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**Sludge Heel Removal by Aluminum Dissolution  
at Savannah River Site – 12390**

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**ABSTRACT**

High Level Waste (HLW) at the Savannah River Site (SRS) is currently stored in aging underground storage tanks. This waste is a complex mixture of insoluble solids, referred to as sludge, and soluble salts. Continued long-term storage of these radioactive wastes poses an environmental risk. Operations are underway to remove and disposition the waste, clean the tanks and fill with grout for permanent closure. Heel removal is the intermediate phase of the waste retrieval and tank cleaning process at SRS, which is intended to reduce the volume of waste prior to treatment with oxalic acid. The goal of heel removal is to reduce the residual amount of radioactive sludge wastes to less than 37,900 liters (10,000 gallons) of wet solids. Reducing the quantity of residual waste solids in the tank prior to acid cleaning reduces the amount of acid required and reduces the amount of excess acid that could impact ongoing waste management processes. Mechanical heel removal campaigns in Tank 12 have relied solely on the use of mixing pumps that have not been effective at reducing the volume of remaining solids. The remaining waste in Tank 12 is known to have a high aluminum concentration. Aluminum dissolution by caustic leaching was identified as a treatment step to reduce the volume of remaining solids and prepare the tank for acid cleaning. Dissolution was performed in Tank 12 over a two month period in July and August, 2011. Sample results indicated that 16,440 kg of aluminum oxide (boehmite) had been dissolved representing 60% of the starting inventory. The evolution resulted in reducing the sludge solids volume by 22,300 liters (5900 gallons), preparing the tank for chemical cleaning with oxalic acid.

**INTRODUCTION**

**Tank History**

Tank 12 is a 2.84 million liter (750,000 gallon) Type I carbon steel waste storage tank at SRS. The tank was constructed with twelve internal support columns of 0.6 meter (2 foot) diameter to support the roof, and approximately 5800 meters (19,000 feet) of 5 cm (2 inch, schedule 40 carbon steel pipe) horizontal and vertical cooling coils. There are 9 main riser openings in the roof for tank access and deployment of equipment and instrumentation.

The tank was commissioned in 1956 and was used to receive and store modified PUREX and THOREX process waste from the H Canyon facility from October 1956

through May 1973. The waste received had two components: an alkaline liquid component and a solids component of metal oxides and hydroxides (sludge). The sludge solids were allowed to separate from the liquid by gravity settling and accumulated in the bottom of the tank. The liquid supernatant was routinely decanted and transferred out of the tank for evaporation.

Waste receipts were discontinued when the first tank leak site was discovered at an elevation of 2.67 meters (105 inches) above the tank bottom in 1974. Supernatant was removed to a level below the leak site. Over the next several years, the residual liquid fraction was allowed to evaporate. The height of the remaining material in the tank settled at 1.88 meters (74 inches). The total volume of dried sludge solids was estimated to be 770,000 liters (203,000 gallons). The material in the tank was undisturbed until sludge removal efforts were initiated in 2006.

The waste removal approach was to slurry and suspend solids through vigorous mechanical mixing and transfer the slurry to another tank. Four long-shafted mixing pumps, configured with two horizontal opposing discharge nozzles, and a submersible transfer pump were installed in the riser openings of the tank. The mixing pumps had been relocated from a previous tank with several thousand run hours. The mixing pump suction was initially situated just above the surface of the settled solids. Approximately 28,200 liters (74,500 gallons) of sodium nitrite solution was added to the tank in four batch intervals from late 2004 to early 2005 to rewet the dried sludge solids.

Supernatant was transferred into the tank for initial sludge suspension, and mixing efforts were started in August 2008. Mixing pumps were operated for 7 days, and a slurry sample was obtained for material characterization and treatment testing. The slurry sample exhibited a very thick, tacky behavior. Soundings (a steel wafer attached to a measuring tape and lowered into the tank) were performed to detect the sludge level and evaluate the extent of solids suspension, and the mixing pumps were lowered further into the tank to continue suspension efforts. The initial transfer out of the tank was attempted in December but was unsuccessful in moving the slurry. An additional slurry sample was obtained to measure the fluid properties. The slurry exhibited a yield stress of 45 Pascal. [1] The slurry was diluted with additional supernatant and the first successful transfer out was performed in March 2009.

