subsurface samples from the top three feet of saltcake or less, and one 8-inch surface cup sample. Because the thin-walled subsurface samples have minimal disturbance, they were used to determine saltcake physical properties such as permeability and density. The bulk composition of some samples were not analyzed due to the use of individual samples in permeability or dissolution tests. Table 3-6 contains information from smaller (nominally 4-inch) cup samples from the saltcake surface or salt wells. For both tables, analytes are listed as “n.m.” when they were not measured. In some cases, sodium could possibly be determined from the salt information provided but it would require additional data processing.

Table 3-5. Concentration of Al and Na in Saltcake Segments on a Wet Basis.

Tank	Sample	Al (wt%)	Na (wt%)	Al/Na (w/w)	water (wt%)	Report
2	T2F-1-1	1.43	25.3	0.057	6.2	WSRC-TR-2004-00131
3	FTF-223	0.174	25.4	0.007	14.1	
	FTF-224	0.419	23.9	0.018	5.1	
	FTF-225	n.m.	n.m.	n.m.	n.m.	
10	HTF-609	n.m.	n.m.	n.m.	9.9	WSRC-TR-2004-00164
	HTF-610	0.286	28.4	0.010	12.8	
	HTF-611	0.204	25.8	0.008	4.4	
29	T29H-B6-1	13.3	21.3	0.624	21.4	WSRC-TR-2004-00130
31	HTK-513	4.95	27.3	0.181	14.1	WSRC-TR-2002-00388
38	HTF-E-03-114	0.138	22.6	0.006	16.8	WSRC-TR-2004-00129
41	HTF-E-03-033 top	0.275	30.0	0.009	24.6	WSRC-TR-2003-00227
	HTF-E-03-034 top	0.0628	23.9	0.003	3.0	
	HTF-E-03-035 bot	0.0797	24.8	0.003	3.5	
	HTF-E-03-145	0.767	20.4	0.038	9.9	WSRC-TR-2004-00165
	HTF-E-03-146	0.262	26.0	0.010	4.1	
	HTF-E-03-147	1.58	25.3	0.062	8.5	

3.3.2.1 Tank 31H Dissolution Test

A dissolution test was performed with HTK-513, an 8-inch saltcake surface sample from Tank 31H.⁴ Characterization of the original Tank 31H saltcake sample revealed the sample to contain 4.95 wt% aluminum. The test contacted 61 grams of saltcake (approximately 30 mL assuming a saltcake density of 2 g/L²⁷) with ten successive volumes of IW, centrifuging and removing the dissolved solution after each dissolution batch. After a cumulative 2.3:1 mass ratio of IW was added, approximately 1.5 mL (2.42 g) of residual insoluble material remained on a wet basis. The apparent density of the wet insoluble solids was 1.6 g/mL. The residual insoluble material was composed of about 87 wt% bayerite and gibbsite and about 7 wt% hematite (Fe₂O₃). When dried, the solids weighed 0.83 grams, for approximately 1.36 grams of dried residual insoluble solids per gram of original saltcake.

Other data generated from the Tank 31H dissolution test include the following:

- dissolution with IW resulted in 13.6 wt% of the aluminum in the original saltcake sample being contained in the residual insoluble solids, and
- the residual insoluble solids had a total solids content of 34.3 wt % (water content of 65.7 wt%).

Comparing the 1.5 mL of residual insoluble solids to the 30 mL of saltcake used, the residual insoluble solids volume corresponded to 5% of the initial salt volume (wet residual solid layer is 0.05 mL/mL original salt). The 0.05 mL/mL value is approximate because of the nature of the 1.5 mL measurement, so some margin could be added to account for uncertainty. For example, if the residual solids volume was as high as 2 mL, which is within reason, then the residual insoluble solids per volume of initial saltcake will increase by 33% to 0.067 mL/mL. Additional caution should be used when applying this data obtained for the volume of a centrifuged layer solids to situations involving the volume of settled layers of solids.

