Contract No:

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X-RAY DIFFRACTION AND SCANNING ELECTRON MICROSCOPE CHARACTERIZATIONS OF THE SALT WASTE PROCESSING FACILITY TANK 101 AND TANK 104 SOLID SAMPLES

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EXECUTIVE SUMMARY

The "as-received" Salt Waste Processing Facility (SWPF) Tank 101 and two Tank 104 samples (pre-wash and washed solids) have been characterized using x-ray diffraction (XRD) and scanning electron microscope/energy dispersive x-ray (SEM/EDX) techniques to address the concerns for the existence of solids, which may interfere with the operational performance of the SWPF crossflow filtration process.

The key results from the Tank 101 and Tank 104 XRD and SEM characterizations include the following.

- XRD data for the Tank 101 and Tank 104 pre-wash sample show the presence of soluble crystalline sodium compounds, mainly sodium nitrate $Na(NO_3)$, which are likely formed from the evaporation of salt solution.
- Insoluble titanium material [sodium titanium oxide ($Na_2Ti_2O_4(OH)_2$], possibly coming from added monosodium titanate (MST), and sodium aluminum hydroxide ($NaAl(OH)_4$), were also observed in this Tank 104 pre-wash sample.
- Only a titanium mineral phase (sodium titanium oxide, $Na_2Ti_2O_4(OH)_2$) is observed in the Tank 104 washed sample; the other minerals had been washed/leached out.
- The Tank 104 washing process mainly took out the sodium salts, elemental mercury, copper, nickel, uranium, silicon, and iron based on comparison of the pre-washed and washed Tank 104 solids; possibly because these elements are not part of the crystalline minerals identified in both the washed Tank 104 sample solids and the pre-wash Tank 104 solids.
- The particle sizes of these Tank 101, Tank 104 pre-wash and Tank 104 washed solids are in the range of 1-4 microns, while Tank 101 re-sample and Tank 104 washed re-sample particle sizes ranged from 4 to 11 microns, and 0.5 to 2.5 microns, respectively.
- The XRD spectra for the Tank 104 washed re-sample and the original Tank 104 washed samples are essentially the same; both samples show the same predominant sodium-titanium mineral composition.
- The re-sampling and characterization of the Tank 101 solids and the Tank 104 washed sample solids confirm the initial mineralogy results for these two tank solids and did not produce any unexpected results.
- A total of seven XRD minerals were identified in the Tank 101 re-sample and five minerals were identified in the August 09, 2021 Tank 101 sample. Both samples had three minerals in common [trona (Na3H(CO3)*H2O, nitratine (NaNO3), and bayerite/ gibbsite (*a*-Al(OH)3*Al(OH)3].
- The solid sample major elemental components were like those of the initial Tank 101 solids and Tank 104 washed sample solids with some differences in the semiquantitative weight percent elemental compositions and particle sizes.
- Minerals like the zeolites (sodalite and cancrinite) and oxalates, which are known to "blind" filtration systems were not identified in any of the SWPF tank solids.
- The SEM/EDX semiquantitative data for particle size and weight percent elemental compositions for these SWPF solid samples, show significant uncertainties and variations in the results and so may not be reliable enough to account for elemental compositions and particle size changes in the SWPF tank samples.

The chemical make-up of the solids in the SWPF feed tank process chemistry is in line with the OLI model predictions, which indicate that relatively small amounts of soluble and insoluble solids are formed. Above all, there was nothing unexpected, in terms of mineral or solids content, found in the Tank 101, Tank 104 pre-wash, and Tank 104 washed solids samples.

Based on these characterization results for the three SWPF tank samples, the SWPF filtration process performance issues cannot wholly be attributed to the presence of these identified solids. There may be other technical reasons responsible for the poor performance of the SWPF filtration process. Apart from other SWPF operational parameters, it may be necessary to investigate the behavior of the accumulation of salt fines from the salt batches as well as the accumulation of small MST fines, which are normally present in the added MST bulk material.

To obtain more useful analytical results for the elemental compositions and particle size distribution for these SWPF tank solids, it is also recommended that tank solids be acid digested and directly characterized for elemental compositions by inductively coupled plasma atomic emission spectroscopy (ICP-AES) instead of relying on SEM/EDX semiquantitative results. A suspension of SWPF tank solids in a typical SWPF tank simulant solution and characterization for particle size distributions is also recommended.

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
MST	Monosodium titanate
%RSD	Percent relative standard deviation
SaM	Sensing and Metrology
SEM/EDX	scanning electron microscope/energy dispersive x-ray
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SWPF	Salt Waste Processing Facility
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance Plan
XRD	x-ray diffraction

1.0 Introduction

A section of the SRS Salt Waste Processing facility (SWPF) is designed to process batches of radioactive salt solutions with monosodium titanate (MST) acting as the actinide and strontium removal adsorbent. This process is followed by crossflow filtration to concentrate and remove the MST solids and the resulting filtrate is processed through the contactors to remove cesium prior to eventual disposal in the SRS Saltstone grout. A sketch diagram for that part of the SWPF process involved mainly with caustic adjustment, addition of MST and MST removal by crossflow filtration is shown in Figure 1. In this part of the processing of SRS radioactive salt solution, the Tank 101 is the alpha strike tank, where feed is added from Tank 49. The Tank 102 is the filter feed tank, which receives the material from Tank 101, and the Tank 104 is the Sludge Solids Receipt Tank, where solids are washed prior to sending it to the Defense Waste Processing Facility (DWPF). The wash water is stored in Tank 105, which was not sampled since solids should not be present in this tank.

