

Savannah River Site Saltcake Retrieval Simulation

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ABSTRACT

A major objective for Savannah River Site (SRS) tank farm operations is the separation of actinides and cesium from salt wastes to permit the processing of the high activity waste fraction in the Defense Waste Processing Facility (DWPF) and stabilization of the lower activity waste as saltstone. Towards this end, efforts are currently underway for the development of the Salt Waste Processing Facility (SWPF) containing the Actinide Removal Process (ARP) and Caustic Side Solvent Extraction Unit (CSSX). Any stream blending will be performed upstream of the SWPF. The Institute for Clean Energy Technologies (ICET) has assisted the site through research aimed at assessing stream stability for blended compositions arising from potential tank farm operations. The primary streams of concern are the DWPF Recycle stream, which consists of DWPF overheads and is routed to the tank farm, high aluminum concentration streams from sludge leaching operations (Batch 5 leachate), and dissolved salt streams originating from saltcake dissolution. Simulation of the salt cake dissolution process was performed using an ESP model (Environmental Simulation Program, V8.0, OLI Systems Inc). The simulation was constructed to evaluate stream parameters during the process operations of saltcake dissolution, blending for corrosion control, and introduction into the CSSX process. Saltcake dissolution was simulated using the DWPF recycle stream as diluent. These simulations indicated that dissolution using the DWPF recycle stream was effective and could replace the use of inhibited water. The resulting dissolved salt streams were cooled and evaluated for solids formation and adherence to corrosion specifications. Calculations were performed evaluating the effectiveness of using the Batch 5 leachate for corrosion control of non-compliant streams. The results showed that the addition of leachate in significant volume percentages was sufficient to raise corrosion inhibition to minimum requirements. Finally, the introduction of the corrosion compliant dissolved salt fractions into the CSSX process was modeled to determine the effect of waste stream carryover into the 0.05M HNO₃ CSSX scrub section. In particular, solids formation was evaluated versus waste stream carryover. The predicted solids, as expected, were related to process stream pH values, with Gibbsite and Dawsonite as the predominant species.

INTRODUCTION

High level wastes (HLW) at SRS fall into three broad categories: sludge wastes, salt wastes, and a mixture of the two. Salt wastes are a result of the concentrated aqueous streams from evaporator processing of waste streams which have been routed to the tank farm. These wastes, once in the tanks, cool and precipitate solids and the result is a saltcake consisting of the solids and an interstitial saturated salt aqueous solution. Initial reclamation of these wastes for disposal involves the re-dissolution of the saltcake with an inhibited water solution. The dissolved salt streams are further processed in the SWPF which contains the ARP for actinide removal and the

CSSX for cesium removal. The low activity waste stream exiting the CSSX process will be stabilized as saltstone while the high activity waste streams from both the ARP and CSSX units will be sent to the DWPF for vitrification.

In preparation for the planned start up of the full-scale SWPF in 2011, a number of efforts are addressing waste acceptance criteria and the performance of individual unit operations [1, 2]. Since most of the SRS waste streams, with the exception of high activity sludge batches destined for the DWPF, will be processed through the SWPF, the associated compositions of these streams, either alone or blended together, will have a direct impact on the stability of the waste within the SWPF. Of particular concern is the thermodynamic prediction of solids precipitation within the CSSX process. In this process, the extractive solvent, after being contacted with the waste stream for removal of cesium, is scrubbed with a 0.05 M HNO₃ solution. The purpose of this scrub step is to remove co-extracted sodium and potassium ions, as well as, extracted nitrate and hydroxide counterions. The removal of these species improves the effectiveness of the cesium extraction which occurs in the subsequent stripping section [3]. Carryover of waste from the CSSX contacting section into the scrub and stripping sections increases the risk of solids precipitation which could severely impact the mechanical operation of the CSSX process contactors. The potential for solids precipitation can be assessed through thermodynamic modeling as well as experimental studies specifically designed to examine solids re-precipitation.

The Institute for Clean Energy Technology (ICET) at Mississippi State University has been assessing streams stabilities across the salt waste reclamation process with the exception of the ARP. The highly saturated conditions, along with the wide variety of elemental components, greatly complicate the prediction of solution chemistry and solids precipitation within process streams. Much of the work conducted at ICET has focused on developing an understanding of the salt chemistry found in Hanford and SRS tanks. A number of experiments have been performed and have led to the development of the V7DBLSLT thermodynamic database for use with the OLI Systems Inc. Environmental Simulation Program (ESP). This work consisted of extensive solubility measurements of specific sodium salt systems at temperatures and ionic strengths typical of the site waste [4, 5]. Recent efforts have focused on the use of DWPF recycle for saltcake dissolution, the use of Batch 5 leachate from sludge processing for corrosion inhibition, and in determining the potential for solids precipitation within the CSSX scrub contactors [6, 7]. To this end, ESP simulation models have been constructed to predict stream compositions from the dissolution and subsequent CSSX processing of SRS tanks 25F, 31H, 37H, and 38H.