Two separate bulk waste retrieval campaigns were performed to provide feed for sludge batch preparation and ultimate vitrification in the Defense Waste Processing Facility (DWPF). A total of ten liquid addition, mixing and transfer evolutions were conducted between September of 2008 and August of 2010. Mixing pumps were operated for approximately 10 days for each evolution with the entire pump assembly continuously rotated to alter the pump discharge nozzle orientations within the tank. Following the last transfer out of the tank, a video inspection was performed which identified, mounded sludge sediment in four major areas along the tank wall; the largest mound existed along the east wall in a dense area of internal cooling coils. The sludge volume was reduced to approximately 83,300 liters (22,000 gallons) and bulk waste removal efforts were declared complete. [2]

The heel removal phase is the intermediate phase prior to acid cleaning and was initiated as an extension of bulk waste removal. The mixing strategy was altered to direct (index) the pump discharge nozzles at the mound locations to disperse the solids. During this first campaign in December 2010, the mixing pump in the northeast quadrant began leaking excessive amounts of bearing water (indicating mechanical seal failure) shortly into the campaign and was inoperable. Mixing continued with the remaining three pumps and the slurry was transferred out.

The leaking pump was replaced in February of 2011 and a second heel removal campaign was initiated. During this effort, two additional mixing pumps began leaking excessive amounts of bearing water and were unable to be operated. Mixing efforts continued with the two operating pumps and the slurry was transferred in March. Video inspection identified that three mounds were still prominent along the west, north and east tank walls. The volume estimate of remaining sludge solids in the tank was 51,900 liters (13,700 gallons). [3] Mechanical heel removal efforts were suspended due to reduced mixing capability.

Figure 1 displays the sludge waste removal history from Tank 12 during the bulk waste and mechanical heel removal campaigns.