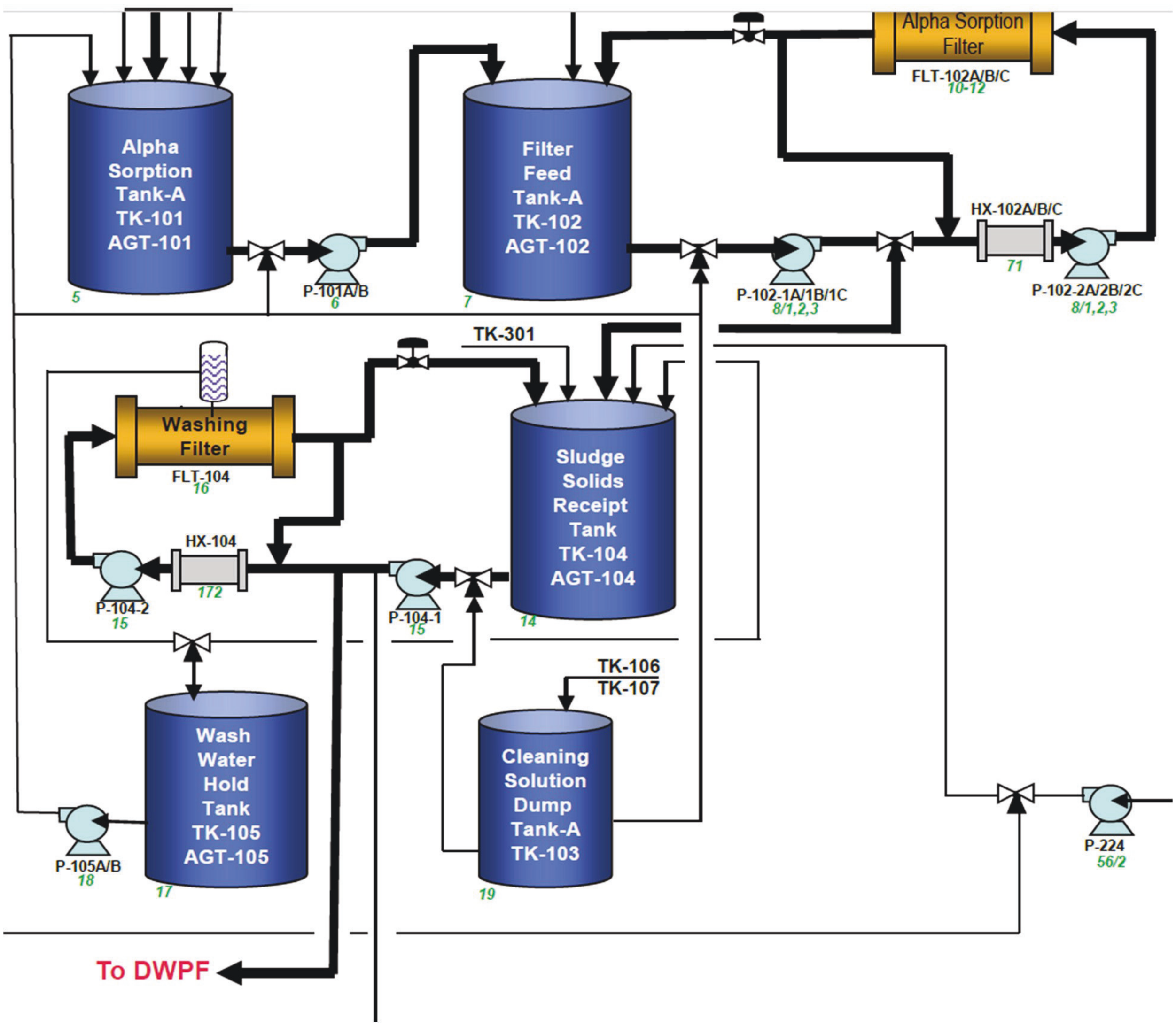


Figure 1. SWPF Caustic Adjustment, MST Alpha Strike, and Crossflow Filtration Set Up Diagram.

It is suspected that the degradation in the filtration performance in the SWPF crossflow filtration process to remove solid particles from SRS salt solution liquid waste may be due to the presence of fine particulates plugging the crossflow filter. As a result of this filtration throughput concerns, SWPF sent tank samples from Tanks 101 and 104 to be characterized by Savannah River National Laboratory (SRNL).

A total of three SWPF Tank sample solids or pastes were sent to SRNL for x-ray diffraction (XRD) and scanning electron microscope/energy dispersive x-ray (SEM/EDX) characterizations. The original sampling plan for characterizing the SWPF tank solids called for the collection of solids from the SWPF Tank 102 and the delivery of this sample to SRNL for XRD and SEM/EDX analysis. However, there were problems with SWPF Tank 102 sampling pump and so sampling from Tank 102 was not possible. Monosodium titanate (MST) is added to the Salt Batch feed in Tank 101 and then a large volume of the salt solution feed was filtered down and transferred to Tank 104 where the sample was collected. In this case, the content of the Tank 102 is considered equivalent to Tank 104. The Tank-104 pre-and post-washed samples were prepared by transferring about 10 mg of the wet and pasty solids from the original sample vial into a new vial which was then sent to SRNL for solids characterizations. The post-wash Tank 104 sample along with the Tank 101 samples were also sent up to SRNL for XRD and SEM/EDX characterizations. Please note that Tank 101 sample was pulled prior to sodium concentration adjustment [6.4 to 5.6 M Na] and prior to MST addition.
The SWPF Tank 104 pre-wash solid material was the first of the tank solid samples delivered to SRNL on July 26, 2021 for characterization while the two other samples [Tank 104 post-wash (washed tank 104 solids) and Tank 101 sample] were sent to SRNL on August 09, 2021.

The analytical sample sizes for Tank 101 and Tank 104 washed samples sent to SRNL on August 09, 2021 were too small for SEM and XRD characterizations. SEM/EDX and XRD data from the small sample size were questionable because of undesired large shift in spectral baseline and low signal intensity for some of the minerals. So, SWPF resampled these two tanks and delivered a relatively larger amount of each sample for these two tank samples [TK-104 (post-wash) solids; sample ID S-210920-00106, and TK-101 solids; sample ID S-210912-00001] on September 24, 2021 for repeat SEM/EDX and XRD characterizations. The second characterization information on these two tank samples is used to confirm the results from the initial analysis with smaller amounts of the solid samples. This time the samples weights were 0.14 g for the Tank 101 sample and 0.45 g for the Tank 104 washed sample.

Objectives

The SWPF customer requested the characterization of the "as-received" Tanks 104 (pre-wash and washed samples) and Tank 101 solids. The tasks requested to be performed by SRNL are summarized in SLA-SRNS-00177 Item x (shown in attachment A).

2.0 Experimental Sample description and Setups

Figure 2 is a picture of the "as-received" SWPF Tank 104 pre-wash solid in a glass vial (July 26, 2021 sample). The yellow-pasty material is at the bottom of one corner of the glass container as shown in Figure 2, insert A. Grains of the Tank 104 washed sample is shown in Figure 2, insert B and traces of the Tank 101 sample inside a centrifuge tube is shown in Figure 2, insert C. Pictures of the September 24, 2021 re-samples for Tanks 101 and 104 post-wash samples are shown in Figure 3. As presented in Figure 3 (inserts A and B), the weights of the samples were more than a factor of 10 greater than the corresponding samples sent to SRNL in July 2021, as shown in the corresponding images for these same samples in Figure 2 inserts B and C. However, these re-sampled materials were wet and required about 72 hours of air-drying in the glove box before use.

After the receipt of the SWPF tank samples in the SRNL Shielded Cell facility, the "as-received" SWPF tank samples were repackaged before sending them directly to the Sensing and Metrology (SaM) group for SEM/EDX and XRD preparations and characterizations.