MODELING APPROACH

All simulations conducted as part of this study have utilized the ESP 8.0 (Build 58, OLI Systems Inc) program. The equilibrium model was constructed using the Public, Corrosion, and V7DBLSLT databases and does not include kinetic calculations. No vapor or organic phases were allowed and redox reactions were not simulated. SRS tank compositions were gathered from reported data [8-10] and input into the ESP format as molecular species. A comparison of relative major groups is shown in Fig. 1. It can be seen that a significant range of group compositions is covered within the chosen tanks.

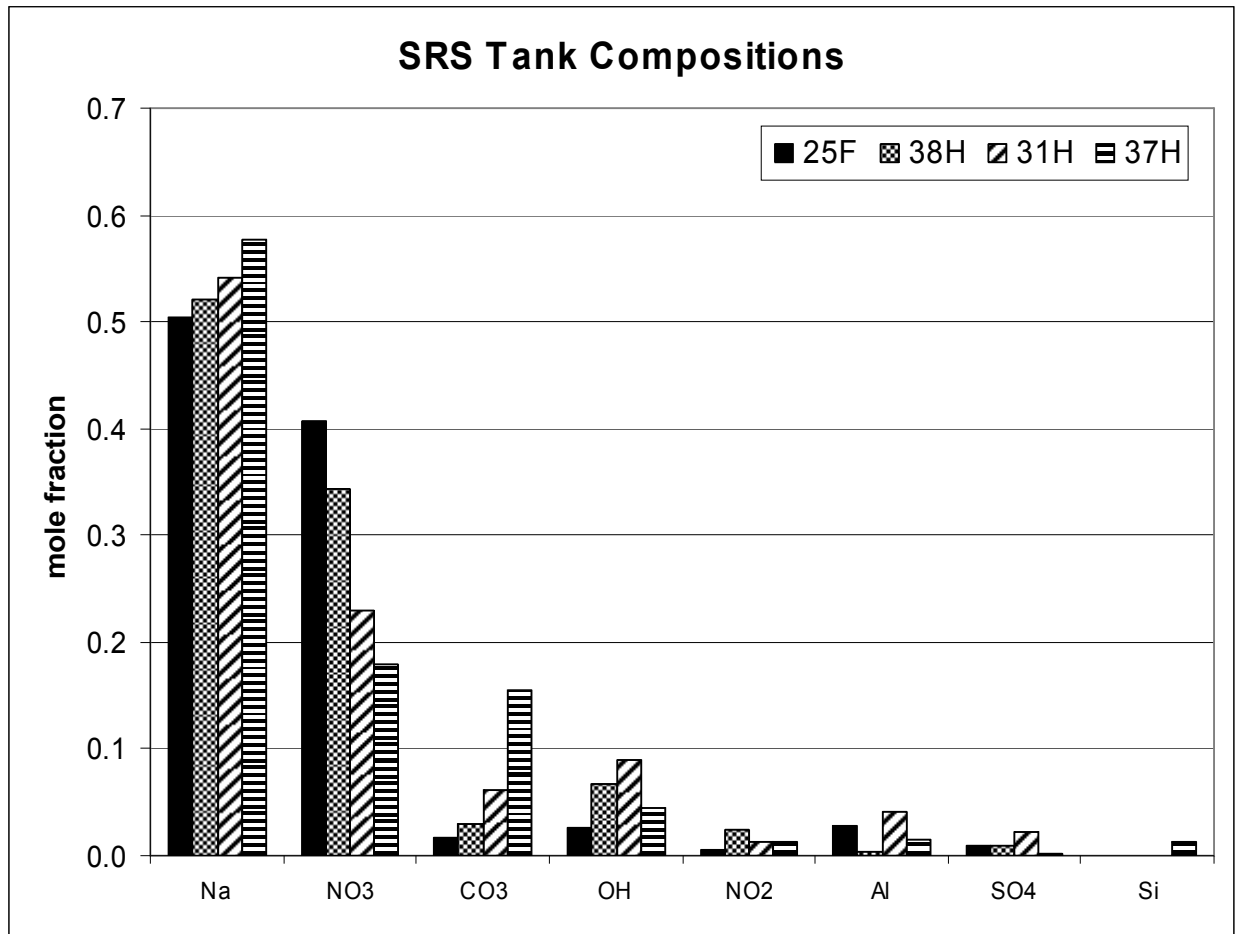


Fig. 1. SRS Initial Tank Compositions

A simple block flow diagram of the entire process, as modeled, is shown in Fig. 2. The removal of actinides and strontium by sorption onto monosodium titanate (MST), as planned prior to the CSSX process, was not modeled. In addition, any processing post-CSSX was not considered.