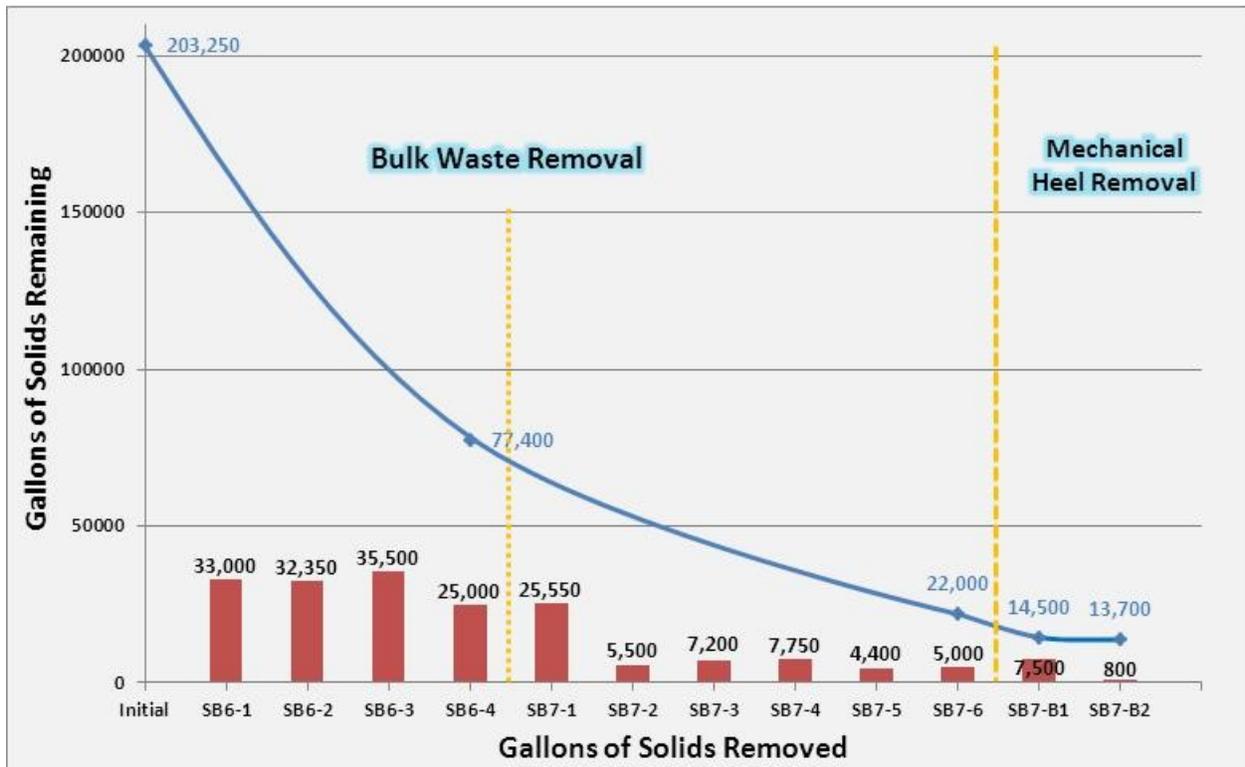


Figure 1. Sludge waste removed from Tank 12 during bulk waste and heel removal campaigns.

## **BASIS FOR TREATMENT SELECTION**

### **Impacts of Continued Mixing**

As a result of the failed mixing pumps and the physical properties of the sludge solids, continued mixing and transfer campaigns were inefficient at reducing the remaining solids volume. The diminishing returns during the two heel removal evolutions demonstrated an inability to appreciably affect the mounded areas in the tank with only two pumps, and only a small volume of solids were transferred out.

A single spare mixing pump was on hand, however, a second replacement pump to provide the maximum mixing capability was not available for at least seven months. In order to resume mechanical mixing efforts, if pursued after pump replacement, several additional transfers would have been necessary and integrated with ongoing activities. A dedicated tank for transfer receipts would also have been necessary to store the sludge solids until the next processing window.

Schedule delays associated with pump replacement, receipt tank availability, and transfer coordination would negatively impact the regulatory commitment date for ultimate tank closure.

### **Impacts of Excess Oxalic Acid**

Chemical treatment of sludge heels using oxalic acid solution has been demonstrated to be an effective technique for removal of residual material. However, the liquid waste system is not able to accommodate a large quantity of oxalate solids. The spent oxalic acid solution must be neutralized so that it can be safely stored in carbon steel tanks. In order to neutralize the acid stream, sodium hydroxide (either from supernatant or purchased sodium hydroxide) is needed to adjust the solution pH to 12 or greater. The result of the neutralization is the re-precipitation of dissolved oxides and hydroxides and the formation of solid sodium oxalate.

The solid sodium oxalates formed in the neutralization tank are eventually transferred along with the bulk sludge solids into a sludge batch to be fed to DWPF. The treatment process for preparing sludge feed involves removing the soluble salts by washing with water. Sodium oxalate is a sparingly soluble material that only begins to readily dissolve at low sodium concentrations (less than 2.5 M) and is relatively soluble below 1.25 M. The oxalate solids in the sludge complicate batch characterization and results in additional sludge washing cycles.

Wash water from sludge preparation is transferred to steam evaporators for volume reduction and then stored in waste tanks. Increased wash water volume increases the demand on evaporator systems and tank space for storage. Additionally, the dissolved oxalate eventually ends up re-precipitating in either the evaporator concentrate or evaporator feed tank, and the solids will build up in the evaporator tanks and appear to be salt. This will reduce the space available for actual salt in the system and will

eventually result in an evaporator concentrate tank becoming unusable. The oxalates have also been identified as a contributor to periodic plugging problems during evaporator operation.

Eventually, waste removal is performed on the evaporator system tanks. The solid sodium oxalate will be removed with the sludge solids. During sludge batch preparation, the solids will again dissolve and end up in the evaporator system. If the spent wash water is used to dissolve salt, then the oxalate will precipitate in the salt tank. If the spent wash water is used to adjust salt batch molarity, then the oxalate will precipitate in the salt batch blend tank. If the spent wash water is returned to a sludge hub tank, then the solids will re-precipitate and require removal during the next sludge removal operation.

Although the desired end condition for heel removal efforts is less than 37,900 liters (10,000 gallons) of sludge solids, the heel volume should be reduced to the extent practical.

### **Benefits of Aluminum Dissolution**

Prior sample analysis of the Tank 12 sludge in support of Sludge Batch 6 indicated the predominant form of aluminum oxide to be the monohydrate (boehmite). The mass fraction of boehmite in the total insoluble sludge solids was found to be at least 78%. Previous demonstrations on the Tank 12 sludge resulted in effective dissolution of aluminum. First, a caustic dissolution test was performed by Savannah River National Laboratory (SRNL) on the 3 liter sample at a process temperature of 60°C and was successful in dissolving approximately 60% of the aluminum oxides over a period of 26 days [4]. The post dissolution sample exhibited a significant reduction in slurry yield stress and a slight reduction in mean particle size. Second, aluminum dissolution was performed on a large batch of Tank 12 sludge to prepare feed for DWPF. The evolution was conducted at a process temperature of ~70°C over an 8 week period and resulted in a 72% reduction of total aluminum [5].