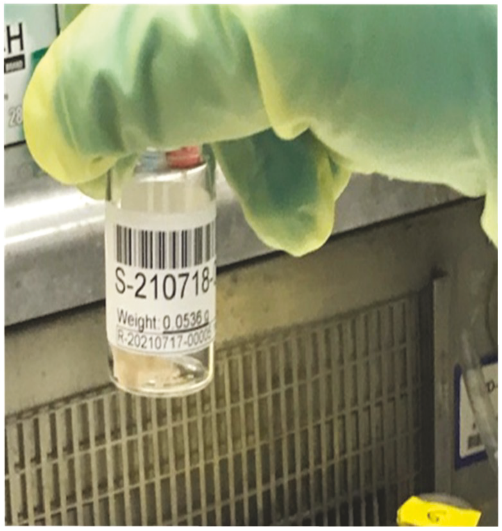
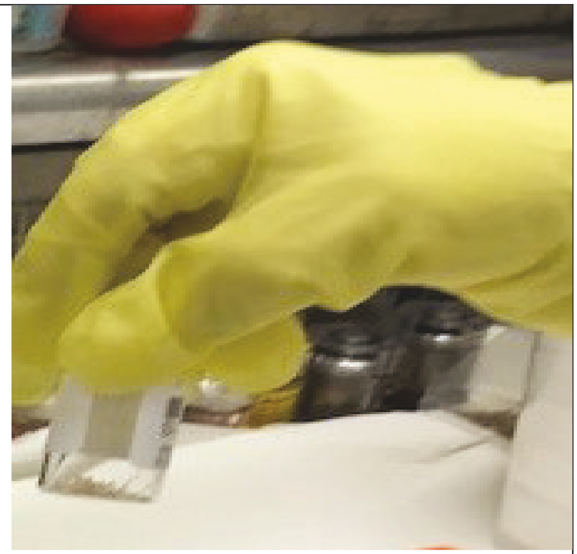
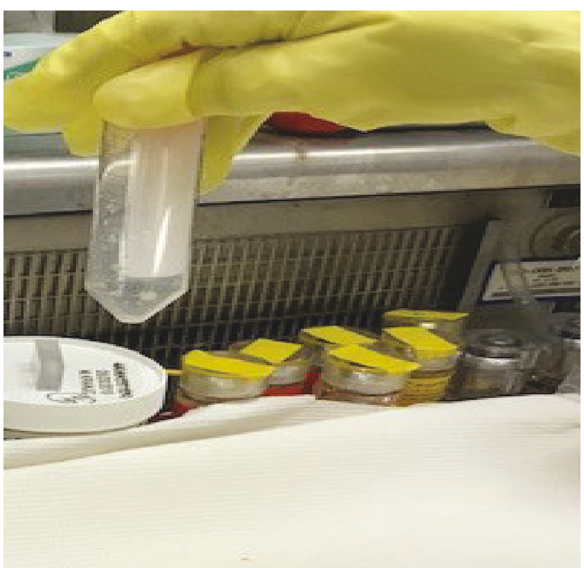
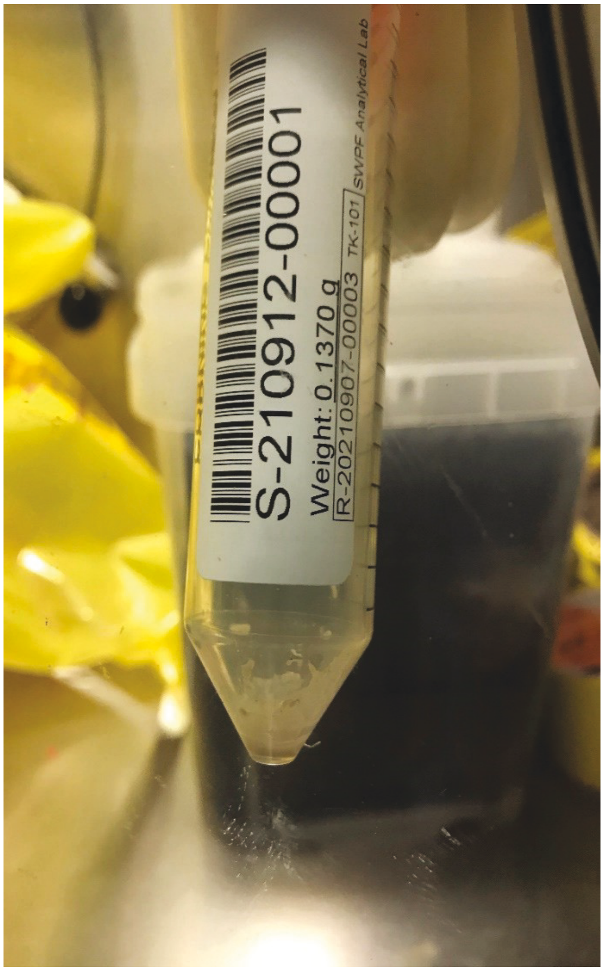
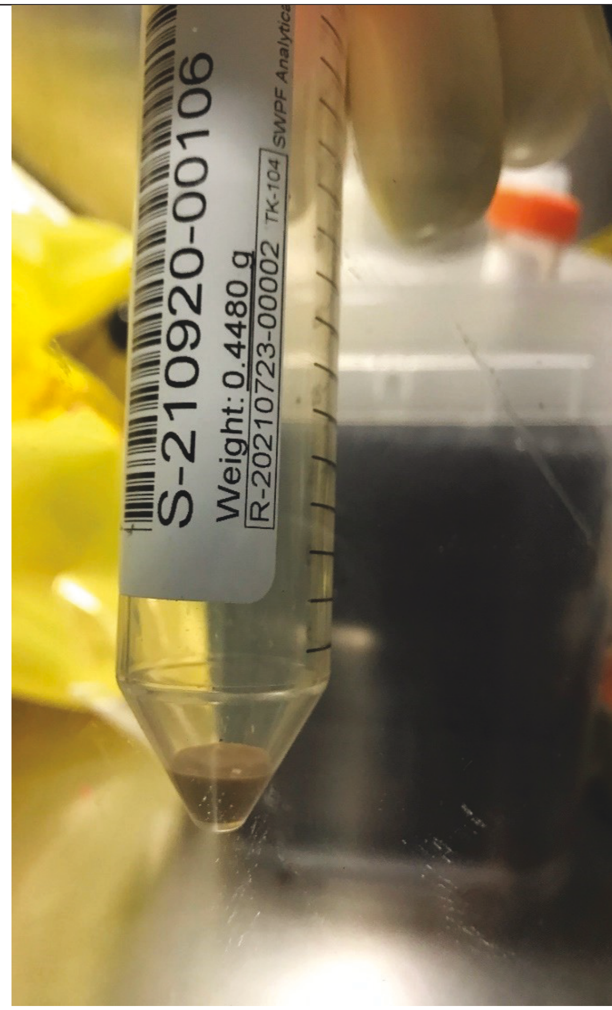
	
Insert A: SWPF Tank 104 pre-wash solids in a glass vial (yellow paste at the bottom of the glass tube.	Insert B: Grains of Tank 104 washed solids in a glass vial (August 09, 2021 sample).
	Intentionally left blank
Insert C: Traces of SWPF Tank 101 solids at the bottom of a centrifuge tube (August 09, 2021 sample).	

Figure 2. Tank 104 samples (pre-wash and washed) and Tank 101 samples solids





Insert A: Tank 101 solids: Sample S-210912-00001 (September 24, 2021 re-sample)	**Insert B:** Tank 104 washed solids (post-wash) Sample S-210920-00106 (September 24, 2021re-sample)

Figure 3. Re-samples for Tank 101 solids and Tank 104 <u>washed</u> solids.

