

# CHLORIDE WASHER PERFORMANCE TESTING

J. T. Coughlin  
D. R. Best

June 2007

Robotics, Remote & Specialty Equipment Section  
Engineered Equipment and Systems Directorate  
Savannah River National Laboratory  
Aiken, SC 29808

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Prepared for the U.S. Department of Energy Under Contract Number  
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**SRNL**  
SAVANNAH RIVER NATIONAL LABORATORY

**REVIEWS AND APPROVALS**

**AUTHOR(S):**

*Jerry T. Coughlin* 6/25/07  
 J. T. Coughlin, Remote & Specialty Equipment Date

*D. R. Best* 6/26/07  
 D. R. Best, Process Engineering Technology Date

**TECHNICAL REVIEWER:**

*Robert Pierce* 6/26/07  
 R. A. Pierce, Separations Science Programs Date

*Charles H. Brown* 6/26/07  
 C. H. Brown, HB-Line Engineering Date

**APPROVERS**

*A. P. Fellingner* 6/26/07  
 A. P. Fellingner, Manager, Remote & Specialty Equipment Date

*Sharon S. Marra* 6/26/07  
 S. L. Marra, Manager, Robotics, Remote & Specialty Equipment Date

*G. J. Zachmann* 6/26/07  
 G. J. Zachmann, Manager HB-Line Engineering Date

## EXECUTIVE SUMMARY

Testing was performed to determine the chloride (Cl<sup>-</sup>) removal capabilities of the Savannah River National Laboratory (SRNL) designed and built Cl<sup>-</sup> washing equipment intended for HB-Line installation. The equipment to be deployed was tested using a cerium oxide (CeO<sub>2</sub>) based simulant in place of the 3013 plutonium oxide (PuO<sub>2</sub>) material. Two different simulant mixtures were included in this testing -- one having higher Cl<sup>-</sup> content than the other. The higher Cl<sup>-</sup> simulant was based on K-Area Interim Surveillance Inspection Program (KIS) material with Cl<sup>-</sup> content approximately equal to 70,000 ppm. The lower Cl<sup>-</sup> level simulant was comparable to KIS material containing approximately 8,000-ppm Cl<sup>-</sup> content.

The performance testing results indicate that the washer is capable of reducing the Cl<sup>-</sup> content of both surrogates to below 200 ppm with three ½-liter washes of 0.1M sodium hydroxide (NaOH) solution. Larger wash volumes were used with similar results – all of the prescribed test parameters consistently reduced the Cl<sup>-</sup> content of the surrogate to a value below 200 ppm Cl<sup>-</sup> in the final washed surrogate material.

The washer uses a 20-micron filter to retain the surrogate solids. Tests showed that 0.16-0.41% of the insoluble fraction of the starting mass passed through the 20-micron filter. The solids retention performance indicates that the fissile masses passing through the 20-micron filter should not exceed the waste acceptance criteria for discard in grout to TRU waste.

It is recommended that additional testing be pursued for further verification and optimization purposes. It is likely that wash volumes smaller than those tested could still reduce the Cl<sup>-</sup> values to acceptable levels. Along with reduced wash volumes, reuse of the third wash volume (in the next run processed) should be tested as a wash solution minimization plan. A 67% reduction in the number of grouted paint pails could be realized if wash solution minimization testing returned acceptable results.

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**LIST OF ACRONYMS**

- KIS K-Area Interim Surveillance Inspection Program
- DOE United States Department of Energy
- RRSE Robotics, Remote & Specialty Equipment
- RASP Radionuclide Assay System - Photon
- SRS Savannah River Site
- SRNL Savannah River National Laboratory
- TRU Transuranic





## 1.0 INTRODUCTION AND BACKGROUND

The K-Area interim surveillance (KIS) inspection program necessitates the opening of a significant number of 3013 containers. After the K-Area inspection is completed, the container contents, principally PuO<sub>2</sub>, will be charged to the HB-Line dissolvers for dissolution. Timely processing of the material is important as significant delays will reduce vacancy in K-Area Lag Storage. Ample Lag Storage space must be available prior to material shipments to K-Area from other Department of Energy (DOE) sites.

Some of the 3013 containers to be processed contain PuO<sub>2</sub> with significant concentrations of Cl<sup>-</sup>. Before addition of this material to the HB-Line process, the Cl<sup>-</sup> content must be reduced. If the Cl<sup>-</sup> levels are not lowered and the material is charged to the process in its current state, there would be a greater risk of chloride-induced corrosion cracking damage to the process facility.

Initially, two different methods were considered to accomplish Cl<sup>-</sup> reduction in the 3013 material. Each method employed an aqueous wash solution to dissolve the Cl<sup>-</sup> salts, but differed in how the wash was separated from the surrogate. One method used a centrifuge while the other relied on filtration. The centrifuge-based device was eventually abandoned due to safety concerns surrounding use of rotating equipment in a glovebox environment<sup>1</sup>. Several different versions of the filtration concept were designed. Limited scoping tests<sup>2</sup> were performed by the Robotics, Remote & Specialty Equipment (RRSE) Section. Vacuum filtration was tested<sup>3</sup> and selected as the preferred method using a formal alternatives study<sup>4</sup> lead by HB-Line Engineering with attendance from other key HB-Line, WSMS and SRNL personnel.

RRSE took the preferred concept, completed equipment design, and followed the process through equipment fabrication. RRSE also tested the process equipment. This report documents the Cl<sup>-</sup> removal characteristics determined during testing. The testing work was initiated under a Task Technical Request<sup>5</sup> and defined in a Task Technical Plan.<sup>6</sup>

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## 2.0 APPROACH

### 2.1 Chloride Washing Testing

To determine the effectiveness of  $\text{Cl}^-$  removal capabilities of the Washer, full-scale equipment (Figure 2-1) was tested using a surrogate 3013 material. Samples of the wash solution and the surrogate were extracted at identified points in the process and submitted for  $\text{Cl}^-$  content analysis. By examining the  $\text{Cl}^-$  present in the samples, the effectiveness of the  $\text{Cl}^-$  removal could be evaluated for various test parameters.