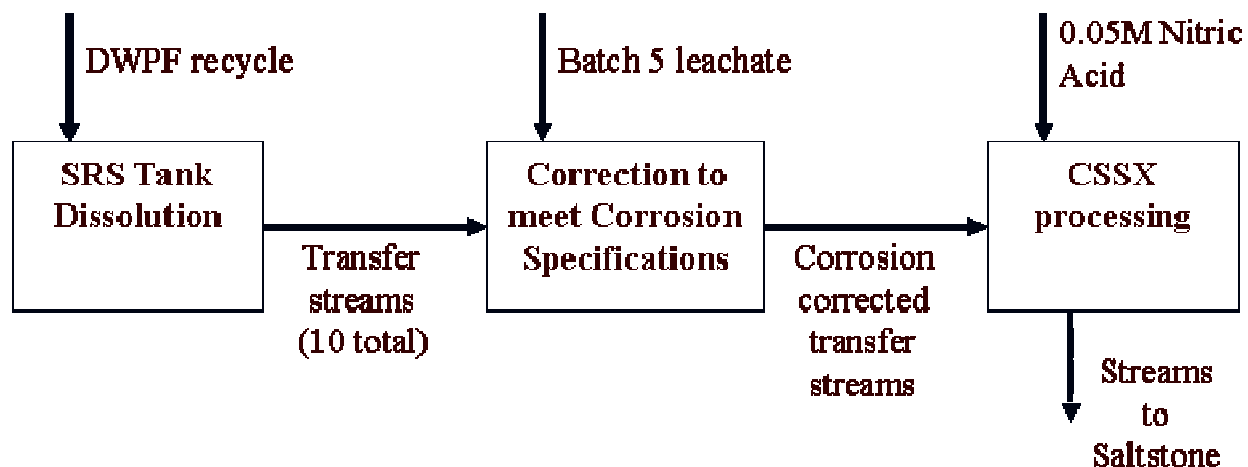


Fig. 2. Modeled Process Block Flow Diagram

SIMULATION RESULTS AND DISCUSSION

Salt Dissolution

After input of tank composition data into the ESP model, the saltcake dissolution process was modeled in increments based on the amount of flush utilized as a weight percent of initial tank total contents. For this study, each increment was 10% of the original contents and was given a corresponding stream number. For example, dissolution stream 1 results from the dissolution of the tank waste in which the flush amount is 10% of the original tank contents. Stream 2 denotes the dissolution fraction resulting from the subsequent flush utilizing an additional flush amount equaling 10% of the original tank contents (20% dilution by weight total), and so on. In this way, the total dissolution effluent was divided into discrete fractions that could be analyzed for composition, as well as, precipitation potential in subsequent processing. Simulations for each tank were carried out at 30°C up to 100% diluent addition by weight. As depicted in Fig. 2, the flush medium was the alkalized DWPF recycle stream with a predicted pH of 13.48 and ionic strength of 1.3. This stream is collected from process operations in DWPF as a byproduct of vitrification. It is currently the largest volume source fed to the tank farm [11]. The ability to use this stream in the dissolution process would significantly reduce the quantity of inhibited water required, as well as, eliminate the need to treat much or the entire DWPF recycle stream. Table I lists the major components of the recycle stream as utilized in the process simulations.

The dissolution process for SRS Tank 38H is represented in Table II as the amount and type of solids remaining during dissolution up to 100% diluent addition. In this tank, the major solids present are NaNO_3 and $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$. It is seen that the nitrate solids are more quickly dissolved than the other species and are completely removed by 100% flush addition. Additional recycle stream is necessary for complete removal of the $\text{Na}_2\text{C}_2\text{O}_4$ while a very small amount of Cr_2O_3 shows no tendency to dissolve.

Table I. Reduced molecular composition of the DWPF Recycle Stream (pH=13.48) [6, 7].

species	mole fraction
H ₂ O	9.79E-01
Na ₂ O	1.07E-02
HNO ₂	7.16E-03
CO ₂	2.04E-03
HNO ₃	1.09E-03
HCOOH	4.72E-05
Al ₂ O ₃	2.49E-05
SiO ₂	8.81E-06
SO ₃	4.04E-06
HgO	2.73E-06
HF	1.06E-06
MnO	3.15E-07

Table II. Solids remaining in SRS Tank 38H during 0% to 100% (by weight) dissolution using DWPF Recycle stream as dissolving medium.