3.0 Results and Discussion

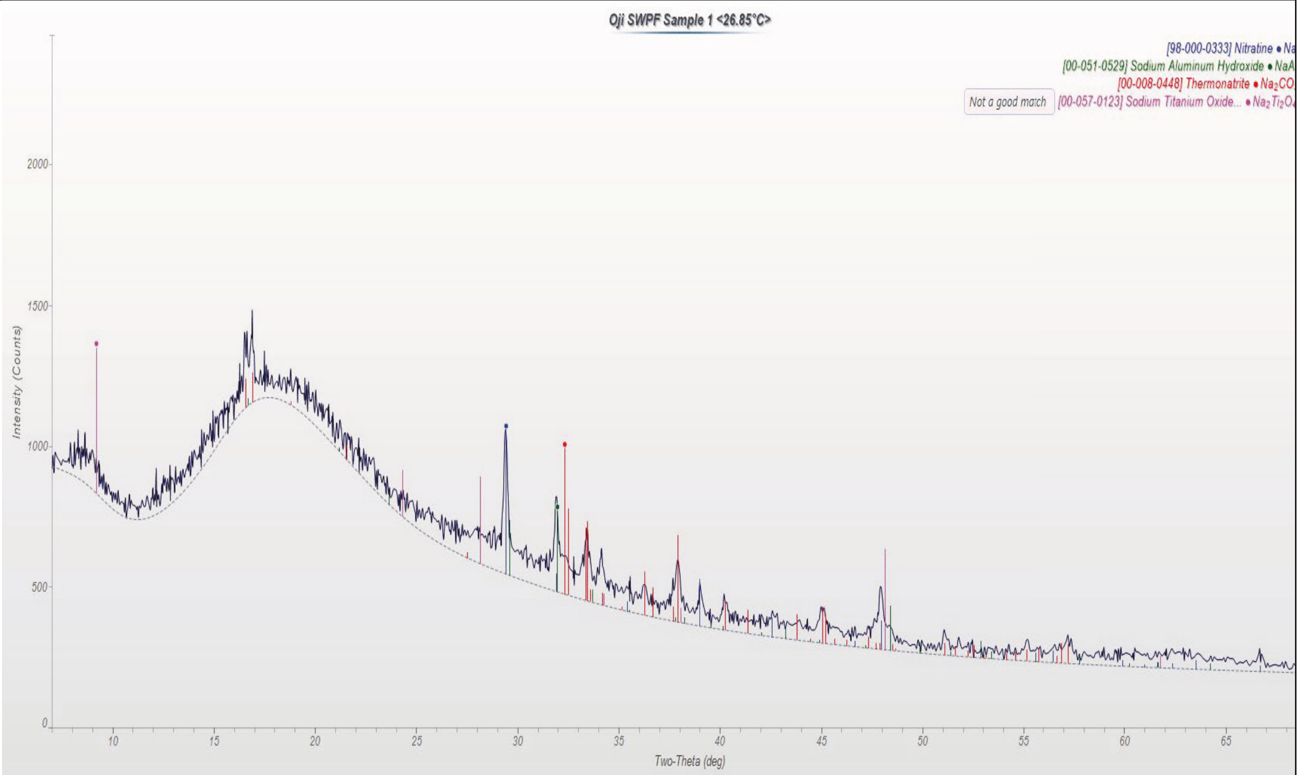
3.1 Tank 104 Pre-wash

The XRD spectra and SEM/EDX images for the SWPF Tank 104 pre-wash solids are presented in Figures 4, and 5a -5e. The XRD spectra for the SWPF Tank 104 pre-wash solid sample, as shown in Figure 4, insert A, indicate the presence of a few crystalline materials mainly sodium nitrate or nitratine $Na(NO_3)$, sodium aluminum hydroxide ($NaAl(OH)_4$), sodium carbonate or thermonitrite ($Na_2CO_3.H_2O$). Monosodium titanate (MST) is an amorphous, non-crystalline material and thus has a poor XRD spectral signature. Therefore, in this Tank 104 pre-wash sample the presence of sodium titanium oxide ($Na_2Ti_2O_4(OH)_2$), as seen in the XRD spectra in Figure 2, insert A and the abundance of elemental titanium in the SEM/EDX spectra in Figures 5a-5d, is used to confirm the presence of MST in the Tank 104 pre-wash sample. It is worth noting that the large background shift in the XRD spectra in Figure 4, insert A, is partly an artifact of the glass slide used to mount the sample and the background shift due to amorphous MST mineral.

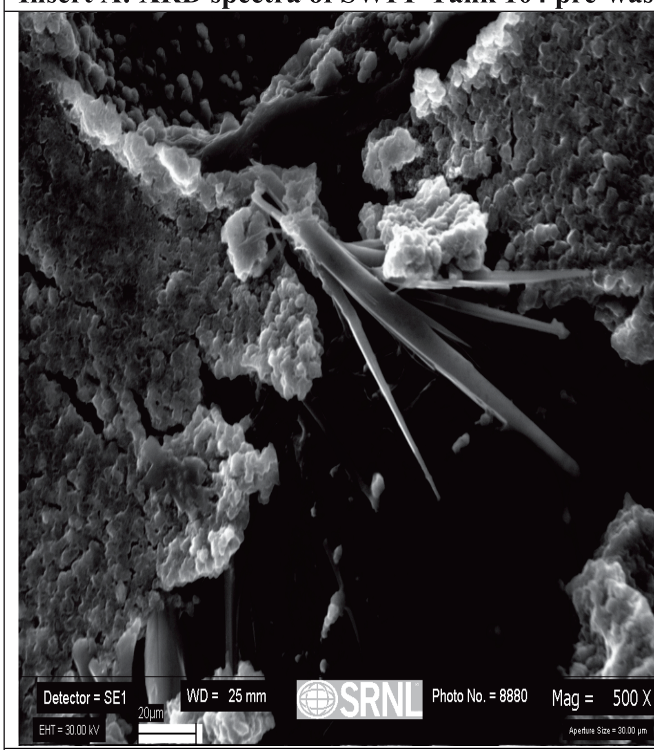
As shown in the SEM photographs in Figure 4, inserts B and C, the SWPF Tank 104 pre-wash solids show needle shaped and porous solid materials (a mixture of dried sodium salts, and titanium oxide minerals), which are probably crystalline sodium nitrate or sodium aluminum hydroxide crystals in the solid. The principal elemental compositions of this SWPF Tank 104 pre-wash solid material are iron, sodium, silicon, aluminum, titanium, nickel, chromium, mercury, and uranium as shown in Figures 5a-5e. In Figure 5c, uranium seems to be bound or absorbed onto the titanium mineral (Monosodium titanate). Thus, based on these elemental constituents, especially the relatively significant identifiable peaks for titanium, the presence of MST (sodium titanium oxide/ mono sodium titanate mineral), as seen earlier in the XRD data, can be inferred in the Tank 104 pre-wash solid material.

The particle size information for this Tank 104 pre-wash solids is based on the image from Figure 4 insert B, which constitutes the bulk of the material. Image analysis of these particles show that the particles sizes are in the 1-4 microns range with 2-3 microns being the most common diameter of these particles. Based on SEM-EDX of the various particles, their major elemental compositions include mostly Al-Na-Ti, and Na-O-Ti.

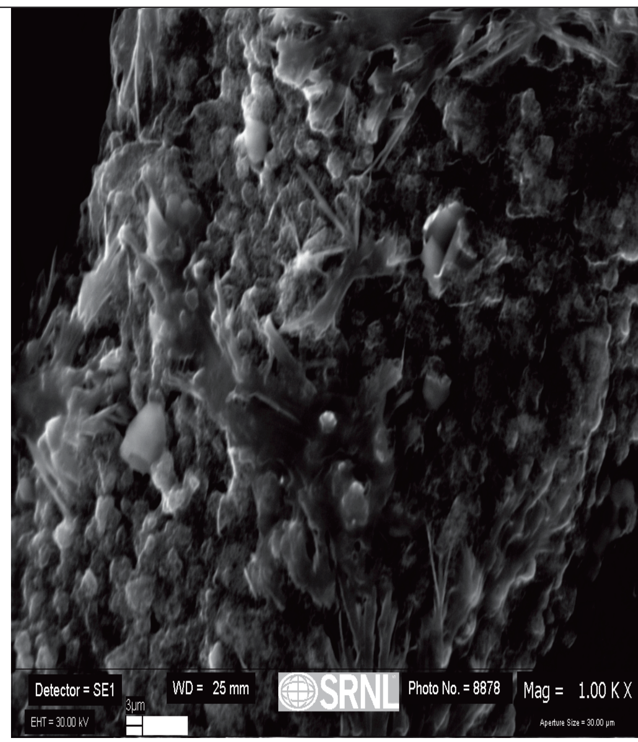
Based on these XRD and SEM/EDX characterization results for the pre-wash tank 104 solid sample, the identified minerals, and chemical elements, which make up the Tank 104 pre-wash solids, may not be uniformly distributed throughout the Tank 104 pre-wash solids. For example, elemental mercury and uranium are seen only at certain locations within the SWPF Tank 104 pre-wash solid sample.