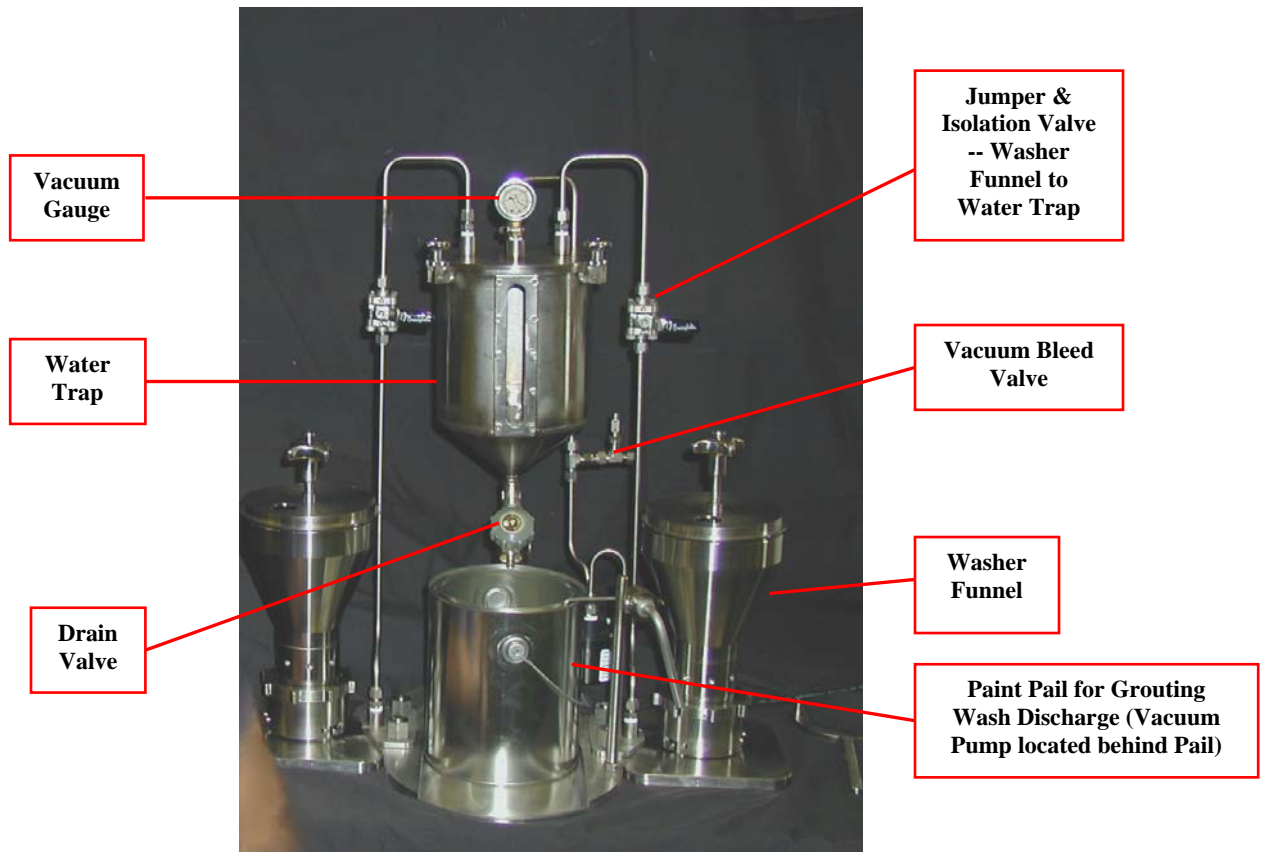


Figure 2-1 Chloride Washing Equipment Developed by RRSE<sup>7</sup>

### 2.2 Test Parameter Variables

It is known that the KIS material going to HB-Line possesses high  $\text{Cl}^-$  variability. Therefore, it is important that the washer's  $\text{Cl}^-$  removal performance be documented over a similar  $\text{Cl}^-$  range. Both high- and low-chloride simulants were prepared to address the greater part of the 3013  $\text{Cl}^-$  variability planned for receipt in HB-Line. The remaining test parameters (wash volume and number of washes) were adjusted to check  $\text{Cl}^-$  removal as a function of wash volume for low- and high-chloride simulants.

A total of eight Chloride Washer runs were completed. Table 2-1 lists the specific parameters used in each particular run. Data were analyzed to identify changes in  $\text{Cl}^-$  removal performance resulting from

parameter variation. The choice of parameters and run totals chosen for this testing was a joint decision between SRNL and HB-Line Engineering and subsequently documented in the Task Technical Plan.

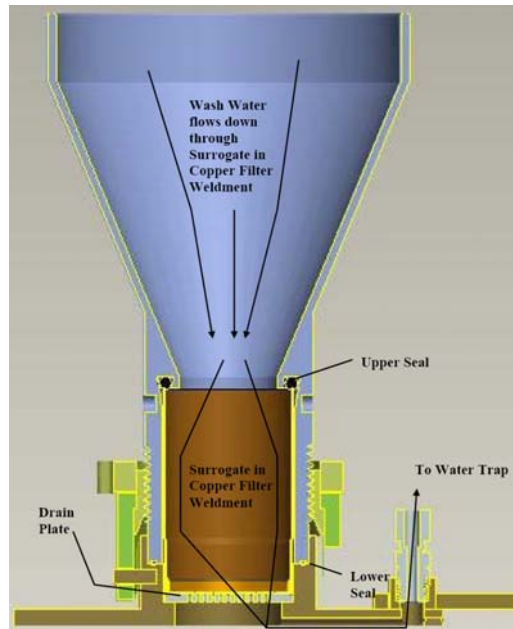
**Table 2-1 Test Parameters**

	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6	RUN 7	RUN 8
Surrogate Cl <sup>-</sup> Level	Low				High			
Number of Washes	3	3	3	5	3	3	3	5
Wash Solution Volume (L)	1/2	3/4	1	3/4	1/2	3/4	1	3/4

**2.3 Method used to Separate Soluble and Insoluble Fractions of the Surrogate Mixture**

The RRSE-designed washer uses an aqueous solution of 0.1 M NaOH<sup>a</sup> to dissolve the Cl<sup>-</sup> salts in the surrogate without dissolving PuO<sub>2</sub>. Once the wash solution is mixed with the surrogate, a vacuum pump is energized, drawing the wash solution through the 20-micron filter shown in Figure 2-2. Other information shown in Figure 2-2 is the location of the surrogate and the wash solution flow path to the water trap where the wash solution is collected.

The copper filter weldment is a formed copper tube that is electron-beam welded to a phosphor-bronze filter cloth. These materials were selected, as they readily dissolve once charged to the dissolver.<sup>8,9</sup> Liberated of the copper container, the PuO<sub>2</sub> is in full contact with the dissolver solution thus enhancing dissolution. Three advantages associated with the use of copper are: (1) MC&A issues are simplified, (2) materials are not withdrawn from the dissolver through the charge chute – deletes the operational use of a charge can and (3) a small reduction in TRU waste is realized.



**Figure 2-2 Cross-Section of the Washer Funnel Showing the Wash Solution Flow Path**

<sup>a</sup> NaOH is only required to ensure that the pH balance of the wash solution remains alkaline.

## 2.4 Surrogate Preparation Method

The testing and all results presented herein are based on surrogate material – that is  $\text{CeO}_2$  is used in place of  $\text{PuO}_2$ . Ceric oxide is commonly used in the Department of Energy Complex to simulate  $\text{PuO}_2$ . The surrogate was fired at  $950\text{ }^\circ\text{C}$  for at least two hours. It was thought that  $950\text{ }^\circ\text{C}$  oxide may make  $\text{Cl}^-$  removal more difficult compared to the same material fired at a lower temperature.<sup>10,11</sup> Furthermore, the 3013 Standard requirements dictate that most of the KIS material will be fired at  $\sim 950\text{ }^\circ\text{C}$ .

Two  $\text{Cl}^-$  levels were used in the performance testing. Enough surrogate material was prepared to complete four runs at each  $\text{Cl}^-$  level. Each surrogate mixture prepared per the work instruction<sup>12</sup> includes the approximate amount of  $\text{Cl}^-$  salt that would be found in 500-gram quantities of either 10,000-ppm or 80,000-ppm KIS material<sup>b</sup>. The constituent quantities included in each surrogate mixture are listed in Table 2-2. The values show the relative quantities of each component mixed prior to the last furnace heating. It is likely that minor  $\text{Cl}^-$  losses occurred in the furnace.