species	0	20	40	60	80	100
	gram moles					
NaNO ₃	48,216,852	37,932,377	27,479,786	17,104,605	6,448,643	0
Cr ₂ O ₃	2,356	2,356	2,356	2,356	2,356	2,356
Na ₂ C ₂ O ₄	407,110	402,252	393,106	383,064	372,994	352,962
Na ₂ CO ₃ •H ₂ O	3,990,456	3,360,088	2,221,419	960,506	0	0
Na ₃ FSO ₄	45,957	46,579	34,359	20,215	6,955	0
Na ₆ SO ₄ CO ₃	629,386	550,990	452,979	356,059	237,965	0
Na ₇ F(PO ₄) ₂ •19H ₂ O	12,484	12,455	12,391	12,290	12,160	0
total solids	53,304,600	42,307,100	30,596,400	18,839,100	7,081,080	355,326

Tank 38H contains mostly NaNO₃ solids with a much lesser amount of Na₂CO₃•H₂O. In contrast, the waste in SRS Tank 37H contains higher levels of carbonate and silica, Fig. 1. Table III depicts the dissolution of the waste in 37H. It is seen that, initially, there exist roughly equivalent amounts of NaNO₃ and Na₂CO₃•H₂O. As in tank 38H, the nitrate is removed first, while the remaining solids dissolve more slowly. Only about half of the Na₂CO₃•H₂O, and approximately 2/3 of the total solids, is removed through 100% dissolution. In addition, further hydration of the Na₂CO₃•H₂O to Na₂CO₃•10H₂O is predicted at approximately 100% (wt)

dissolution. With the exception of the NaNO_3 and $\text{Na}_6(\text{SO}_4)_2\text{CO}_3$ solids, all remaining species require dissolution in excess of 100% (by weight) for removal.

Table III. Solids remaining in SRS Tank 37H during 0% to 100% (by weight) dissolution using DWPF Recycle stream as dissolving medium.

species	0	20	40	60	80	100
	gram moles					
NaNO_3	15,912,690	9,822,204	3,657,866	0	0	0
$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	17,286,688	16,844,103	16,173,823	14,590,416	11,480,270	8,229,983
$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	0	0	0	0	0	424,521
$\text{Na}_6(\text{SO}_4)_2\text{CO}_3$	108,187	58,813	0	0	0	0
NATROLITE	403,802	405,210	399,217	394,027	387,473	379,756
$\text{NAPHOH} \cdot 12\text{H}_2\text{O}$	210,335	210,270	210,095	208,809	173,866	116,397
Total Solids (kg)	3,773,920	3,182,630	2,550,280	2,040,570	1,638,780	1,331,860

Transfer Stream Corrosion Control

The transfer streams generated by the dissolution process will be collected and stored, for some minimum amount of time, pending further processing by the MST and CSSX technologies. It is also assumed that the streams will cool from the dissolution temperature of 30°C . A temperature of 23°C was arbitrarily chosen for all post-dissolution simulations. Therefore, it becomes important for these streams to be adjusted to a state which conforms to corrosion control specifications. These specifications set minimum levels of nitrite and hydroxide based on the nitrate concentration. The ESP simulation model was used to evaluate the addition of the Batch 5 leachate from sludge processing, in place of caustic, to ensure adequate corrosion inhibition. The simulated composition of this leachate stream, which has a pH of 14.7 and an ionic strength of 5.37, is shown in Table IV. Fig. 3 shows the effect of leachate addition on corrosion compliance for Tank 25F dissolution transfer streams, which have been cooled to 23°C . In this plot, the minimum requirement for corrosion compliance is shown as the red line. While transfer streams 1 through 3 (corresponding to 10 to 30%, by weight, dissolution) are within corrosion specification without treatment, a leachate addition rate of 20% (by volume) is required to ensure corrosion compliance for the remaining streams. A similar relationship is observed in each of the other SRS wastes that have been simulated; 31H, 37H, 38H. The first streams obtained from dissolution contain higher concentrations of nitrate, hydroxide, and nitrite, which maintain adequate corrosion inhibition levels. As the dissolution proceeds, and the NaNO_3 tank solids are removed, these concentration levels fall until minimum corrosion inhibition requirements are no longer met. In addition, as the transfer stream nitrate concentration continues to decrease, the minimum requirement for corrosion protection also changes, as is seen in Fig. 3.

Table IV. Reduced molecular composition of Batch 5 Leachate Stream (pH=14.7) from sludge processing [6, 7].

species	mole frac
H ₂ O	0.939417
Na ₂ O	0.042947
HNO ₂	0.006516
Al ₂ O ₃	0.005376
HNO ₃	0.004159
HgO	0.000515
CO ₂	0.000513
SO ₃	0.000333
HCl	0.000090
H ₂ C ₂ O ₄	0.000045
MnO	0.000035
HF	0.000029
K ₂ O	0.000019
P ₂ O ₅	0.000006