Insert A: XRD spectra of SWPF Tank 104 pre-wash solids

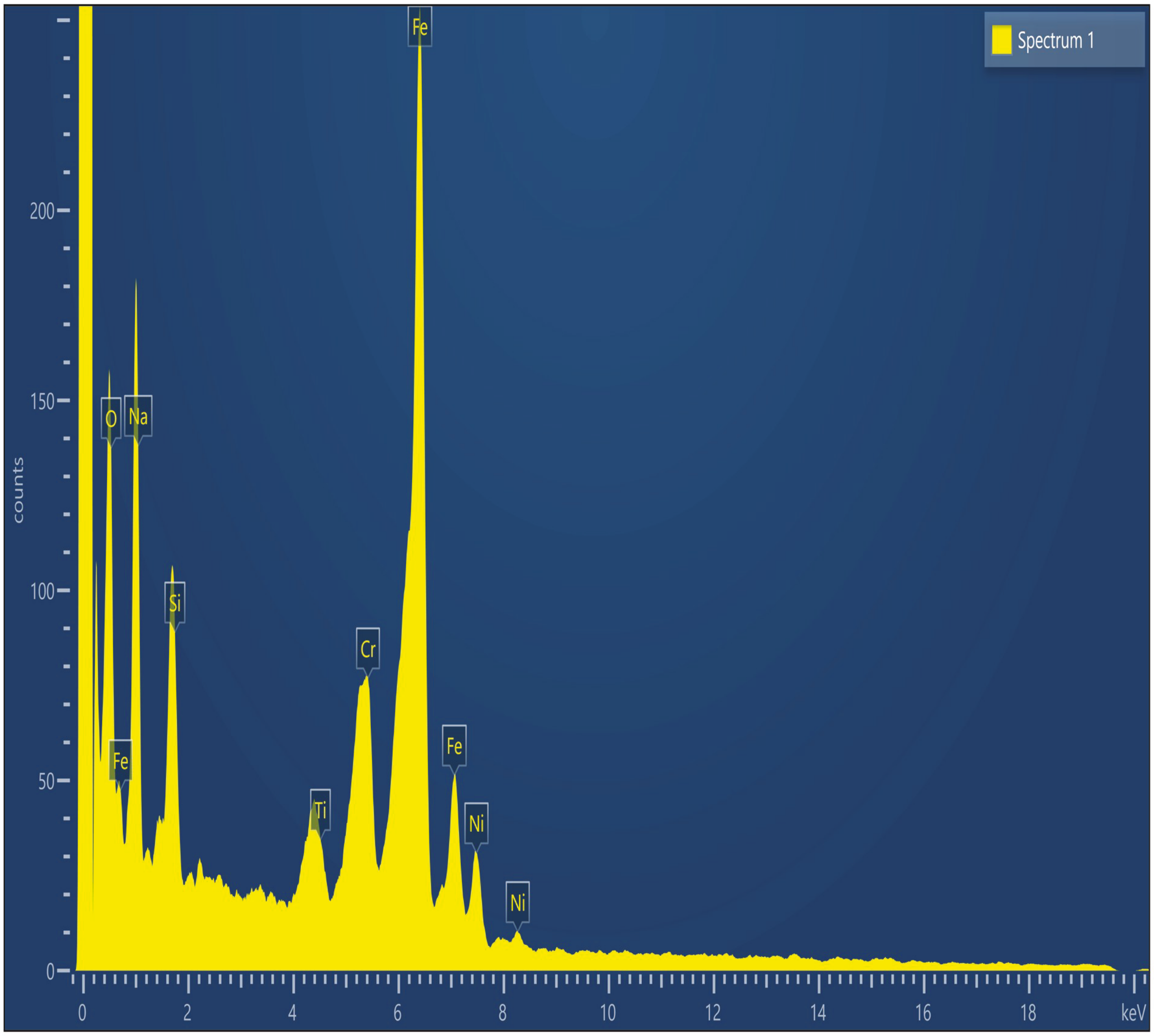


Insert B: A:500-fold magnification of the Tank 104 solid



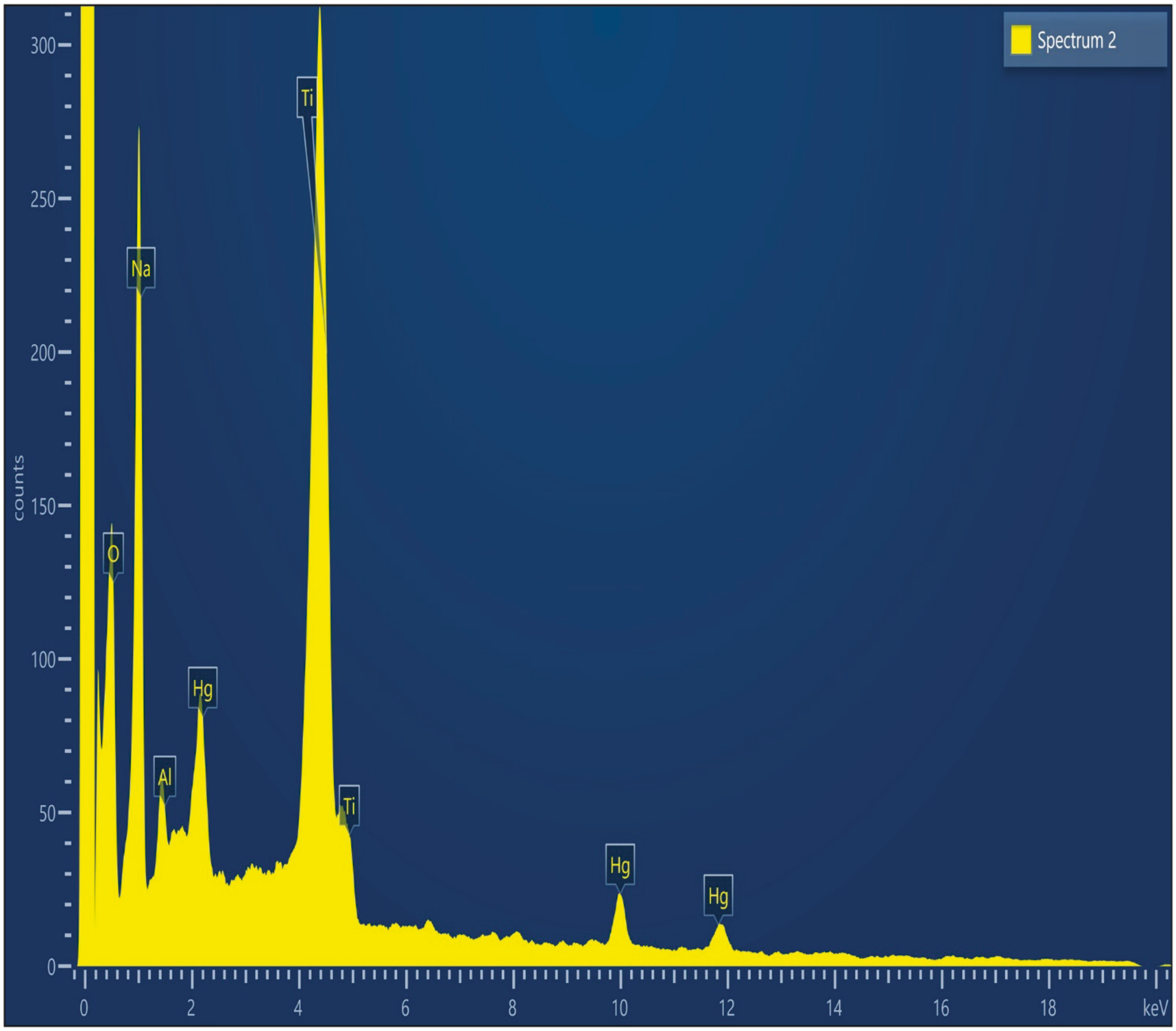
Insert C: 1000-fold magnification of the Tank 104 solid

Figure 4. XRD Spectra (Insert A) and SEM photo images of the Tank 104 pre-wash solid particles