In addition to reducing the volume of solids remaining in the tank prior to acid cleaning, this treatment method could provide additional benefits. The high caustic effluent stream could be removed in a single transfer and is compatible with the carbon steel waste tanks and the liquid waste system chemistry. The aluminum rich supernatant stream could also be readily fed through the salt decontamination process and dispositioned in the Saltstone facility, and could be utilized to augment hydroxide adjustment of the salt feed. Dissolving some of the sludge heel would also reduce the amount of waste to be vitrified in DWPF.

An aluminum dissolution campaign to reduce the remaining heel was proposed as an opportunity to reduce the remaining sludge volume, affect the sludge physical properties and reduce the impacts of increased oxalates without delaying schedule.

## METHOD

### Flowsheet

A flowsheet was developed to establish necessary operating conditions and evaluate process effectiveness. The form of aluminum in the sludge was assumed to be all boehmite. The mass of boehmite was estimated from the remaining heel volume (51,900 liters), the volume fraction of actual sludge in wet solids based on previous experience (30%), sludge density (2.4 kg/L), and projected mass fraction of aluminum in sludge based on previous sample analysis (78%). The mass of boehmite in the remaining sludge heel was estimated to be 29,120 kg.

Initial flowsheet development was based on experience gained from previous dissolution campaigns performed. A kinetic model that was developed for dissolving aluminum from sludge based on previous dissolution test data, in tank demonstration data and literature data, is shown in Equation 1 [4].

$$t = \frac{\alpha \cdot F(wf, \alpha)}{\eta \cdot A \cdot C_{OH}^0} e^{\frac{14800}{T}} \quad (\text{Eq. 1})$$

where:  $t$  is the dissolution time in hours or days if divided by 24 hour per day,  
 $\alpha$  is the mole ratio at initial conditions of free hydroxide ion in the liquid phase relative to aluminum in the solids phase,  
 $\eta \cdot A$  is a group of constants representing the effects of specific surface area of boehmite solids and the variation of sodium hydroxide activity on the boehmite dissolution rate constant,  
 $C_{OH}^0$  is the liquid phase concentration of free hydroxide prior to aluminum dissolution in moles per kg water,  
 $T$  is the dissolution temperature in Kelvin,  
 $wf$  is the weight fraction of initial aluminum remaining in the solids after dissolution, and  
 $F(wf, \alpha)$  describes the variation of aluminum concentration in the liquid phase as a function of time for a given set of initial conditions.

One premise that the model is based on is that the solids were in complete suspension and the entire particle surface area was in contact with the dissolution medium. Additionally, the model was based on evolutions where the supernatant was being continuously mixed so there would be no localized areas of high aluminum concentrations that would slow the reaction rate. Although it has been demonstrated that aluminum will dissolve even when only limited surface area is exposed, the rate at which dissolution occurs was uncertain. Effectiveness would be dependent on the penetration depth of the supernatant into a mound of sludge solids (interstitial liquid) and the ability to sweep supernatant with higher aluminum concentration away from mounds and expose fresh waste. Still, the model served as a benchmark for

WM2012 Conference, February 26 – March 1, 2012, Phoenix, Arizona, USA

determining initial conditions and evaluating mixing effectiveness or “surface dissolution” during the evolution.

Preliminary target conditions were set based on experience and previous evolutions. A target process temperature of 70°C was proposed. A minimum hydroxide concentration of 3.2 M was targeted. An initial goal of 75% dissolution was established to provide a projection of time required to conduct the evolution. The model projection for the duration necessary was 86 days. A goal of 60% dissolution was projected to require 56 days. This duration was projected to be reduced by 40%-50% if a temperature of 75°C was maintained. [5]

### **Operational Strategy**

Because a spare mixing pump would not be available for several months, the evolution would be performed with only three mixing pumps. Although not effective to fully suspend the remaining sludge particles, the existing pumps were located closest to the remaining mounds and were believed to be adequate to continuously “sweep” the heated caustic across sludge mound surfaces, dissolving the boehmite in the outer layer and exposing additional solids. Pumps would be run to focus the discharge nozzle at the mounds.