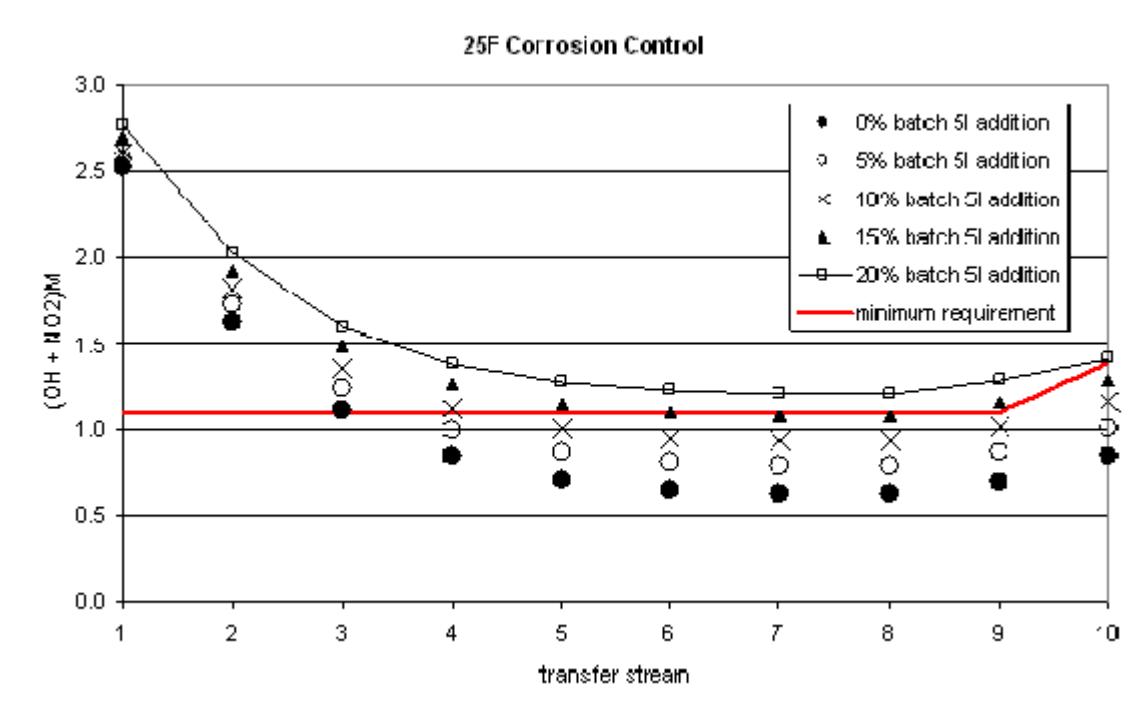


Fig. 3. Effect of Batch 5 Leachate addition to Tank 25F Dissolution Transfer Streams (23°C)

In addition to the achievement of corrosion compliance, the elevation of the hydroxide and nitrite ion concentrations by the addition of Batch 5 leachate helps prevent precipitation of solids due to cooling of the transfer stream from 30 to 23°C. The relationship between solids precipitation (in weight %) and leachate addition is shown in Table V. Since all dissolution transfer streams were initially 100% aqueous at 30°C, the total solids shown for 0% leachate addition are the result of stream cooling to 23°C. The solids precipitated in streams 1 through 8 are NaNO₃ and are predicted in significant amounts. For streams 9 and 10, the solids are comprised of Gibbsite, Al(OH)₃, and are predicted at much lower levels. It can also be seen that the addition of leachate for transfer streams 1 through 8 results in a significant reduction in total solids as the additions rate is increased to 10% (volume). This reduction is from the dissolution of NaNO₃. Further additions show small increases in total solids which are comprised almost completely of Gibbsite. Transfer streams 9 and 10 do not show significant solids formation upon cooling which indicates that NaNO₃ solids are not predicted. For these streams, addition of leachate results in a small increase in the predicted solids, Gibbsite.

The relationship observed for leachate addition to 25F for prevention or reduction of solids precipitation generally holds for simulations involving other SRS tanks; 31H, 37H, 38H. Significant solids precipitation from cooling is predominantly a result of NaNO₃ formation and can be significantly reduced by the addition of leachate. Transfer streams generated later in the dissolution process are less likely to precipitate NaNO₃ due to lower stream nitrate concentrations. Therefore, the effect of leachate addition on higher dissolution stream fractions will depend on other stream ions such as aluminum, silica, and/or carbonate.