For several reasons, the Tank 31H dissolution test result is seen to be a good basis for most salt tanks:

- the saltcake was relatively high in aluminum content,

- the dissolution test was performed in a relatively controlled manner, and
- information was collected on the volume of bayerite and gibbsite residual solids.

3.3.2.2 Thin-Walled Saltcake Core Samples

Salt well samples prior to salt dissolution indicated the propensity of Tank 41H to precipitate gibbsite upon salt dissolution.²⁸ However, a set of core samples from the top 3 feet of Tank 41H did not have particularly high levels of aluminum and would not have given such an indication.²⁹ Using OLI modelling on the sample results, the expectation was that 6.5 inches of gibbsite would be formed if the saltcake is dissolved down to 77-inches in the tank.³ This corresponds to around 2 to 3% of the residual volume as gibbsite. The sample analysis that this was based on was not necessarily conservative, so there may have been more than 6.5 inches of gibbsite formed during salt dissolution. A Tank 41H core sample taken after the June 2003 dissolution campaign revealed that the top three inches of the surface sample (HTF-E-03-145) was 0.767 wt% aluminum.¹⁸ In dissolution testing of the HTF-E-03-145 material, 0.88 g of residual insoluble solids (on a dry basis) was obtained from 58.8 g of saltcake and 22.5 g of supernate. The dried residual insoluble solids correspond to 1.5 wt% of the original as-received 3-inch segment and were 27.4 wt% aluminum, with XRD identifying gibbsite and bayerite.

A Tank 29H saltcake core sample was collected from the saltcake surface in an apparent salt well that was approximately 7-feet lower than the surface of the bulk saltcake.³⁰ The salt well was likely due to the previous mining of a hole in the salt below the B6 riser for the installation of a slurry pump. The Tank 29H saltcake sample was partially used for characterization and partially used for a flow-through dissolution test. The characterization portion of the Tank 29H sample had a very high aluminum concentration for saltcake (13.3 wt%). A supernate sample collected from the salt well at the time of sampling indicated a different composition (lower hydroxide and higher nitrite) than the interstitial liquid contained in the Tank 29H saltcake sample. This is additional evidence of previous saltcake dissolution that could have biased the composition of the Tank 29H saltcake sample. These circumstances surrounding the collection of this sample from the B6 riser should be considered when determining how to use the results of the Tank 29H sample in this evaluation of residual insoluble solids. The remaining portion of the Tank 29H saltcake sample was drained and dissolved with flow through dissolution. There was gibbsite precipitation that caused a blockage of our flow-through dissolution. Ultimately, about 40 mL of gibbsite/bayerite was produced from about 53 mL of saltcake. The volume of residual aluminum hydroxide was 75% of the initial salt volume. While this result for the amount of gibbsite and bayerite resulting from the dissolution of Tank 29H saltcake is very conservative, it is not recommended for use in this determination of a residual solids layer that is not meant to be absolutely bounding.

Tanks 3F³¹ and 10H²⁷ saltcake sample flow-through dissolution tests appeared to proceed without appreciable gibbsite formation. Trivial amounts of solids, likely sludge, were present. Tank 38H samples have tended to show sodium aluminosilicate and other silicon-containing compounds in the residual solids rather than aluminum hydroxide.³²

Table 3-6. Concentration of Al, Na, and Water Insoluble Solids (IS) from Saltcake Surface, Salt-Well, and Criticality Analysis Samples, on a Dry Basis.