The minimum tank volume of 430,000 liters (114,000 gallons) was necessary to provide the 1.07 meters (42 inches) net positive suction head (NPSH) for mixing pump operation. Rather than filling the tank with fresh water and adding caustic to the desired hydroxide concentration, concentrated recycle water from DWPF with a hydroxide concentration of approximately 3.5 M would be utilized to supplement the amount of caustic necessary. Additionally, the supernatant would be used as part of a batch for salt processing.

The dissolution rate is highly dependent on the reaction temperature; therefore, maximizing the solution temperature would have the largest influence on effectiveness. Excess caustic was also proposed offset dilution from pump bearing water leakage and would also help promote reaction kinetics.

The ability of the three pumps to maintain tank temperature was also uncertain. An operating scenario was developed that would aggressively run the evaporator without cooling to the drop tank to concentrate and pre-heat the supernatant. Heat of dilution from the additional caustic would also increase temperature. Transfer of the supernatant into Tank 12 would be accomplished with a steam jet that would further promote pre-heating of the dissolution medium. Activities for the initial supernatant transfer, and the delivery and unloading of additional caustic were closely coordinated to minimize temperature loss.

Two months were allotted to conduct the evolution. This was the duration available following replacement of one of the failed pumps and ability to accommodate the

supernatant into the salt process. Periodic samples of the supernatant would be analyzed to provide indication of dissolution progress.

## RESULTS AND DISCUSSION

### Mixing Pump Operation

Aluminum dissolution of the Tank 12 sludge heel was initiated in June 2011 and performed over a period of two months. Hot evaporator concentrate was used to provide the initial heating of the dissolution medium and supplement the hydroxide concentration to reduce the amount of caustic added. Following the additions of heated supernate and before the caustic was added to the tank, a baseline sample was taken after the three mixing pumps were operated in a full rotational mode at maximum speed (1695 RPMs) for approximately two hours. The mixing pumps were restarted after sampling and ran for the entire two day duration of caustic additions. Six tanker trucks of 50 wt% caustic were added with a total volume of 79,500 liters (21,000 gallons).

The aluminum dissolution process performed several mixing campaigns, with each campaign designed to erode sludge/solid mounds in a particular quadrant of the tank and to mix and suspend as much solids as possible. After the completion of caustic additions, the first long term tank mixing campaign commenced. For a period of nine days, the mixing pumps ran at maximum speed in both fully rotational mode and indexed, where the mixing pump's discharge nozzles were aimed at known sludge/solid mounds to help in sludge erosion. This tank mixing method of repetitively suspending the solids by way of mixing the tank in the rotational mode and then followed by an aggressive indexing campaign that concentrated on a certain area of the tank where sludge/solid mounds were known was repeated throughout each mixing campaign.

The sample result of 03-Aug indicated a slowing rate of dissolution attributed to diminished mixing effectiveness. The later mixing campaigns were modified to promote a uni-directional vortex flow around the outer tank wall. Table I provides a summary of the pump run campaigns.

Table I. Tank 12 Heel Dissolution Mixing Pump Summary.

Run End Date	Pump Run Campaign	Run Time (H)	Cumulative Run Time (H)	Strategy
04-Jul	A	276	276	Mixers indexed at east mound
13-Jul	B	222	498	Mixers indexed at north mound
23-Jul	C	239	737	Mixers indexed at all mounds
03-Aug	D	247	984	Mixers indexed at east mound
16-Aug	E	139	1123	Mixers indexed to promote vortex
29-Aug	F	167	1290	Mixers indexed to promote vortex
31-Aug	G	44.5	1334.5	Mixers indexed to promote vortex

## Tank Temperature

The minimum tank liquid temperature for this process was targeted at 70°C, but higher temperatures were desired to increase the dissolution rate and overall effectiveness. The initial source of heat came from the transfer in of the pre-heated supernatant. Temperature readings at the sending tank indicated an initial temperature of 70°C. The steam transfer jet was expected to increase the temperature an additional 5 - 10°C. Although some cooling was anticipated when blended with the remaining heel in Tank 12, this provided a good starting point of 65°C.