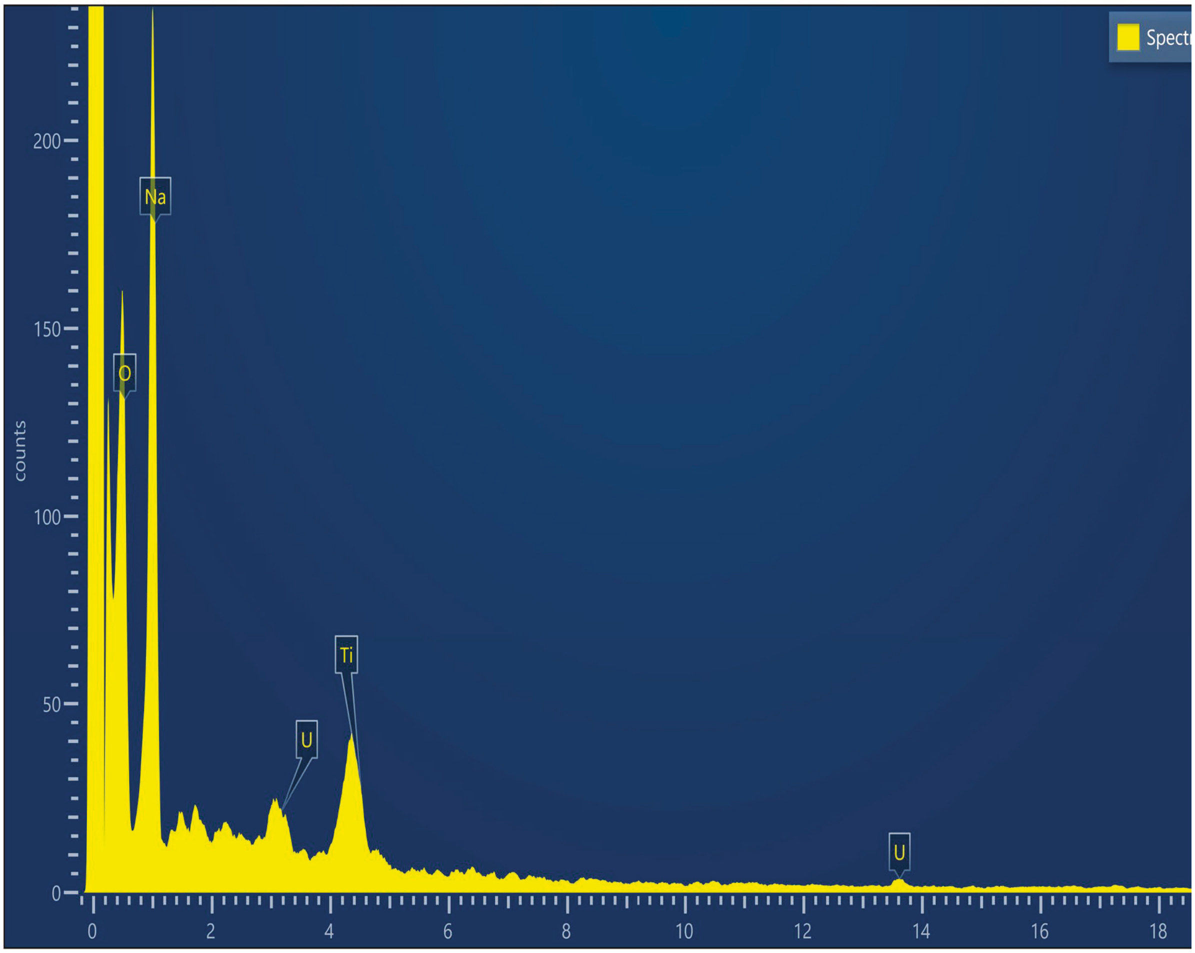
Al, Na, Si, Ti, Cr and Ni are the predominant elements present in the sample.

Figure 5a. SEM/EDX elemental composition for SWPF Tank 104 pre-wash solid material.



Elements present in the sample also include Hg, and Ti. Mercury is not uniformly distributed in the sample.

Figure 5b. SEM/EDX elemental composition for SWPF Tank 104 pre-wash solid material.



U and Ti are also present together in some regions of the sample; meaning U is probably bound on the titanium substrate.

Figure 5c. SEM/EDX elemental composition for SWPF Tank 104 pre-wash solid material.

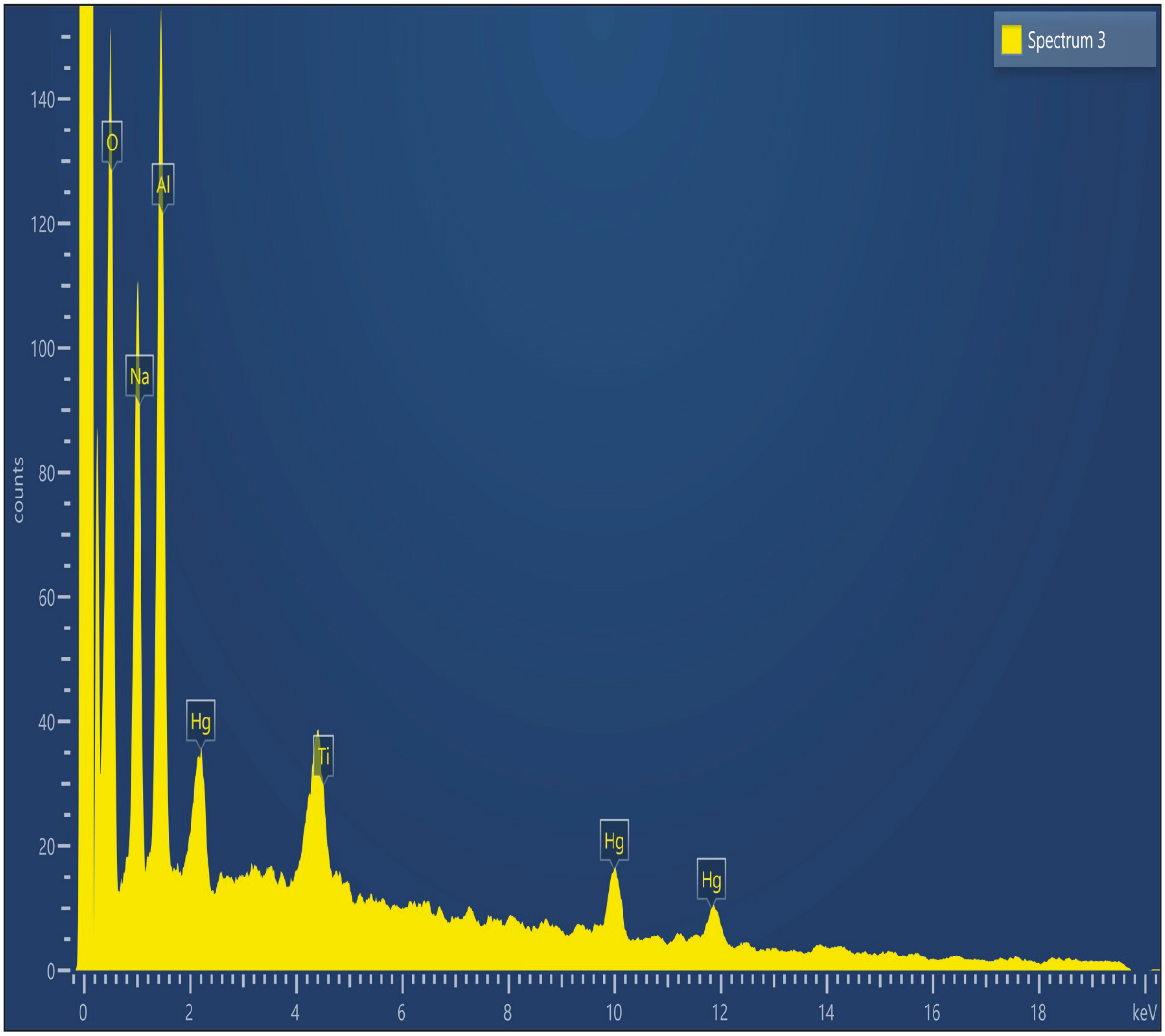
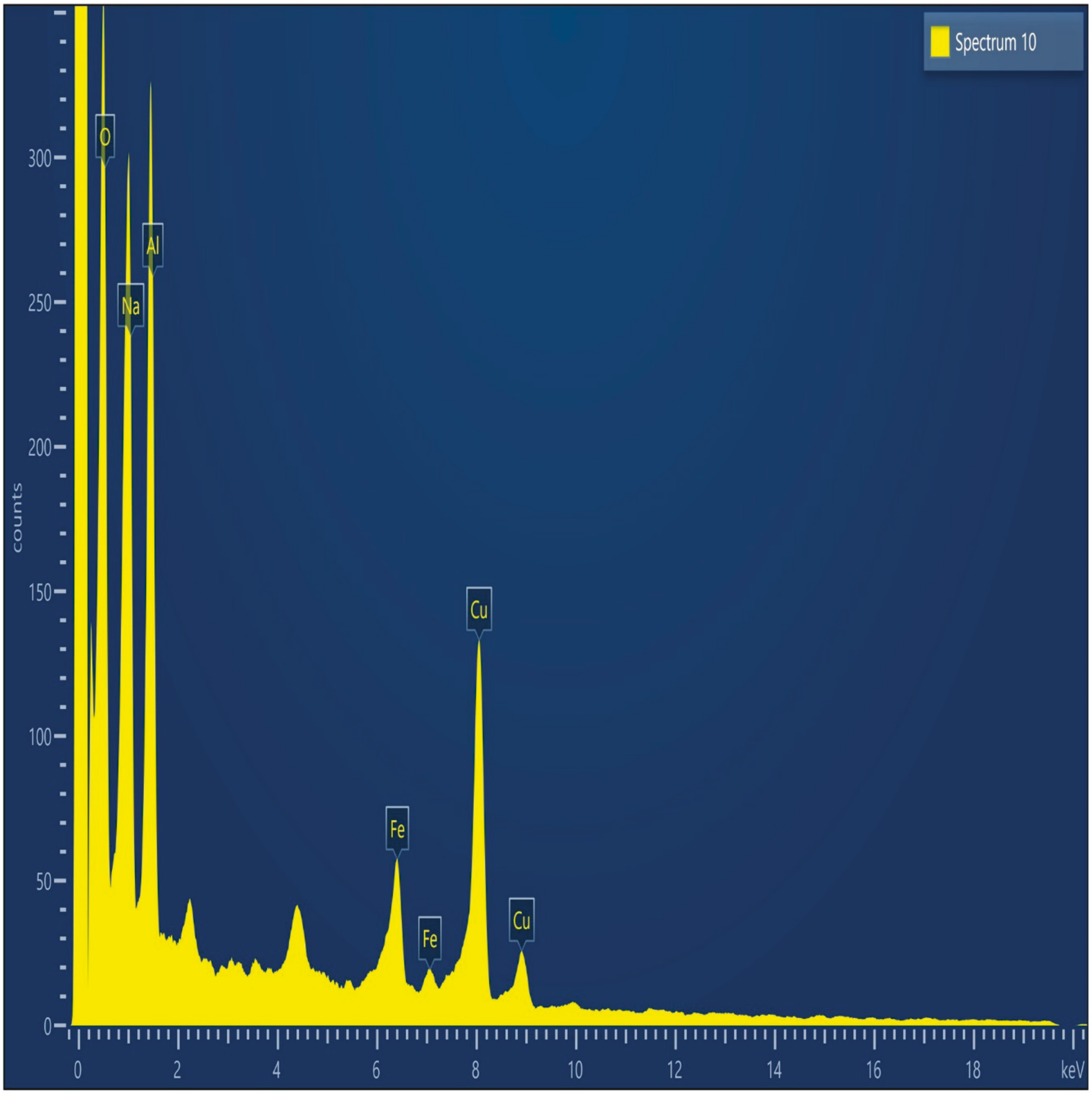