Table V. Effect of Batch 5 Leachate Addition on Selected Tank 25F Dissolution Transfer Stream Total Solids.

leachate addition (% by volume)	1	4	7	8	9	10
	total solids (% by weight)					
0	3.67	3.06	2.90	3.09	0.08	0.15
5	1.94	1.49	1.39	1.62	0.17	0.32
10	0.66	0.22	0.17	0.27	0.24	0.44
15	0.73	0.27	0.23	0.24	0.31	0.54
20	0.78	0.32	0.28	0.29	0.36	0.63

CSSX Processing

As seen in Fig. 2, dissolution streams, after cooling to 23°C, addition of leachate to ensure corrosion compliance, and removal of precipitated solids, were modeled as feed to the CSSX process. Table VI lists the amount of leachate addition utilized for corrosion control for transfer streams from each tank. As stated earlier, the MST process for removal of actinides and strontium was not modeled. The CSSX process contacts the waste stream with a specialized organic solvent to remove cesium [3, 12]. Fig. 4 represents the portion of the CSSX process, which is examined in this report. The organic solvent passes through the extraction and scrub section of CSSX which is comprised of several in-line centrifugal contactors.

Table VI. Transfer Stream Corrosion Control Adjustments for SRS Tanks Prior to CSSX Simulations.

38H	31H	37H	25F
Batch 5 leachate addition (volume %)			
15	15	10	20

The Cs, along with small amounts of tank waste, is removed from the bulk tank waste stream in the extraction section. The scrub section uses 0.05M HNO₃ to remove the carryover tank waste while the Cs is transported, with the solvent, into the next section of the process. The ESP model was constructed to evaluate the effect of the tank waste carryover into the scrub section contactors. The tank waste carryover into the CSSX scrub section was modeled as a percentage, by weight, of the mixture of waste and 0.05M HNO₃ seen in the scrub section contactor. For example, a 1 gram carryover of waste into 99 grams HNO₃ is considered 1% carryover. In particular, the potential for solids precipitation in the scrub contactors was simulated.

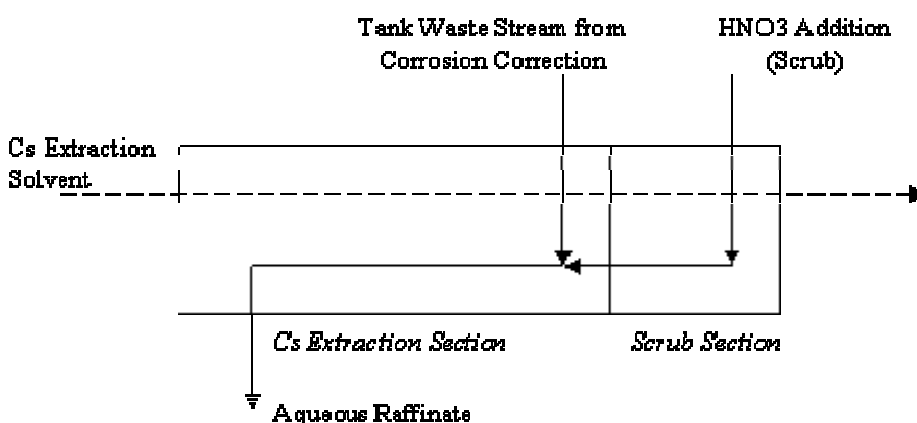


Fig. 4. CSSX (Cs Extraction and Scrub)

Relatively minor carryover of the high pH transfer streams into the CSSX scrub section HNO₃ stream results in significant elevation of the HNO₃ stream pH. Fig. 5 shows the effect of waste from Tank 38H transfer streams 1, 4, and 7 on the pH of the scrub section HNO₃. At a waste carryover rate of 1% (by weight), the pH of the HNO₃ stream has increased from 1.38, for the neat stream, to approximately 2.0 for the contaminated stream. For transfer stream 1 which originates from initial saltcake dissolution, a slightly higher carryover rate of 2% dramatically increases the scrub stream pH to 11.0. Transfer streams 4 and 7, which are the dissolution stream fractions from 40% and 70% (by weight dissolutions) require a higher carryover rate, approximately 7.0%, to produce the same pH effect. The transfer stream carryover versus pH

relationships shown in Fig. 5 are very similar to those simulations conducted for tanks 25F, 31H, and 37H.

ESP simulations of waste carryover were also used to predict the type and quantity of solids precipitation versus HNO₃ stream pH. Fig. 6 shows the result of the simulation for waste carryover of transfer stream 4 from tank 25F. The mole fraction of the predicted solids are plotted, as well as, the total solids (in weight %). Four regions can be approximated across the pH range shown. The first region defined by waste carryover that results in a scrub stream pH below about 4.0, predicts the generation of UO₂ solids in amounts which are essentially zero, 10⁻⁷ weight %. In the approximate pH range of 4.0 to 6.0, Gibbsite is predicted in measurable amounts of between 0.01 to 0.03 weight%. In the third pH region, 6.0 to 10.0, Dawsonite, (NaAlCO₃(OH)₂), is predicted in quantities equaling 0.5 weight%. For scrub stream pH values above 10.0, Gibbsite returns as the only predicted solid. Simulation with transfer stream 7 carryover resulted in the same approximate pH regions and predicted solids. Transfer stream 1 deviates from Fig. 6 in that Gibbsite is the only solids shown and is predicted in similar quantities for all pH regions above 4.0. Modeling of tanks 31H, 37H, and 38H shows solids formation involving Gibbsite and Dawsonite in the same approximate regions and in similar quantities as observed in tank 25F. Tank 37H, which contains higher levels of carbonate and silica than each of the other tanks, exhibited a minor degree of Quartz formation concurrently with the Gibbsite in region 2, and the Dawsonite in region 3.