The addition of the 50 wt% caustic increased the tank temperature from 65°C to 70°C. Mixing pumps continued to run following the caustic addition and provided sufficient heat to continue to increase tank temperature steadily.

Tank temperatures primarily ranged between 75°C and 85°C throughout the evolution. Cooling coils were periodically used to control tank temperature below the mixing pump mechanical seal maximum operating temperature. The tank temperature profile for the entire evolution is shown in Figure 2.

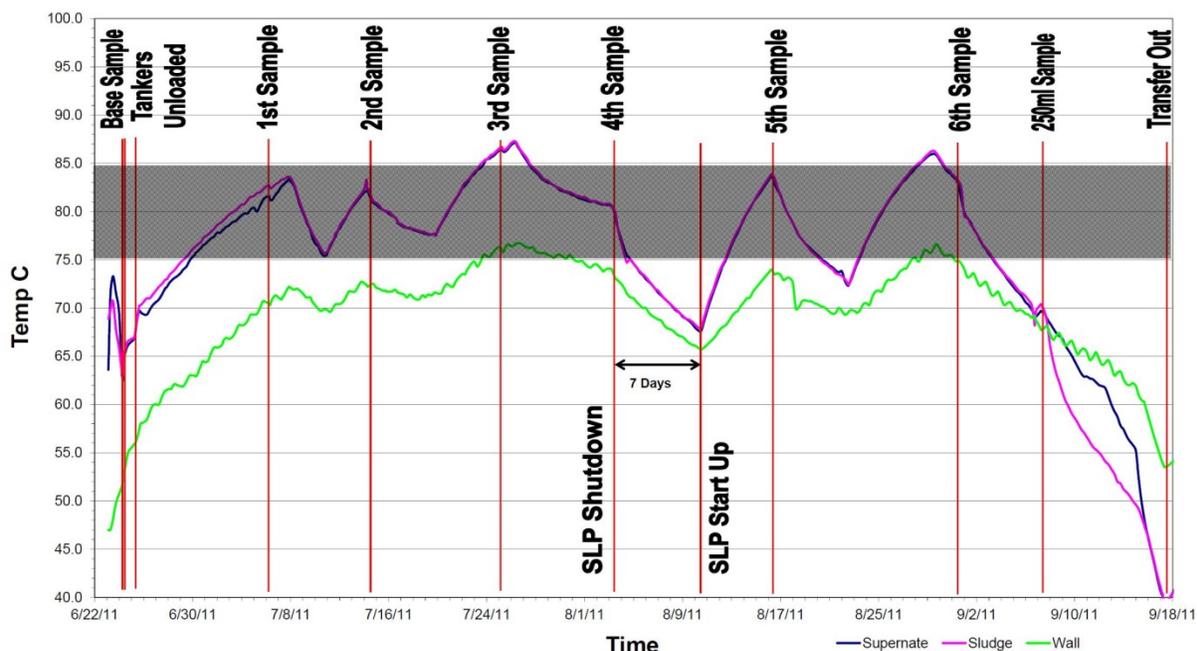


Figure 2. Tank and supernatant temperatures during dissolution campaign.

The duration of each mixing campaign was scheduled to be about 10 days in length. Actual mixing campaigns ran between 8 days to 13 days long. After each mixing campaign, a tank liquid sample was taken to monitor the progress of in-tank aluminum dissolution. The schedule was generated to perform a 10 day mixing campaign, turn off

pumps to obtain a tank sample, and then resume mixing operations for the next mixing campaign.

After the completion of the fourth mixing campaign, mixing operations were halted until the sample results were obtained. All tank cooling coils were isolated during this period to maintain the tank's temperature as high as possible. As Figure 2 indicates, the tank's temperature continued to decrease an additional 13°C. With positive sample results, the fifth mixing campaign commenced and after six days of mixing, the fifth sample was taken and mixing suspended.

After four days without tank mixing, the fifth sample results proved that some progress was still being made and therefore the sixth and final mixing campaign was run for a total length of 9 days. It should be noted that the fifth and sixth mixing campaigns were designed to promote a vortex action in the tank by aiming the mixing pumps discharge nozzles in such a way to promote this action.

After evaluation of the final sample indicating dissolution had slowed or ceased, mixing was suspended and the tank was allowed to cool prior to transferring the supernatant out on September 27, 2011.