Figure 5d. SEM/EDX elemental composition for SWPF Tank 104 pre-wash sample



Addinal elements present in this Tank 104 pre-wash sample include copper and iron

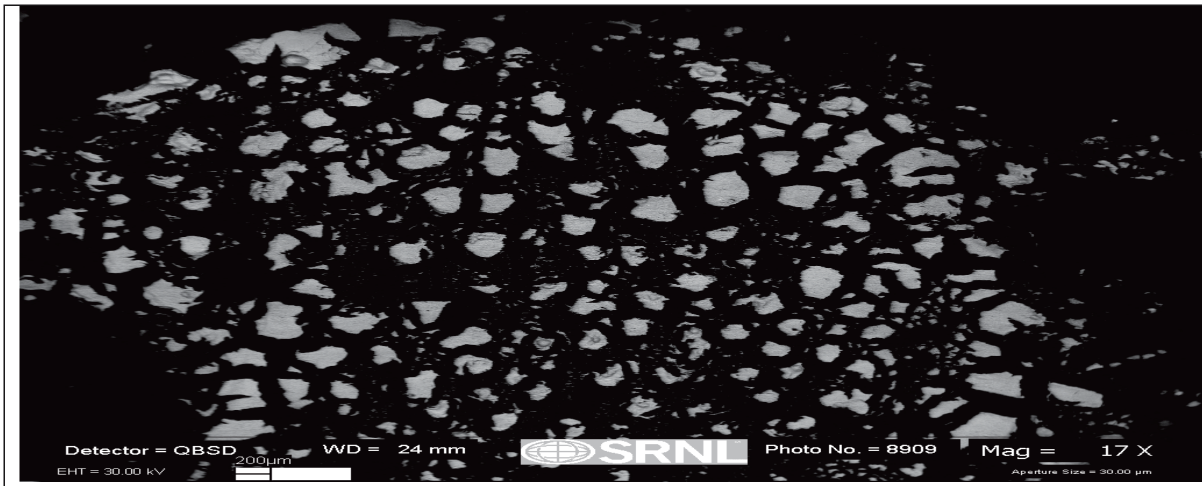
Figure 5e. SEM/EDX elemental composition for SWPF Tank 104 pre-wash sample

3.2 Tank 104 washed solids

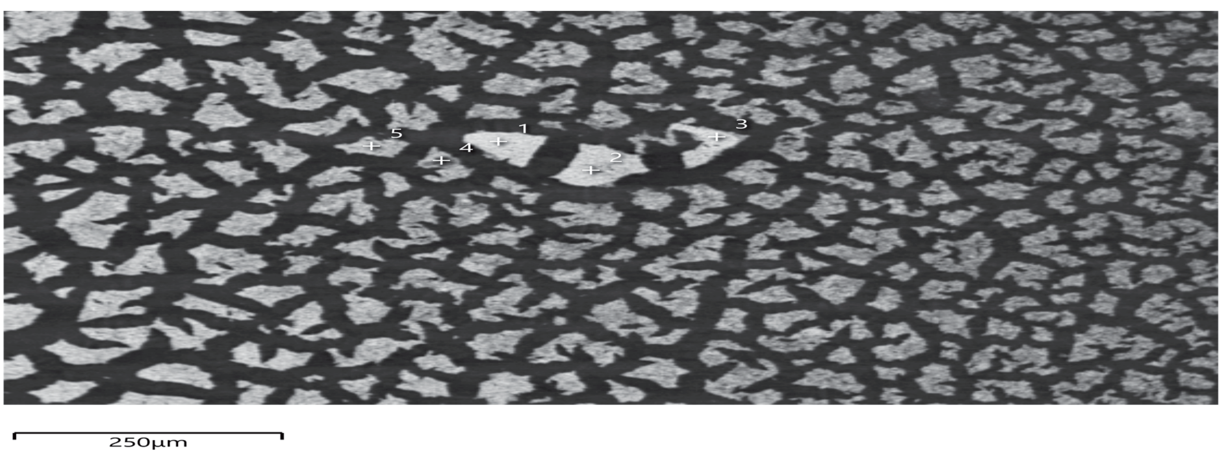
A representative SEM photo image of the Tank 104 washed sample solids is shown in Figure 6, insert A and the XRD spectra for this washed Tank 104 solids sample is shown in Figure 7, Insert A. In the XRD spectra, shown in Figure 7, insert A for this washed Tank 104 sample, only titanium mineral [Sodium titanium oxide, $Na_2Ti_2O_4(OH)_2$] is left; other minerals, as seen in the pre-washed Tank 104 solid sample [Figure 4, insert A] had been leached out during the washing process. The significant base line shift in Figure 7, insert A is partly because of the presence of titanium mineral (sodium titanium oxide, $Na_2Ti_2O_4(OH)_2$)), which is an amorphous material. The SEM/EDX representative spectra for the Tank 104 washed solids is also presented in Figures 8.