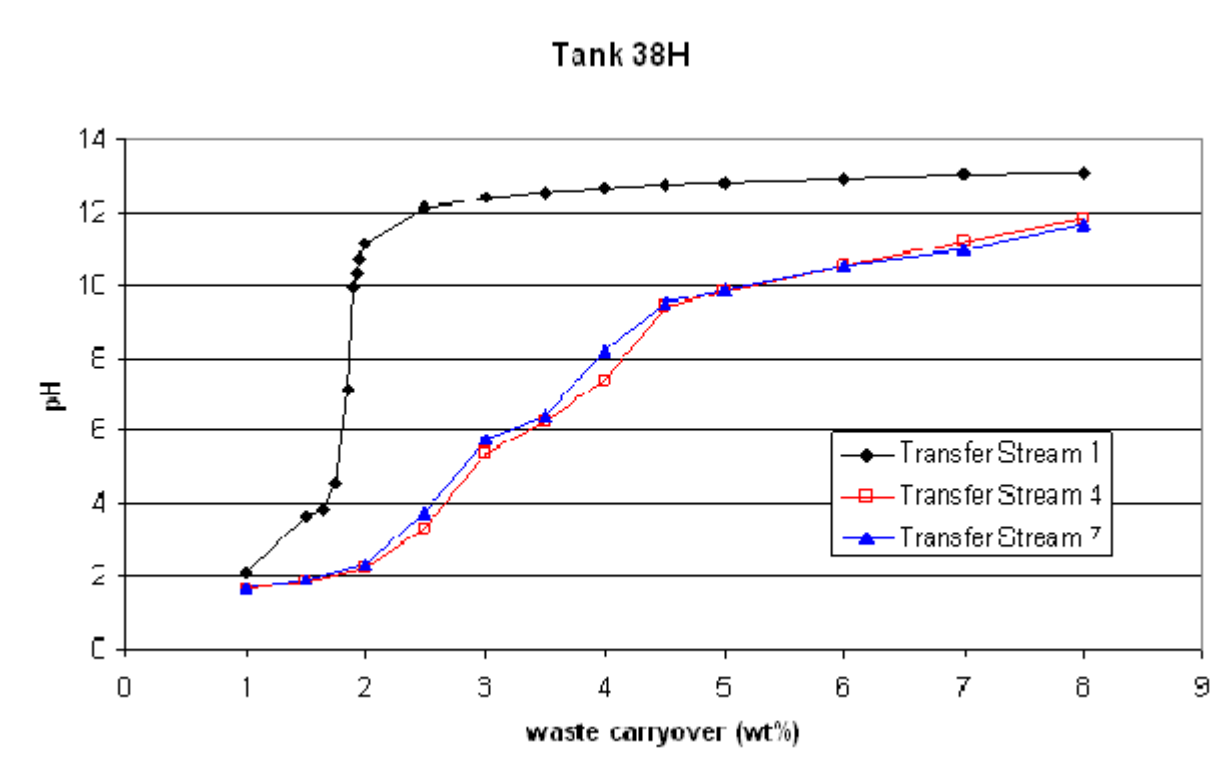


Fig. 5. Effect of Waste Carryover from Transfer Streams 1, 4, and 7 on pH for SRS Tank 38H