Tank	Sample Name or Year	Saltcake			Insoluble Solids		IS/salt (dry wt%)	Report
		Al (dry wt%)	Na (dry wt%)	Al/Na (w/w)	Al (dry wt%)	Na (dry wt%)		
1	1981	4.79	n.m.	n.m.	n.m.	n.m.	1.6%	J. Fowler, 1994
10	unknown	0.23	n.m.	n.m.	n.m.	n.m.	2.2%	
18	1981	1.53	n.m.	n.m.	n.m.	n.m.	< 0.1%	
23	1984	0.0021	n.m.	n.m.	n.m.	n.m.	n.m.	
19	1980	3.32	n.m.	n.m.	n.m.	n.m.	4%	
37	2001	1.06	28.7	0.037	8.82	17.3	1.7%	DPST-80-569
	HTK-493,494	2.28	25.8	0.088	25.2	2.45	2.5%	WSRC-TR-2001-00517
	HTF-059	1.11	28.3	0.039	15.3	3.69	1.5%	WSRC-TR-2002-00244
38	HTF-395,396	0.201	29.8	0.007	6.65	10.4	0.8%	WSRC-TR-2005-00277
	T38HTF088,089	0.139	30.5	0.005	n.m.	n.m.	< 0.1%	WSRC-RP-2001-00574
	HTF-093	n.m.	n.m.	n.m.	n.m.	30.9	n.m.	WSRC-TR-2001-00516
	HTF-003	0.21	30	0.007	1.5	17.0	0.5%	WSRC-RP-98-00725
41	ITP-309	0.37	40	0.009	0.12	10.0	1.4%	WSRC-TR-96-0410
	#1-1 (1983)	0.212	28.0	0.008	0.621	8.5	2.0%	WSRC-TR-94-057
	#2-1 (1983)	0.107	27.9	0.004	0.655	23.2	0.6%	
	#2-2 (1983)	0.247	26.6	0.009	n.m.	n.m.	n.m.	
	#2-3 (1983)	0.101	27.3	0.004	0.484	23.9	0.9%	
	T41-HTF-E-173	2.76	24.9	0.111	29.6	0.72	0.2%	WSRC-TR-2002-00343
46	T46FTF048-1	1.11	24.2	0.046	0.773	21.7	n.m.	WSRC-TR-2001-00519
	T46FTF048-2	2.04	37.4	0.055	29.1	0.59	0.7%	

3.3.2.3 Other Saltcake Surface and Salt-Well Samples

In the early 1980s, salt characterization was carried out on small samples from Tanks 1F, 10H, 18F, 19F, and 23H.³³⁻³⁴ Subsequently, small samples for criticality analysis were collected either from the saltcake surface or from wells mined into saltcake by the addition of water. Such samples were collected from Tanks 37H, 38H, 41H, and 46F.^{28, 35-42} Examining the as-received salt samples in Table 3-6, all of the aluminum concentrations are less than the aluminum concentration of the Tank 31H sample in Table 3-5. The insoluble solids from Tank 41H salt well sample T41-HTF-E-173, Tank 46F salt well sample T46FTF048-2, and Tank 37H salt well samples HTK-493 and 494 all were shown to have high concentrations of aluminum in the residual insoluble solids (25 to 30 wt%) and identification of aluminum hydroxide in the solids by XRD.^{28, 36, 42-43} The Tank 19F sample had the highest mass of dry residual insoluble solids per mass of saltcake, but the residual insoluble solids from Tank 19F in-tank dissolution were comparatively trivial.^{17, 34} All of the rest of the tank samples in Table 3-6 had a mass of residual insoluble solids below 2.5 wt% of the saltcake mass, with many samples exhibiting residual insoluble solids below 1 wt%. However, since it may take the precipitation of aluminum hydroxide several days to reach equilibrium, it is possible that the relatively quick dissolution tests of Table 3-6 did not provide ample time for aluminum hydroxide precipitation and thus the amount of insoluble solids may be biased low in some cases. In general, insoluble solids per saltcake dry mass data in Table 3-6 for the saltcake surface and salt-well sample results match well with the 1.36 wt% (dry basis) value obtained for the Tank 31H dissolution test discussed in Section 3.3.2.1.