**Table 2-2 Mixture Quantities Used in each Surrogate per Chloride Washer Run**

	LOW-CHLORIDE SURROGATE	HIGH-CHLORIDE SURROGATE
Compound	Mass(g)	Mass(g)
$\text{CeO}_2^c$	305.4	264.5
Salt <sup>d</sup> (44% NaCl+56% KCl)	9.2	73.6
$\text{Fe}_2\text{O}_3^{e,4}$	6.5	6.9

An appreciable density difference exists in the insoluble parts of the surrogate and the actual KIS material. The  $\text{CeO}_2$  is  $7.13\text{ g/cm}^3$  while the  $\text{PuO}_2$  is  $11.46\text{ g/cm}^3$ .<sup>13</sup> This difference causes a much larger volume (and thickness) of insoluble surrogate material to remain after washing<sup>f</sup>. It is desired that the filter cake thickness of the surrogate and KIS material be similar. To minimize the difference, the mole fraction of the insoluble to the soluble parts of the surrogate mixture was set equal to that found in the KIS material being simulated. As a result, the total starting mass of the surrogate was less than the 500 g mass of KIS material it represented.<sup>g</sup>

The  $\text{Cl}^-$  salt mass, prior to calcination was equal to that contained in the full 500 g mass at the two  $\text{Cl}^-$  levels mentioned above. It is likely that minor  $\text{Cl}^-$  losses occurred in the furnace reducing the equivalent KIS material equivalent content to an average of about 70,000 ppm for the high-chloride material and about 8,000 ppm for the low-chloride.

A schematic diagram for each test is shown in Figure 2-3. The schematic is entered at the point the initial surrogate is prepared and exited when the surrogate is added to the Chloride Washer. It should be noted that the relative constituent masses of each run are well known when the run is mixed, but after the two-

<sup>b</sup> Chloride volatilization during the final  $950\text{ }^\circ\text{C}$  furnace surrogate preparatory step reduced the salt mass a small amount from the initial concentration.

<sup>c</sup> Prepared in accordance with RSE-WI-2007-0004 Section 1.

<sup>d</sup> Prepared in accordance with RSE-WI-2007-0004 Section 2.

<sup>e</sup> The ferric oxide was added because it might simulate the presence of plutonium trichloride if it interacted with the NaCl-KCl.

<sup>f</sup> This statement assumes that equal starting total and soluble masses existed in both materials compared.

<sup>g</sup> It should be noted that depending on the actual bulk density of the KIS material, it may not be possible to fit 500 grams in the Copper Filter Weldment (Figure 2-2).

hour 950 °C furnace hold, a fraction of the Cl<sup>-</sup> will volatilize.<sup>h</sup> The initial dry surrogate Cl<sup>-</sup> analysis data presented within Table 3-4 indicate a Cl<sup>-</sup> loss on the order of 5-20%.

## 2.5 Sample Withdrawal Activity Prior to Surrogate Placement in Washer

After the furnace run, a sample was removed and labeled “Partial Sample” (see Figure 2-3). A second sample labeled “Remainder of Sample” was also collected after weighing. The second sample removal was added to the first partial sample. This occurred because extra material was needed to ensure sufficient quantity existed for the analysis of three aliquots per sample – a decision made after the first sample removal, necessitating the second partial withdrawal. The starting weight of the material added to the Washer was then corrected for any sample removal, as indicated by the last “Run Weighed” block shown directly before the “To Washer” block.

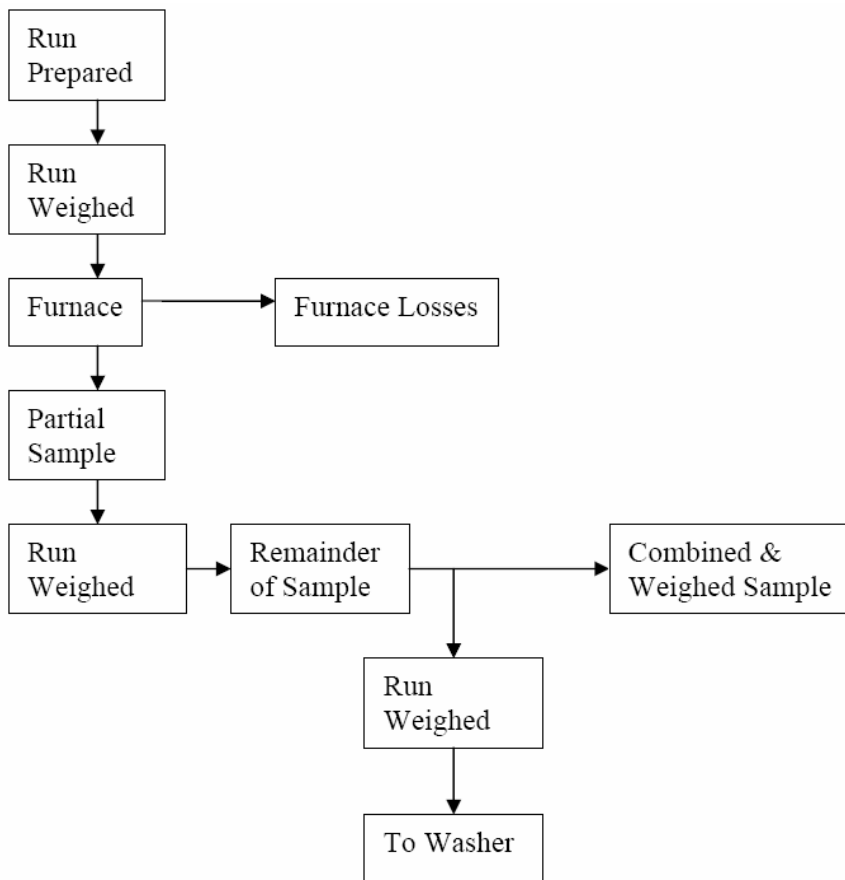


Figure 2-3 Schematic of Surrogate Handling Prior to Placement in Washer

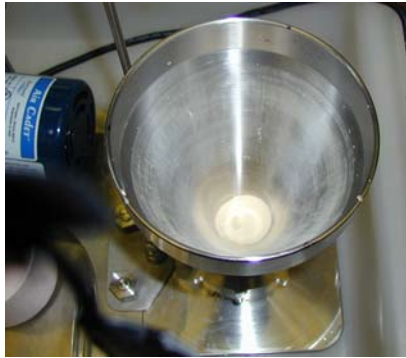
## 2.6 Chloride Removal Performance Test Steps

A general list of the steps used in testing is included below. The sampling steps included in the listing below are not expected to be part of the actual facility process (these actions are highlighted in gray).

<sup>h</sup> During this same period the masses of mixture components other than the chloride may also change as well.

plan was to closely duplicate the processing steps intended for the facility and yet gather the necessary data for demonstration of equipment performance. The testing was performed in accordance with the applicable work instruction.<sup>14</sup>

1. Load a Copper Filter Weldment into the Washer.
2. Place 250 mL of wash solution into a wash bottle for later use.
3. Place remainder of the wash solution (that remaining after filling squirt bottle) in the mixing beaker.
4. Sample the dry surrogate and package for lab analysis of Cl<sup>-</sup>.
5. Weigh the surrogate to be added to washer and then pour into the solution-filled mixing beaker
6. Stir for one-minute
7. Soak for five-minutes
8. Stir again for one-minute
9. Pour the material into the Washer Funnel.
10. Use the 250 mL of reserved wash solution to rinse the mixing beaker.
11. Vacuum filter the first wash by energizing the pump and operating the valves as necessary (see Figure 2-4).