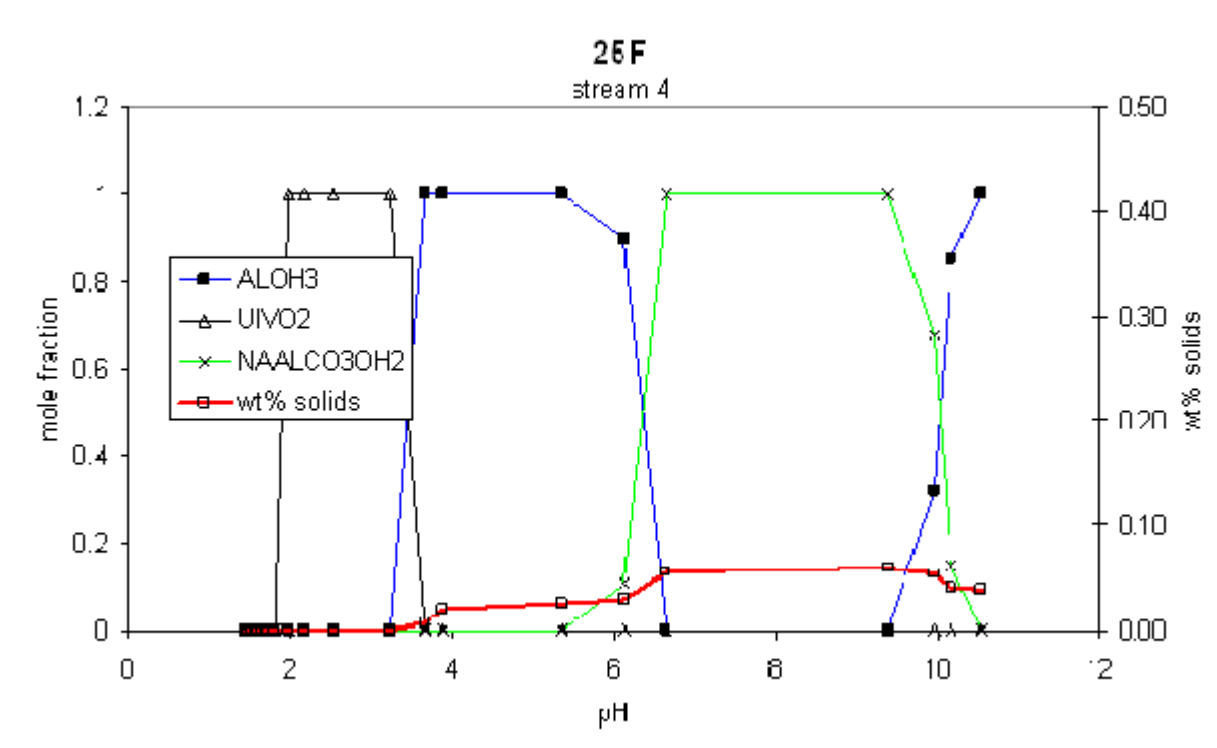


Fig. 6. Effect of pH on Precipitated Solids Type and Amount for Tank 25F Stream 4 Waste Carryover into CSSX Scrub Section.

CONCLUSIONS

An equilibrium model was developed to simulate tank waste dissolution, corrosion control adjustment, and CSSX scrub section waste carryover for SRS tanks 25F, 31H, 37H, and 38H. The model utilized the ESP OLI thermodynamic equilibrium program with no kinetic calculations.

The waste dissolution process was modeled with DWPF recycle as the dissolving medium. Dissolutions were in discrete fractions corresponding to 10% (by weight) of the initial tank contents and over the range of 0% to 100% diluent addition by weight. Results indicated that dissolution using the DWPF recycle stream was effective and could replace dissolution with inhibited water. Sodium nitrate tends to always dissolve first. Other solid species, such as carbonates, are not as easily dissolved as are nitrates and if present in elevated levels will potentially require dilution above 100% (wt).

Dissolution stream fractions, denoted as transfer streams, were cooled to 23°C and evaluated for corrosion compliance based on stream nitrate, nitrite, and hydroxide concentrations. Formation of solids upon cooling was identified as to species and quantities. Batch 5 leachate from sludge processing was evaluated as an additive for corrosion compliance and solids reduction. Simulations suggest that the addition of leachate in volume percentages between 10% and 20% are sufficient to raise corrosion inhibition to minimum requirements. Also, leachate addition to

transfer streams which require no corrosion adjustment assists in the reduction of solids precipitated due to cooling. This is a result of re-dissolution of nitrate solids.

The aqueous fractions of corrosion compliant streams, at 23°C, were modeled as feed to the CSSX process and the effect of waste carryover into the CSSX scrub section was evaluated. Simulations for each tank produced similar results for waste carryovers in the range of 1% to 8% (by weight) for pH and solids formation. Initial dissolution fractions caused increases in scrub stream pH to values of 11.0 for carryovers as low as 2% (wt). Later dissolution fractions required higher carryover rates, 7%, to produce the same effect. Predicted solids formation appears to occur in 4 approximate regions based on scrub stream pH. For streams with pH values below 4.0, predicted solids quantities are essentially zero (10^{-7} weight percent). The region of pH values between 4 and 6 showed measurable quantities, 0.01 to 0.03 weight percent, of Gibbsite formation. Likewise, for pH values above 10, Gibbsite was predicted. The region with pH values between 6 and 10 varied according to dissolution fraction, with Gibbsite predicted for earlier dissolution streams and Dawsonite predicted for later dissolution stream fractions. Recent test results reported for small-scale CSSX contactors have indicated that waste carryover rates in the scrub section are below 1% (by weight) [13]. Carryover rates simulated in this study significantly exceed 1% in order to cover a more complete scrub stream pH range. Although outside the scope of this study, contactor flow patterns could produce concentration gradients which result in localized pH values corresponding to carryover rates significantly above 1%.

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