### **Sample Data**

Periodic sampling and analysis was used to measure progress and determine the endpoint of the reaction. A total of seven samples were obtained throughout the evolution analyzing for aluminum and free hydroxide in the supernatant. In order to obtain rapid turnaround of analytical data and evaluate progress, these samples were analyzed at the F/H Process Laboratory.

In order to establish the initial conditions, a sample was obtained and analyzed prior to the addition of caustic. The initial baseline sample (25-Jun) was obtained prior to the addition of caustic; the values given in the table for aluminum and free hydroxide were adjusted to reflect initial conditions after the caustic addition. Data from the process sample taken after 11 days (06-Jul) indicated a rapid increase of aluminum in solution with a corresponding decrease in free hydroxide, which was likely due to dissolution of aluminum in the sludge layer distributed over the entire tank bottom (large surface area).

Data from the process sample taken 19 days into the evolution indicated dissolution was progressing but that the rate had slowed. Aluminum data reported in the 25-Jul sample was inconsistent with the previous samples. Conditions in the tank did not support any re-precipitation of aluminum compounds and, based on a substantial amount of time remaining from the original planned duration, mixing operations continued.

The final planned process sample was taken on 31-Aug. Mixing pumps were secured until results were returned and, if results indicated dissolution was still occurring, could

be restarted. After reviewing the sample data indicating a declining rate of reaction, the evolution was terminated and the tank was allowed to cool prior to transfer.

The analytical data is provided in Table II.

Table II. Tank 12 Heel Dissolution Supernatant Sample Data

Date	Cumulative Days	Tank Volume (L)	Aluminum (M)	Free OH <sup>-</sup> (M)
25-Jun	0	689,400	0.0496 <sup>a</sup>	5.355 <sup>a</sup>
06-Jul	11	689,400	0.3011	4.9281
14-Jul	19	689,400	0.3553	5.0100
25-Jul	30	691,400	0.3196	4.6558
03-Aug	39	691,400	0.3610	4.7470
16-Aug	52	685,300	0.4140	4.5538
31-Aug	67	680,100	0.3986	4.7085
07-Sep	74	673,000	0.450	NM <sup>b</sup>

<sup>a</sup> Calculated values based on sample data obtained prior to caustic addition.

<sup>b</sup> Free hydroxide was not measured.

The final sample taken (07-Sep) was analyzed at the Savannah River National Laboratory (SRNL). Although this sample was obtained for a different purpose, the soluble aluminum concentration of the supernatant was measured. The 0.45 M aluminum value reflects the dissolution of 16,300 kg of boehmite solids from the sludge heel. Additionally, based on decreasing measurements of silica in the supernatant, some sodium aluminosilicate compounds were likely formed and represent an estimated 140 kg of boehmite dissolved. The 16,440 kg of total boehmite dissolved represents 60% of the original estimated aluminum solids in sludge.

### Visual Inspection

During and after the transferring out of the supernate, the mapping of the settled solids of the tank bottom was performed. Sludge mounds were visibly impacted or eliminated in several areas of the tank. Mounds previously located along the north and south walls were nonexistent. The mound located along the west wall was noticeably reduced. Although a prominent mound still existed along the east wall, the overall size had been reduced. The estimated remaining volume of sludge solids from video inspection was reasonably consistent with the sample data.

Figure 3 provides a comparison of some “before” and “after” photographs taken from inside the tank.