**Figure 2-4 View Looking Down Into the Washer Funnel at the End of a Wash**

12. When the vacuum gauge registers a drop in vacuum level, make note of the rate of decrease. The point at which the decrease in vacuum slows signals the end of the wash cycle and the need to de-energize the pump.
13. Drain the water trap; pull and package a small liquid sample of the first wash discharge.
14. Extract a core sample of the post-first-wash surrogate and package for lab analysis.
15. Pour the next volume of wash water into the washer funnel and vacuum filter by energizing the pump as before.
16. De-energize the pump as before by paying close attention to the vacuum gauge reading.
17. Drain the water trap; pull and package the sample discharge for that wash cycle.
18. Repeat the previous three steps as necessary to complete the pre-determined number of washes for the run.
19. Remove and package the final washed surrogate sample. At this point the material is considered ready for addition to the HB-Line Dissolver (see Figure 2-5).

A schematic of various process flows during the test operation of the Washer follows in Figure 2-6.



Figure 2-5 Final Washed Surrogate

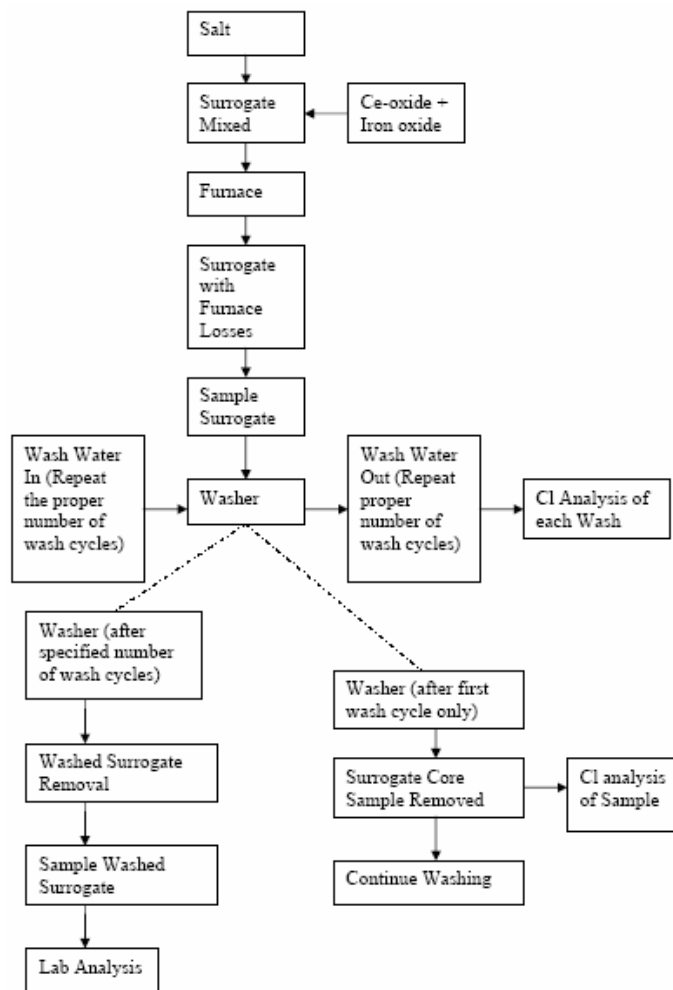


Figure 2-6 Schematic of Process Flowsheet throughout the Chloride Washer Performance Testing



## 2.7 Sampling Plan used to Obtain Performance Data

Sufficient sample analysis was planned to determine the progress of the Cl<sup>-</sup> removal during the washing process. The surrogate initially had well-known Cl<sup>-</sup> content prior to heating to 950 °C in the furnace. After the furnace heating, some uncertainty exists in the exact Cl<sup>-</sup> content due to Cl<sup>-</sup> losses. The Cl<sup>-</sup> lost was not equal to the total surrogate weight loss in the furnace because many other reactions which were not quantified may also have occurred. The final weight was dictated by the summation of all of the furnace reactions and not just the Cl<sup>-</sup> volatilization. The post-furnace surrogate sample analysis discussed in Section 2.5 was intended to re-establish a starting Cl<sup>-</sup> mass for the material.

The core sample analysis provided Cl<sup>-</sup> content status after the first wash. This sample was a core-type sample as it was hoped to get Cl<sup>-</sup> indication with minimal disturbance of the surrogate filter cake. Each surrogate was uniformly cored to a depth of 1.5 inches on six of the eight runs. The core was located along the central axis of the washer in all cases. Deeper coring may have been possible, but concerns over damaging the filter during sampling process and consequently losing the run restricted the depth.

The sampling of the final surrogate provided Cl<sup>-</sup> content in the surrogate after all washing was completed. The entire washed surrogate contents were delivered to the analysis lab for mixing on a mortar and pestle. Analysis results from this sample submission were considered to be the most representative of all of the solid sample analyses in terms of representing the true Cl<sup>-</sup> level in the contents of the entire run. This was due to the thorough mixing treatment that the entire run's contents received. Regardless of uncertainty associated with Cl<sup>-</sup> losses during calcination or solution analysis, the final surrogate analyses are the critical data.

In addition to the three surrogate samples, each wash cycle's discharge solution was also sampled and analyzed for Cl<sup>-</sup>. An advantage of the liquid sample is its inherent homogeneity when compared to a solid mixture; the Cl<sup>-</sup> mass in the liquid wash can also be correlated with surrogate analyses to verify the Cl<sup>-</sup> removal.

## 2.8 Analysis Methodology

The Process Science Analytical Laboratory (PSAL) in SRNL provided all of the Cl<sup>-</sup> analysis support related to the Chloride Washer testing. Each sample delivered was larger in size than the minimum needed to perform a single analysis of the material for Cl<sup>-</sup>. To provide an indication of homogeneity in the sample, three aliquots were analyzed from each sample with all results reported. The analysis procedure used by the PSAL is as follows:

The Cl<sup>-</sup> analysis was performed using a potassium hydroxide (KOH) digestion and then analyzing the digestion on a Dionex DX 500 Ion Chromatograph (IC). The surrogate materials were received as a dry powder (initial sample) and wet slurries (core and final samples). All samples were homogenized by mixing the entire sample using an agate mortar and pestle. A 0.25 g aliquot of sample and 2.0 g of KOH were added to a 50-mL alumina crucible. The crucible was placed on a heating block set at 375 °C. A gyrotory shaker was used to mix the sample while heating. The flux was heated for 15 min and then removed from the heating plate. Once the crucible was cool, 10 mL of de-ionized (DI) water was added to the crucible to dissolve the Cl<sup>-</sup> from the flux. The sample was poured into a 50-mL volumetric flask and diluted to the 50-mL mark with DI water. The sample was filtered using a 0.45-micron filter and then 10X and 100X dilutions with DI water were performed.