Applicable to the Tank 9H saltcake composition in relation to Tank 10H saltcake, evaporator bottoms were dropped to Tank 10H and then sent on to Tank 9H for final cooling.³³ Along with sodium nitrate, which was the primary salt, this technique tended to drop out the less soluble salts such as carbonate and sulfate in Tank 10H, while leaving the more soluble materials such as hydroxide, nitrate, and aluminate primarily

for Tank 9H. Thus, there are reasons to expect the Tank 9H saltcake to have a higher aluminum concentration than the Tank 10H saltcake.

3.4 Evaluation of the Insoluble Slurry

In relation to developing the basis for undissolved solids resulting from saltcake dissolution, the following points have been noted, From Sections 3.2 and 3.3

- Tank 31H dissolution test provides a good overall basis for most tanks, with a relatively high aluminum in the saltcake (4.95 wt%), with the volume of residual solids (mostly aluminum hydroxide) remaining after dissolution corresponded to 5% of the initial saltcake volume.
- A Tank 25F dissolution test with a drained saltcake composite (1.7 wt% aluminum) was performed. Aluminum hydroxide precipitated from the first two dissolution steps, which corresponded to about 16 vol% of the saltcake dissolved overall. Some sample segments had locally higher aluminum, so this result is not bounding for every location in the tank.
- A dissolution test with a Tank 28F sample that had higher aluminum content than Tank 25F also produced bayerite solids upon dissolution.
- Dissolution of a Tank 41H sample taken from the surface of the saltcake after in-tank dissolution resulted in residual insoluble solids that were primarily aluminum hydroxide.
- Dissolution of salt well samples from Tanks 41H, 46F, and 37H showed solids with aluminum hydroxide, supporting the potential of in-tank formation of these phases during saltcake dissolution.
- Some tank samples and actual campaigns did not have appreciable aluminum hydroxide precipitation (Tank 19F actual campaign, Tank 3F, 10H, and 38H samples)
- A very high aluminum saltcake sample from Tank 29H (13.3 wt% aluminum) was dissolved and exhibited a volume of residual aluminum hydroxide that was about 75% of the volume of initial saltcake used in the dissolution test. Because there is evidence that this sample was taken from a salt well where in-tank salt dissolution had been performed, it is not expected to be representative of the bulk Tank 29H saltcake.
- For Tank 44F specifically, when oxides of the sludge metals are considered, the sludge content may contribute to a significant slurried sludge volume that would need to be considered in addition to any aluminum hydroxide present.

There are several challenges with interpreting saltcake insoluble solids data from dissolution tests and applying to in-tank saltcake dissolution. First, quantitative dissolution testing most conveniently provides a mass balance for amount of residual insoluble solids per amount of saltcake used. However, the desired format of data useful to in-tank salt dissolution is on a volume of insoluble solids resulting from a volume of salt. Because the saltcake tends to have a high solids content and the layer of residual insoluble solids appears to have a low insoluble solids content (at short settling times), some of the tests show a relatively large volume layer of residual insoluble solids with a low weight of insoluble solids. Second, many of the dissolution tests were run on core sample composites and thus provide results more applicable to the average tank composition and not specifically for a higher aluminum layer that would be expected to have a higher potential for aluminum hydroxide formation. Third, the greatest current need for a technical basis is for the potential for residual saltcake insoluble solids from the initial stages or batches of dissolution, while most of the data from dissolution tests is for the residual insoluble solids resulting from the entire salt dissolution process. Assuming that the saltcake is uniform, based on testing such as the Tank 25F sample dissolution test, it is conservative to expect that the formation of aluminum hydroxide will be most prevalent in the initial batches of salt dissolution. This bias toward aluminum hydroxide precipitation in early batches is expected to be most pronounced for dissolution methods that allow for flow of fluid through the saltcake when compared to saltcake dissolution in discrete layers. There is not a strong basis for relating the insoluble solids remaining after the entire dissolution process to quantify the degree of precipitation occurring during the initial stages of saltcake dissolution. Fourth, there is a possible sampling bias. Surface samples are more prevalent than other samples and do not appear to represent the entire tank. Based on the saltcake core sample information to date, it is difficult to determine if surface samples are representative of the bulk

saltcake aluminum content, or whether they are biased high or low with respect to the bulk aluminum content. Criticality samples pulled from a caisson or from an area of dissolution-mined salt are intended to be biased high in saltcake insoluble solids, potentially adding to their conservatism for this application.