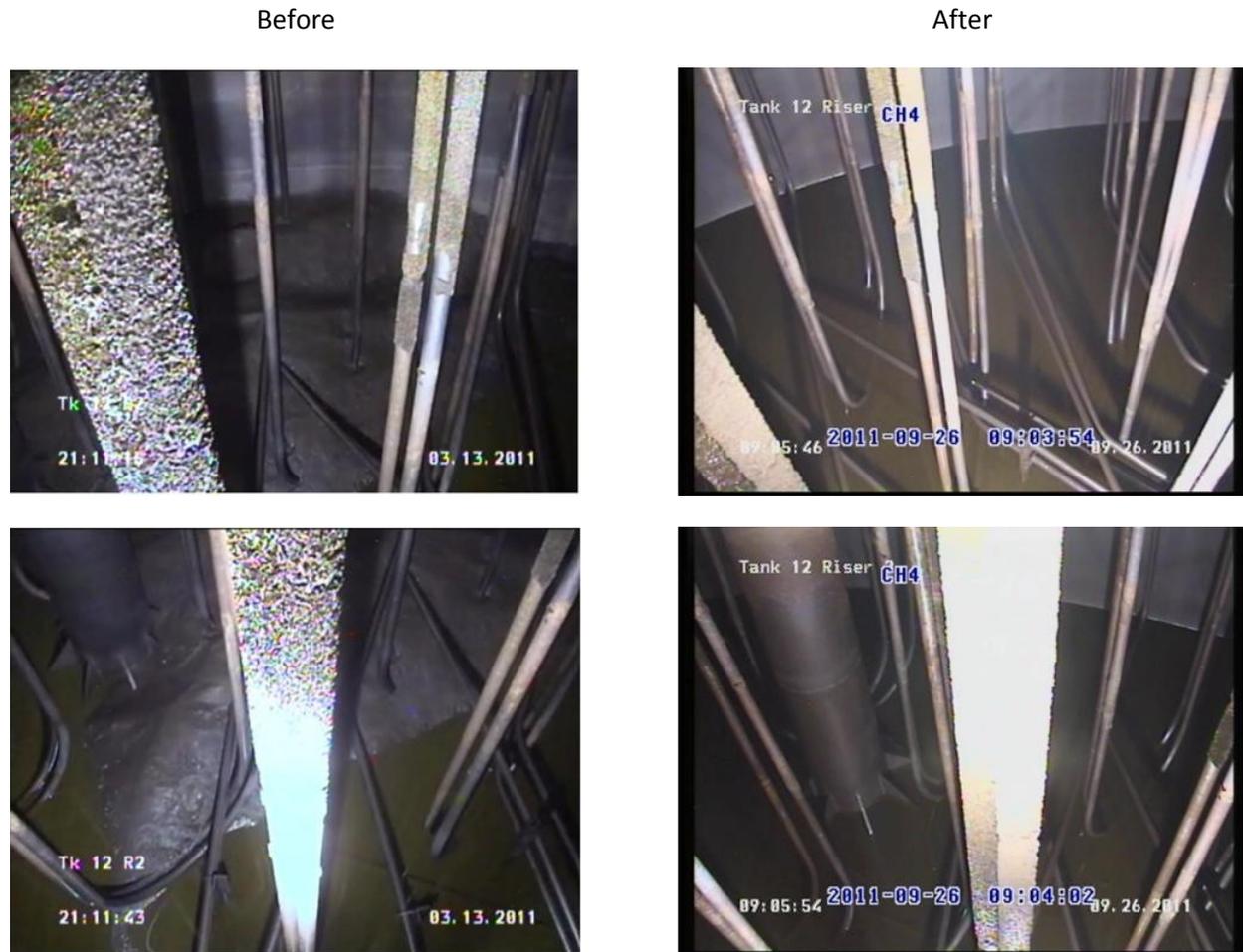


Figure 3. Tank 12 interior photographs before and after aluminum dissolution.

### Benefits and Future Application

This treatment method achieved a sludge mass removal of over 42% from the initial estimate (a 60% reduction in boehmite solids), reducing the remaining heel volume to approximately 29,500 liters (7,800 gallons) for acid dissolution of the remaining sludge and completion of tank cleaning in preparation for final closure.

The primary advantage of this treatment step is that, in general, existing equipment and procedures for chemical additions, tank mixing and heating, and transfers can be utilized. No special equipment is required and the predominant cost is associated with chemical procurement. Overall, the benefits of aluminum dissolution provide for a cost-effective alternate or additional treatment step to typical waste removal processes.

Additional benefits from the reduction of aluminum solids in downstream processes include fewer high level waste canisters generated, the ability to disposition the

WM2012 Conference, February 26 – March 1, 2012, Phoenix, Arizona, USA

aluminum-laden material in a salt batch for less expensive disposal in grout, a decrease in the volume of material to be processed in the Tank Farm evaporators, and a reduction in the receipt tank space required for Tank Farm operations.

Due to the successful implementation in Tank 12, this treatment step is being planned for the heel removal phases for Tanks 11 and 15, which have similar-type sludge. Future application should be considered for heel removal from additional SRS tanks, and Hanford tanks containing high aluminum sludge.

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