A Dionex DX 500 IC with an AS-11HC column was used to analyze the sample. The IC method used an isocratic run using a 20 mM NaOH solution. The detector was set at 300 mA, the pump at 1.5 mL/min and a run time of seven minutes. Chloride standards of 0.1 ppm, 0.2 ppm and 0.5 ppm in a 10% KOH matrix were used to calibrate the instrument. The low concentration calibration standards and the water dilutions (which decreased the interference from potassium) allowed for a minimum detection limit of <200 ppm. The value was reported at the moisture content of the received material.

Liquid samples were run with the standard calibration on the IC. Calibration was performed using a 1-ppm, 5-ppm and 10-ppm standards for the anions of interest. Standards were made in DI water. The samples were diluted by 100X, 1000X and 10000X. The instrument and column were the same as used on the surrogate analysis described above.

## 2.9 Performance of the Copper Filter

A piece of non-chloride removal test data of interest to the project was the filter's particulate-retention efficiency. The filter must be capable of allowing adequate wash solution flow to promote reasonable cycle times, but also have adequate insoluble particulate retention.

Insoluble material loss is important to quantify as the material will be grouted in a paint pail with the spent wash solution during the actual processing. The paint pails will be processed through the RASP to determine the fissile-gram-equivalent mass in each paint pail. High material content could lead to an alarm state in the RASP with potential process delays. The paint pails will then be loaded in a TRU-Waste Drum. Each TRU-Waste Drum has a total fissile mass limit of 150 g. Up to 10 paint pails are approved for potential placement in each TRU-Waste Drum as long as the total fissile mass limit for the drum is not exceeded.

A 2.5-micron polishing filter was used to capture the insoluble mass of surrogate passing through the 20-micron filter<sup>i</sup> during the performance testing. This activity was performed per the applicable work instruction.<sup>15</sup>

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<sup>i</sup> Filter material is UNS C51000, the construction is 200 x 900 WPI, Twill Dutch Weave Style

### 3.0 RESULTS

#### 3.1 Equipment Performance

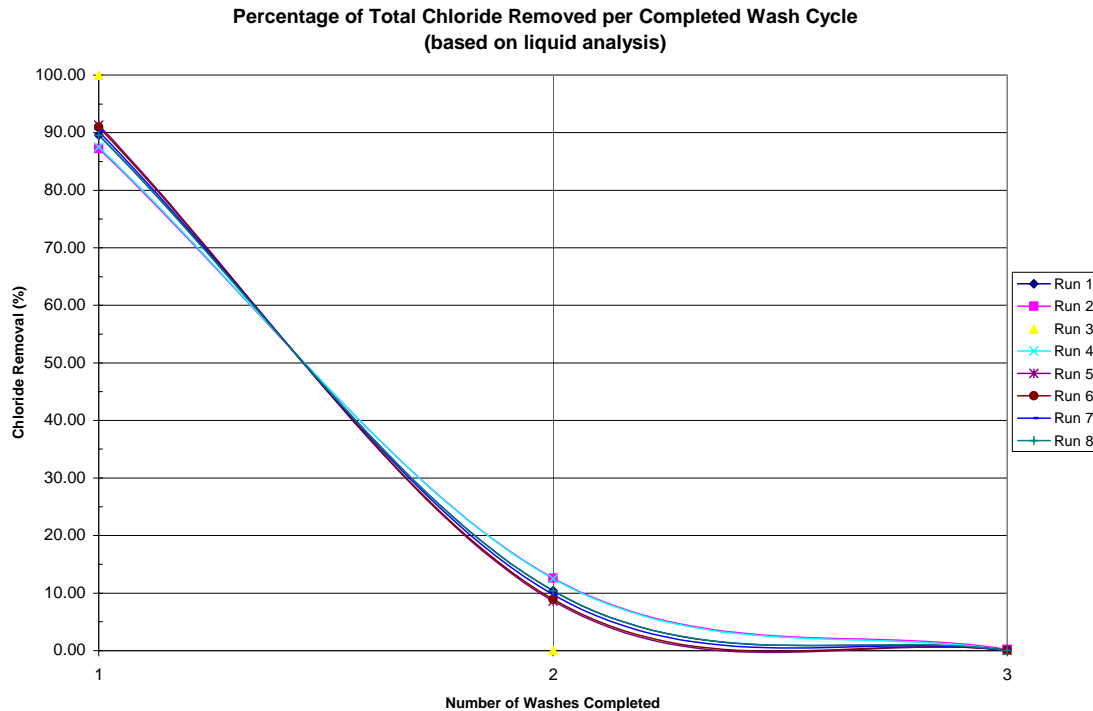
Eight runs of the Washer were completed to determine the Cl<sup>-</sup> removal capabilities of the equipment. Table 3-1 lists test parameters and some abbreviated results associated with all eight runs. The detailed data are contained in Appendix A. Review of the Cl<sup>-</sup> mass present in the Wash discharge showed that approximately 90% of the Cl<sup>-</sup> withdrawn was removed in the first wash as seen in Figure 3-1. The data indicated that essentially the same amount of Cl<sup>-</sup> was removed (within the high- or low-chloride simulant) regardless of the wash volume parameter. The remainder of the Cl<sup>-</sup> was almost entirely removed in the second wash. No Cl<sup>-</sup> removal was detectable beyond the third wash. The data suggest that essentially all of the Cl<sup>-</sup> was liberated with the first wash and then subsequent washes are used to remove Cl<sup>-</sup> solution entrained in the surrogate.

**Table 3-1 Listing of Test Parameters and Results for each Run**

	<b>RUN 1</b>	<b>RUN 2</b>	<b>RUN 3</b>	<b>RUN 4</b>	<b>RUN 5</b>	<b>RUN 6</b>	<b>RUN 7</b>	<b>RUN 8</b>
Surrogate Cl <sup>-</sup> Level	Low	Low	Low	Low	High	High	High	High
Number of Washes	3	3	3	5	3	3	3	5
Wash Solution Volume (L)	1/2	3/4	1	3/4	1/2	3/4	1	3/4
Total Wash Cycle Time (min)	53	87	112	122	36	54	46	102
Cl <sup>-</sup> Analysis of Starting Surrogate (g)	3.99	3.00	3.24	4.03	36.63	36.83	34.42	33.15
Cl <sup>-</sup> Analysis of Final Surrogate (g)	<0.09	<0.08	<0.08	<0.08	<0.07	<0.07	<0.07	<0.07
Wash 1: Cl <sup>-</sup> Removal (g)	3.54	3.79	3.64	3.65	37.00	34.15	33.10	33.41
Wash 2: Cl <sup>-</sup> Removal (g)	0.41	0.55	<sup>j</sup>	0.52	3.49	3.35	3.57	3.87
Wash 3: Cl <sup>-</sup> Removal (g)	-	0.01	-	-	0.02	0.02	0.01	0.02

Two starting surrogate Cl<sup>-</sup> analysis values reported in Table 3-1 appear anomalous (Runs 2 and 3). The data show that all four low-chloride runs have similar liquid wash Cl<sup>-</sup> removals, each summing to approximately 4.0 grams Cl<sup>-</sup> removed per run. Additionally, the final surrogate result for all four is nearly identical at under 0.09 grams. Finally all four were prepared to the same instruction. Parallel behavior for all four samples can be noted for Runs 5-8. As a result, it is strongly suspected that the starting surrogate analysis result in both Run 2 and Run 3 should actually be approximately 4.0 grams and not the gray highlighted values shown in Table 3-1. It is expected that the lab analysis procedure was of good quality as the triplicate results on a particular run showed very little variation (see Appendix B); however, inadequate mixing prior to sample withdrawal may have led to artificially low values for Runs 2 and 3.