The following are the assumptions considered when determining a realistic but biased high residual insoluble solids layer.

1. A layer of residual solids needs to be settled to a layer containing at least a threshold value of insoluble solids before it can retain gasses. If the slurry is too dilute in insoluble solids, there is little resistance to gas bubbles floating into and through the supernatant liquid and releasing into the tank atmosphere. The precise threshold value for insoluble solids is not known.
2. The density of saltcake is assumed to be constant during multi-stage dissolution. This is not precisely true for multiple reasons, including rewetting of drained saltcake and differences in densities of different insoluble components.
3. In-tank saltcake dissolution is an intermediate case between well-mixed dissolution (contact the entire tank with the dissolution fluid) and top-down dissolution (where only the salt from the very upper surface is being dissolved, and the saltcake is dissolved in discrete layers). Multi-batch dissolution experiments are typically performed as well-mixed dissolutions, with the exception of the flow-through dissolutions performed on the thin-wall core samples.
4. Undisturbed saltcake is assumed to not be dissolved and will not precipitate solids
5. Of the loose materials, slurried salts (such as sodium nitrate, sodium carbonate, sodium sulfate, burkeite, strontium carbonate, etc.) can be ignored as they are assumed to retain hydrogen in the same manner as the original saltcake. Saltcake tends to have a much lower pore volume than both sludge slurries or settled sludge.
6. Aluminum hydroxide formation is assumed to be related to the concentration of aluminum in the saltcake (salt solids and interstitial liquid). In actuality, aluminum hydroxide equilibrium solubility is also dependent on hydroxide and other anion concentrations. Additionally, factors that influence precipitation kinetics will impact the timing of aluminum hydroxide formation.
7. An assumed factor of 50% can be applied to a centrifuged gibbsite layer volume to relate it to an in-tank layer after several days of gravity settling.

The following two inputs are used to calculate/estimate the volume of a realistic but biased high residual insoluble solids layer resulting from the dissolution of a volume of saltcake at SRS.

1. From the Tank 31H saltcake dissolution test with realistically high aluminum content, 0.05 mL of residual insoluble solids remained per mL of saltcake used.
2. From the Tank 25F saltcake dissolution test, the aluminum hydroxide precipitation occurred during the first two of the three dissolution contacts. The first two dissolution contacts corresponded to the addition of 59 wt% of the inhibited water and removal of 60 wt% of the saltcake. With the simplifying assumption of constant saltcake density (assumption 2 above), the first two dissolution contacts correspond to removing approximately 60 vol% of the saltcake. Thus, the aluminum hydroxide was precipitated during the dissolution of 0.60 mL per mL of total saltcake.

In order to bias the precipitation of aluminum hydroxide toward the earlier dissolution stages for conservatism, the value from input 1 (0.05 mL residual insoluble solids/mL saltcake) is divided by the value from input 2 (0.60 mL of saltcake producing aluminum hydroxide per mL of saltcake). As a result, it is expected that initial precipitation of aluminum hydroxide could contribute to a settled layer of as much as 0.08 mL/mL of saltcake dissolved (8 vol%).

For the use of this calculated value, it is important to consider that there are several ways in which this evaluation may not be absolutely bounding. Five such potential non-conservatisms are presented and discussed below. First, several of the segments from Tanks 25F and 28F had higher aluminum concentrations than the Tank 31H sample, showing that the composition of the Tank 31H saltcake is not