<sup>j</sup> This wash was not sampled, therefore no data exists to document the actual Cl<sup>-</sup> removal on this wash



**Figure 3-1 Plot of Chloride Removal Based on Liquid Analysis**

Final washed surrogate runs on each particular run were delivered to the laboratory for analysis. After extensive mixing with a mortar and pestle, the final surrogate was sampled and analyzed in triplicate. The analyses results were compared to provide an indication of the homogeneity of the mixture established with the mortar and pestle. All of the final washed material showed a residual Cl<sup>-</sup> level of less than 0.09 g (<200 ppm). Charged to 15 L of HB-Line dissolver solution, a resulting Cl<sup>-</sup> content of less than 5 +/-0.5 ppm would exist in the dissolver.

### 3.2 Wash Cycle Time

Individual wash cycle times were recorded for each test. The start time coincided with energizing the vacuum pump. As a wash was nearing completion, the vacuum gauge reading that had held steady since about two minutes after the pump had started would start to indicate the loss of vacuum. Initial movement of the gauge at the end of the wash appears to coincide with having removed the bulk of the wash liquid through the filter weldment. As the process continued, more of the residual water was pulled out of the material and allowed for increased air flow. Usually 1-3 minutes elapsed from the initial vacuum break at around 26 in. Hg until the rate of decrease slowed to near zero at a value ranging from 15-20 in. Hg vacuum. When the rate of vacuum loss had dropped to near zero, the wash was considered complete and the time was recorded (see Table 3-2).

Some data scatter is present as the wash completion timing was somewhat subjective. The information is included to provide indication of wash durations noted in this testing. In general, the data suggests the flow rate through the filter was reduced on each subsequent wash, probably due to formation of an effective filter cake and potentially some filter blinding. One defined trend was that high Cl<sup>-</sup> runs had shorter wash durations, thought to be most likely due to smaller masses of undissolved solids present on the filter.

**Table 3-2 Approximate Wash Time Required for Each Wash Cycle**

	Run 1 (½-liter Washes)	Run 2 (¾-liter Washes)	Run 3 (1-liter Washes)	Run 4 (¾-liter Washes)	Run 5 (½-liter Washes)	Run 6 (¾-liter washes)	Run 7 (1-liter Washes)	Run 8 (¾-liter Washes)
<b>Wash No. 1 (min)</b>	11.0	18.5	28.25	16.25	8.75	13.25	4.50 <sup>k</sup>	11.25
<b>Wash No. 2 (min)</b>	17.0	33.0	39.5	20.0	12.25	18.5	19.0	20.75
<b>Wash No. 3 (min)</b>	24.5	35.25	44.0	21.5	15.0	22.0	22.25	23.0

### 3.3 Solids Content in the Wash Discharge

The wash discharged from the process was poured through a polishing filter to quantify insoluble solids that had passed through the 20-micron copper filter during each of the first six runs. Six of the eight runs were checked as outlined in Work Instruction RSE-WI-2007-0002, Section 2. The filter paper used was Whatman® Grade 5 (2.5-micron particle retention). The paper was used to capture the solids in the wash, dried and weighed per the instruction<sup>1</sup>. The results are shown in Table 3-3. The values listed in the table are mass totals for each run shown. All runs were washed with 3 wash cycles except for Run 4 which was washed with a total of 5 cycles. No apparent reason for the slightly elevated value in Run 6 was evident.

**Table 3-3 Insoluble Material Mass Captured in Polishing Filter**

	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
Solid Mass Captured in Polishing Filter (g)	0.5	0.5	0.6	0.5	0.7	1.1
Percentage of Insoluble Starting Mass (%)	0.16	0.16	0.19	0.16	0.26	0.41

<sup>k</sup> It was noticed immediately during the testing that this run showed increased flow rate. At the end of the run, the filter was not torn and there was no evidence of excessive particulate loss. No conclusive evidence was seen as to the reason for the higher flow rate.

<sup>1</sup> Each individual filter was labeled and initially weighed. Two extra filters were included in this process with the sole function of acting as controls – no wash solution was poured through them. Each run was poured through a separate filter – the filters were allowed to dry and were periodically weighed until an equilibrium weight was established. The weight difference was considered the solids mass that passed the filter.

### 3.4 Chloride Mass Balance

The Cl<sup>-</sup> mass balance, as used in this report, is defined as the ratio of the Cl<sup>-</sup> mass accounted for in the wash solution and washed surrogate to the Cl<sup>-</sup> mass in the surrogate initially placed in the washer. The Cl<sup>-</sup> mass balance data reported in Table 3-4 yield good Cl<sup>-</sup> mass balances (except for Run 2) when considering the error associated with the sample analysis procedure ( $\pm 10\%$ ).

**Table 3-4 Chloride Mass Balance**

	Run 1	Run 2	Run 4	Run 5	Run 6	Run 7	Run 8
Surrogate Type	Low-Chloride			High-Chloride			
Pre-Furnace Surrogate Cl <sup>-</sup> via Recipe (g)	4.91	4.91	4.91	39.25	39.25	39.25	39.25
Post-Furnace Surrogate Cl <sup>-</sup> via Analysis (g)	3.99	3.00	4.03	36.63	36.83	34.42	33.15
Final Washed Surrogate Cl <sup>-</sup> via Analysis (g)	<0.09	<0.08	<0.08	<0.07	<0.07	<0.07	<0.07
Removed in Wash Solution Cl <sup>-</sup> via Analysis (g)	3.96	4.34	4.17	40.52	37.53	36.69	37.30
Mass Balance	1.02	1.47	1.05	1.11	1.02	1.07	1.13

Some of the potential sources of error are as follows. The laboratory analysis on the single sample withdrawn from the plastic bottle was completed in triplicate – that is, three separate sub-samples and analyses were performed on each individual sample submitted for analysis<sup>m</sup> (see Appendix B). The maximum total variation within a triplicate analysis on a single run was under 4%. Most triplicate results on a particular sample submission were within 2% total variance. The repeatability implies that the sample was very well mixed just prior to analysis.

All of the runs within a particular group (high- or low-chloride) were spiked with the same amount of Cl<sup>-</sup> (within 0.1 g of each other). Each run was prepared separately but still according to the same instruction, being mixed with a spatula in a crucible (in powder form), and held at 950 °C in the furnace. At the completion of the furnace run – the material was allowed to cool and was subsequently transferred to a plastic bottle and shaken to further mix the contents. After thorough shaking, a sample was withdrawn with a spatula. The fact that inter-run analysis results show as much as 28% variance (much larger than the 4% for the intra-run analysis above) indicates that perhaps the surrogate within the plastic bottle (host) was not homogeneous at the time of sampling, and represents the most likely source of mass imbalance. Ideally, the variance on the inter- and intra-run results within the same simulant should be very similar.

<sup>m</sup> This was the practice with every sample submitted for this testing.

## 4.0 CONCLUSIONS

The testing results reported herein lead to the following three conclusions:

- 1) Performance testing shows that three ½-liter washes were adequate to reduce Cl<sup>-</sup> levels in up to 345 g of 3013 surrogate material to below 200 ppm in the final surrogate. Performance for ¾-liter and 1-liter washes showed no advantages over the ½-liter washes. The demonstration evaluated Cl<sup>-</sup> levels ranging from approximately 8,000-70,000 ppm<sup>n</sup> Cl<sup>-</sup> in PuO<sub>2</sub> by using CeO<sub>2</sub> as a surrogate for PuO<sub>2</sub>. The liquid analysis showed that close to 90% of the removal occurred in the first wash when any one set of the test parameters described in this report was used. Charging the HB-Line dissolver<sup>o</sup> with the residual Cl<sup>-</sup> mass comparable with that measured in the testing (after 3 washes) should result in less than 5 ppm Cl<sup>-</sup> present in the solution.
- 2) The 20-micron copper filter provided acceptable solids retention with a maximum insoluble fraction loss per run of 1.1 g, or 0.4% by weight of initial insoluble solids.
- 3) The wash cycle times ranged from 36-minutes total for three-washes at ½ liter per wash volume to 122-minutes total for five-washes at ¾ liter per wash. It was noted that the higher Cl<sup>-</sup> material generally had shorter time requirements to draw a given volume of wash through the filter because the total undissolved mass on the filter was less.

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<sup>n</sup> Based on average post-furnace starting chloride content – reflects furnace losses in the surrogate; equivalent to the chloride mass in 500 g of 3013 material at ~70,000 ppm Cl<sup>-</sup> content (high-chloride) and ~8,000 ppm Cl<sup>-</sup> (low-chloride).

<sup>o</sup> Assumes 15 L of 1.2 specific gravity solution is in the dissolver.

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## 5.0 RECOMMENDATIONS

The current report provides sufficient technical basis for successful washing of 500 g of 8,000-70,000 ppm Cl<sup>-</sup> KIS 3013 materials prior to dissolution in HB-Line. The current testing has consistently shown that a minimum of three ½-liter washes will produce a final washed surrogate with less than 200 ppm Cl<sup>-</sup>.

Future testing is recommended related to any of the following: (1) testing at a smaller wash volume to minimize wash water rejection, (2) testing accident scenarios such as a filter rupture, or (3) additional process validation.

Reduced wash volume testing (3 washes x 1/3 L each) has potential value in that possibly 1 kg of 3013 material could be washed with the rejected wash solution captured within one paint pail. Reuse of the third-wash volume into the next run could potentially allow for containing 3 runs of 3013 material into a paint pail (1-1/2 kg nominal). Reuse would require careful facility review in terms of procedural control to ensure the prevention of reuse of the incorrect wash water (higher Cl<sup>-</sup> values than expected in the process could result). It is worth noting that 3 washes at 1/3 L individual volume, coupled with reuse of the third wash, will reduce the number of paint pails going to TRU waste by 67% when compared to ½-liter washes with no reuse.

Two accident scenarios that might be of interest and are recommended for evaluation are as follows. Operation of the Washer without the drain plate installed. The drain plate provides structural backing for the filter and therefore it is not currently known if the filter material would rupture if the vacuum pump was energized without the plate in position. The second scenario would be to intentionally tear the filter and operate the Washer in the normal manner. This would simulate a torn filter installed in the equipment to make note of the equipment response and the extent of recovery operations. The information would provide data on potential process delays that might be expected if a similar event were to occur in the facility during the campaign. One potential outcome of such an activity may be a procedural requirement to filter the solution drained out of the water trap prior to its placement into the paint pail.

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## 6.0 REFERENCES

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## 7.0 ACKNOWLEDGEMENTS

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**APPENDIX A. CHLORIDE WASHER TEST DATA**

	<b>RUN 1</b>	<b>RUN 2</b>	<b>RUN 3</b>	<b>RUN 4</b>	<b>RUN 5</b>	<b>RUN 6</b>	<b>RUN 7</b>	<b>RUN 8</b>
<b>Solid Data &amp; Calculations</b>								
Surrogate added to the Furnace (g)	321.10	321.10	321.10	321.10	345.00	345.00	345.00	345.00
Surrogate Removed from Furnace (g)	313.50	310.40	312.90	312.50	338.20	337.80	331.20	336.20
Weight Loss in Furnace and Transfer Loss out of the Crucible (g)	7.60	10.70	8.20	8.60	6.80	7.20	13.80	8.80
Surrogate Added to Washer (g)	311.50	307.90	310.60	309.30	335.00	334.30	327.80	336.20
Cl <sup>-</sup> Analysis Results of Dry Starting Surrogate (ppm)	12,800	9,757	10,433	13,033	109,333	110,167	105,000	98,600*
Initial Cl <sup>-</sup> Mass (g)	3.99	3.00	3.24	4.03	36.63	36.83	34.42	33.15
Core sample Removed after Wash 1 (g)	1.20	1.80	1.50	-	1.20	2.00	1.70	-
Core Sample Cl <sup>-</sup> Analysis Reported (ppm)	650	1,347	1,019	-	7,423	6,213	7,303	-
Mass of Cl <sup>-</sup> in 1st Core Sample (g)	0.00	0.00	0.00	-	0.01	0.01	0.01	-
Mass of Wet Solid after Wash #1 (g)	448.90	430.90	439.10	442.00	380.40	373.00	386.60	385.30
Mass of Cl <sup>-</sup> Left in the Wet Solid (g)	0.29	0.58	0.45	-	2.82	2.32	2.82	-
Mass of Cl <sup>-</sup> Removed on Wash 1 (g)	3.70	2.42	2.79	-	33.80	34.51	31.60	-
Mass of Wet Finish Surrogate Removed from Washer (g)	429.40	411.00	420.50	414.80	372.00	373.10	366.80	373.60
Cl <sup>-</sup> Analysis Results of the Wet Finish Surrogate (ppm)	<200	<200	<200	<200	<200	<200	<200	<200
Final mass of Cl <sup>-</sup> (g)	<0.09	<0.08	<0.08	<0.08	<0.07	<0.07	<0.07	<0.07
Mass of Cl <sup>-</sup> Washed Out Based on Solid Analysis (g)	>3.90	>2.92	>3.15	>3.95	>36.54	>36.74	>34.33	>33.07
<b>Liquid Data &amp; Calculations</b>								
<b>Wash #1 Summary</b>								
Wash Mass Added (g)	500	750	1,000	750	500	751.30	994.30	748.00
Wash Mass Removed (g)	361.40	625.20	870.00	617.30	453.40	710.60	933.80	698.90
Cl <sup>-</sup> Level (mg/L)	10,200	6,240	4,313	6,090	91,400	51,900	37,933	51,633
Lab Reported Density (g/mL)	1.04	1.03	1.03	1.03	1.12	1.08	1.07	1.08
Cl <sup>-</sup> Mass Removed (g)	3.54	3.79	3.64	3.65	37.00	34.15	33.10	33.41
<b>Wash #2 Summary</b>								
Wash Mass Added (g)	500	750	1,000	750	500	750	937.50	746.80
Wash Mass Removed (g)	502.80	751.40	1,005.4	754.80	498.60	738.40	944.70	745.80
Cl <sup>-</sup> Level (mg/L)	835.30	741.70	-	702.70	7,213.3	4,676.7	3,856.7	5,346.7
Lab Reported Density (g/mL)	1.02	1.02	1.00	1.02	1.03	1.03	1.02	1.03
Cl <sup>-</sup> Mass Removed (g)	0.41	0.55	-	0.52	3.49	3.35	3.57	3.87

\* Triplicate analysis was not performed – enough material was available to perform one analysis only.