absolutely bounding. The vast majority of the saltcake sample information from the thin-walled core samples and the saltcake surface and salt well cup samples support that the aluminum content of the Tank 31H sample is reasonable to use as a conservative case, though it is not absolutely bounding. Second, the settled layer from the Tank 31H test was centrifuged, likely leading to a more compact layer than would be initially encountered in in-tank saltcake dissolution and aluminum hydroxide precipitation. Margin should be considered to account for the application of the centrifuged solids volume to an in-tank settled solids volume. Third, there may be a larger bias toward precipitation in the early stages of dissolution than accounted for by the adjustment that was based on the first two dissolution stages of the Tank 25F dissolution experiment. Fourth, the precipitation of aluminum hydroxide is based on more than just the aluminum concentration in the saltcake. For example, if hydroxide concentration is particularly low during a specific in-tank salt dissolution versus the saltcake dissolution experiments, it is possible that a larger quantity of aluminum hydroxide could be precipitated in the tank. Fifth, due to the slow kinetics for gibbsite precipitation to reach equilibrium, some of the salt dissolution tests may have had more residual insoluble solids if they were allowed to equilibrate for longer. For saltcake dissolution tests, post-precipitation of aluminum hydroxide or salts would typically be noted if it was observed in the dissolved salt portions. However, most of the dissolution tests were not designed to specifically investigate this phenomenon.

It is recommended that margin be added to account for Tank 31H dissolution test experimental uncertainty (33%) and the application of centrifuged data to several days of in-tank settling (assumption 7, 50%). Multiplying the 0.08 mL residual insoluble solids per mL saltcake dissolved by the two adjustments, the resulting value with additional margin is 0.16 mL residual insoluble solids per mL of saltcake dissolved (16 vol%). Comparing 0.16 mL residual insoluble solids per mL of saltcake to other observations discussed in this report can give an indication of the potential representativeness or conservatism of this value. For example, the aluminum hydroxide precipitation from drained Tank 25F saltcake composite dissolution was also approximately 0.16 mL of insoluble solids per mL of saltcake. This evidence supports that the factor of 0.16 mL residual insoluble solids per mL of saltcake can be applied to multiple saltcake dissolution methods, including cases with and without interstitial liquid draining.

Some saltcake historically and currently at SRS has sludge layers above, below, or alternating with the saltcake. The values calculated for residual insoluble solids per volume of saltcake should apply to dissolution of saltcake with typical minimal amounts of entrained sludge but may not apply to saltcake with significant sludge.

It is expected that in the future, mechanical agitation methods may be needed for tanks with both saltcake and sludge. For example, the Tank 17F saltcake dissolution campaign used mechanical agitation to accommodate salt dissolution in a tank that had alternating layers of sludge and saltcake. Consideration should be taken for tanks like Tank 44F, where sampling indicated that a potential sludge layer may be present around the 110-inch tank level. Such a sludge layer may be large enough to challenge some of the assumptions in this evaluation.

4.0 Conclusions

A review of previous SRS saltcake dissolution and characterization studies was performed to inform the analysis of the amount of residual insoluble solids that may result from saltcake dissolution. A realistic value covering most cases for the maximum volume of a settled layer of residual insoluble solids that would result from dissolution of a volume of saltcake was calculated as 0.08 mL/mL (8 vol%). Applying factors to account for dissolution test uncertainty and differences between in-tank settling and laboratory testing, the recommended value for a settled layer of residual insoluble solids that would result from dissolution of a volume of saltcake is 0.16 mL/mL (16 vol%). This should apply to dissolution of saltcake with typical minimal amounts of entrained sludge but may not apply to saltcake with significant sludge. As it is composed of primarily non-radioactive isotopes, the precipitated aluminum hydroxide material does not contribute to the heat load.

5.0 Recommendations

Continue with literature search and, if warranted, a test program to determine the flammable gas retention and release quantities and mechanisms of residual insoluble solids from saltcake dissolution, especially the aluminum hydroxide phases. If possible, gather data to validate or improve assumptions in this evaluation regarding the relationship between the weight basis and the volume of settled aluminum hydroxide insoluble solids. If it is desired to suppress the formation of aluminum hydroxide during saltcake dissolution, consider including higher levels of sodium hydroxide in the saltcake dissolution fluid.

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