	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6	RUN 7	RUN 8
<b>Wash #3 Summary</b>								
Wash Mass Added (g)	500	750	1000	750	500	748.9	999.7	744.5
Wash Mass Removed (g)	507.4	751.3	999.5	751.0	493.1	748.2	1,001.3	745.1
Cl <sup>-</sup> Level (mg/L)	-	11.1	-	-	48.5	32.8	10.0	23.6
Lab Reported Density (g/mL)	1.02	1.02	1.02	1.02	1.02	1.02	1.02	1.03
Cl <sup>-</sup> Mass Removed (g)	-	0.01	-	-	0.02	0.02	0.01	0.02
<b>Wash #4 Summary</b>								
Wash Mass Added (g)	-	-	-	750	-	-	-	752.8
Wash Mass Removed (g)	-	-	-	749.8	-	-	-	747.8
Cl <sup>-</sup> Level (mg/L)	-	-	-	-	-	-	-	-
Lab Reported Density (g/mL)	-	-	-	1.02	-	-	-	1.02
Cl <sup>-</sup> Mass Removed (g)	-	-	-	-	-	-	-	-
<b>Wash #5 Summary</b>								
Wash Mass Added (g)	-	-	-	750	-	-	-	742.6
Wash Mass Removed (g)	-	-	-	749.0	-	-	-	737.9
Cl <sup>-</sup> Level (mg/L)	-	-	-	-	-	-	-	-
Lab Reported Density (g/mL)	-	-	-	1.02	-	-	-	1.02
Cl <sup>-</sup> Mass Removed (g)	-	-	-	-	-	-	-	-
<b>All Washes Combined Total Chloride Removal</b>								
Total Wash Cl <sup>-</sup> Removal (g)	3.96	4.34	-	4.17	40.52	37.53	36.69	37.30
<b>Overall Material Balance</b>								
Total mass in (g)	1,811.5	2,557.9	3,310.6	4,059.3	1,835.0	2,584.5	3,259.3	4,070.9
Total mass out (g)	1,802.2	2,540.7	3,296.9	4,036.7	1,818.3	2,572.3	3,248.3	4,049.1
Mass un-accounted (g)	9.30	17.20	13.70	22.60	16.70	12.20	11.00	21.80
% Un-accounted	0.51%	0.67%	0.41%	0.56%	0.91%	0.47%	0.34%	0.54%
<b>Analysis</b>								
<b>Chloride removed for all washes</b>								
Difference in TOTAL mass of Cl <sup>-</sup> washed out (Solid analysis - water analysis) (g)	-0.06	-1.42	-	-0.22	-3.97	-0.78	-2.35	-4.23
Percent of difference based on solid analysis	-1.43%	-48.73%	-	-5.61%	-10.87%	-2.13%	-6.85%	-12.78%
<b>Chloride removed for wash #1</b>								
Cl <sup>-</sup> removed on Wash 1 based on solid analysis (g)	3.70	2.42	2.79	-	33.80	34.51	31.60	-
Cl <sup>-</sup> removed on Wash 1 based on water analysis (g)	3.54	3.79	3.64	-	37.00	34.15	33.10	-
Percent Difference for Wash 1	4.08%	-56.27%	-30.44%	-	-9.46%	1.05%	-4.78%	-
<b>Washing efficiency</b>								
% of Cl <sup>-</sup> removed - Wash 1	89.59	87.23	-	87.53	91.32	91.00	90.24	89.58
% of Cl <sup>-</sup> removed - Wash 2	10.41	12.58	-	12.47	8.62	8.93	9.74	10.38
% of Cl <sup>-</sup> removed - Wash 3	0.00	0.19	-	0.00	0.06	0.06	0.03	0.05
% of Cl <sup>-</sup> removed - Wash 4	0.00	0.00	-	0.00	0.00	0.00	0.00	0.00
% of Cl <sup>-</sup> removed - Wash 5	0.00	0.00	-	0.00	0.00	0.00	0.00	0.00

**APPENDIX B: SURROGATE TRIPLICATE ANALYSIS RESULTS**

	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6	RUN 7	RUN 8
<i>Initial Surrogate Cf (ppm)</i>								
sample 1	12800	9720	10600	13200	110000	110700	107000	-
sample 2	12900	9730	10400	13000	110000	110800	103000	98600 <sup>P</sup>
sample 3	12700	9820	10300	12900	108000	109000	105000	-
<i>Surrogate Core Cf after Wash 1 (ppm)</i>								
sample 1	645	1350	997	-	7310	6200	7160	-
sample 2	693	1340	1000	-	7360	6180	6810	-
sample 3	612	1350	1060	-	7600	6260	7940	-
<i>Final Washed Surrogate Cf (ppm)</i>								
sample 1	<200	<200	<200	<200	<200	<200	<200	<200
sample 2	<200	<200	<200	<200	<200	<200	<200	<200
sample 3	<200	<200	<200	<200	<200	<200	<200	<200
<i>Wash 1 Liquid Cf level (mg/L)</i>								
sample 1	10200	6320	4340	6100	91200	51500	37400	52000
sample 2	10200	6200	4270	6100	91600	52200	38700	51200
sample 3	10200	6200	4330	6070	91400	52000	37700	51700
<i>Wash 2 Liquid Cf level (mg/L)</i>								
sample 1	833	745	-	701	7310	4690	3840	5360
sample 2	840	737	-	704	7150	4660	3880	5330
sample 3	833	743	-	703	7180	4680	3850	5350
<i>Wash 3 Liquid Cf level (mg/L)</i>								
sample 1	2510 <sup>q</sup>	11.3	<10.0	<10.0	48.4	32.7	12500 <sup>r</sup>	23.6
sample 2	2500	11.1	<10.0	<10.0	48.5	32.8	12600	23.5
sample 3	2500	11.0	<10.0	<10.0	48.7	32.9	12800	23.7

<sup>P</sup> Only enough sample was available for one analysis.

<sup>q</sup> Sample was an average of all three washes

<sup>r</sup> Sample was an average of all three washes

**Distribution:**

S. L. Marra, 773-A  
A. P. Fellingner, 723-A  
A. J. Tisler, 773-A  
J. B. Fiscus, 730-A  
R. A. Pierce, 773-A  
A. D. Marzolf, 723-A  
W.Y. Cheng, 723-A  
B. V. Nguyen, 704-2H  
D. P. Lemonds, 704-2H  
D. R. Best, 786-1A  
C. C. Herman, 999-W  
S. D. Fink, 773-A  
C. H. Brown, 704-2H  
G. J. Zachmann III, 704-2H  
D. W. Murdoch, 704-2H  
S. L. Tibrea, 730-A  
J. R. Pelfrey, 773-42A  
J. W. Wong, 773-42A  
F. M. Heckendorn, 773-A  
M. W. Stokes, 723-A