The elemental composition for all particle locations in the Tank 104 washed solids contained only Na and Ti. As shown in Figures 7, insert A, the washing process seemed to have taken out most of the interstitial salt solutions/solids in the sample matrix.

The washed Tank 104 solids data did not contain elements like Hg, Cu, Ni, U, Si or Fe, which were present in the Tank 104 pre-wash sample as shown earlier in Figures 5a- 5e above. However, the SEM /EDX images for this Tank 104 washed sample still contained elements like titanium and sodium, which were also abundant in the Tank 104 pre-wash sample. The presence of both Ti and Na in this washed Tank 104 sample indicates that the principal mineral observed in the XRD spectra for both SWPF tank solids is the same: sodium titanium oxide, $Na_2Ti_2O_4(OH)_2$. The washing process mainly took out elemental mercury, copper, nickel, uranium, silicon, and iron; possibly because these elements are not part of the crystalline mineralogy for the washed Tank 104 sample solids but were seen in the XRD for the pre-wash Tank 104 solid sample.

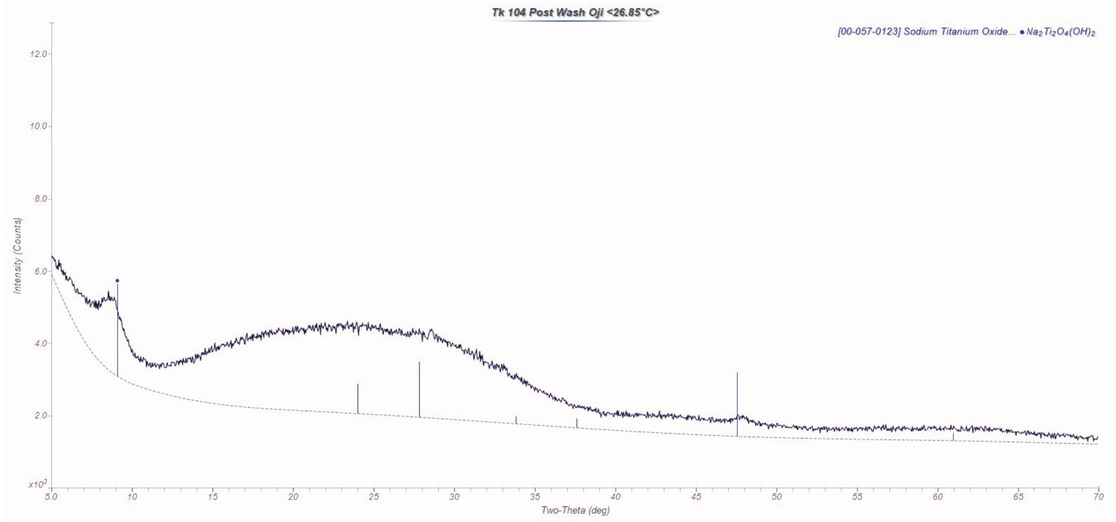


Insert A: SEM photo image of the Tank 104 washed solid particles (August 09, 2021 sample)

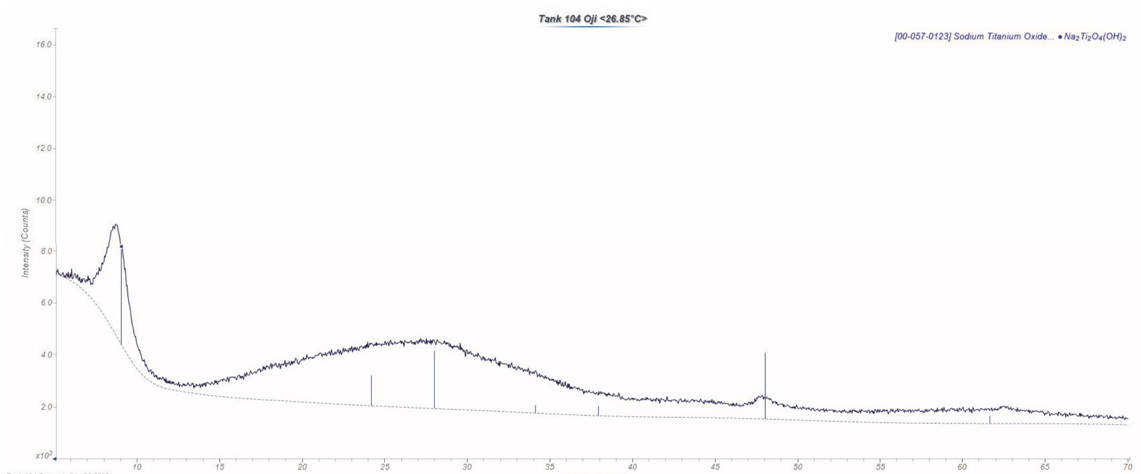


Insert B: SEM photo image of the Tank 104 washed solids, September 24, 2021 re-sample

Figure 6. SEM photo image of the Tank 104 washed solid particles.

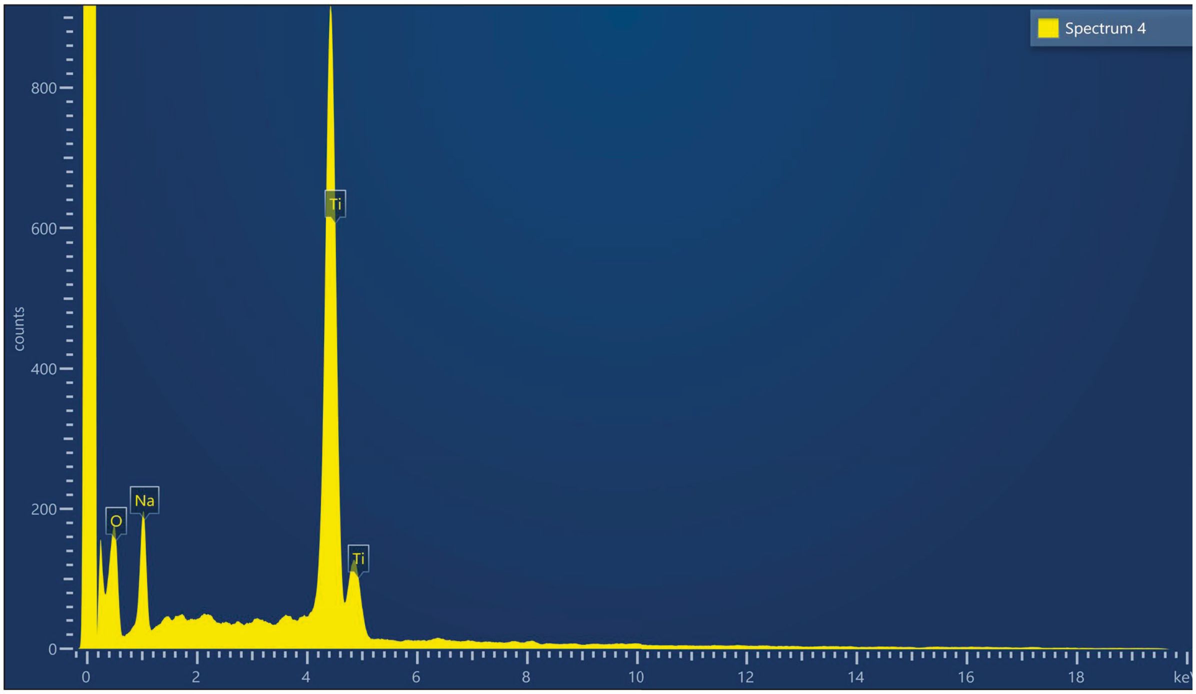


Insert A: XRD Spectra for the Tank 104 washed solid sample (August 09,2021 sample)



Insert B: XRD Spectra for the Tank 104 washed solid re-sample (September 24,2021 re-sample)

Figure 7. XRD Spectra for the Tank 104 washed solid samples (August and September 2021 samples); only titanium mineral is identified in both XRD spectra.



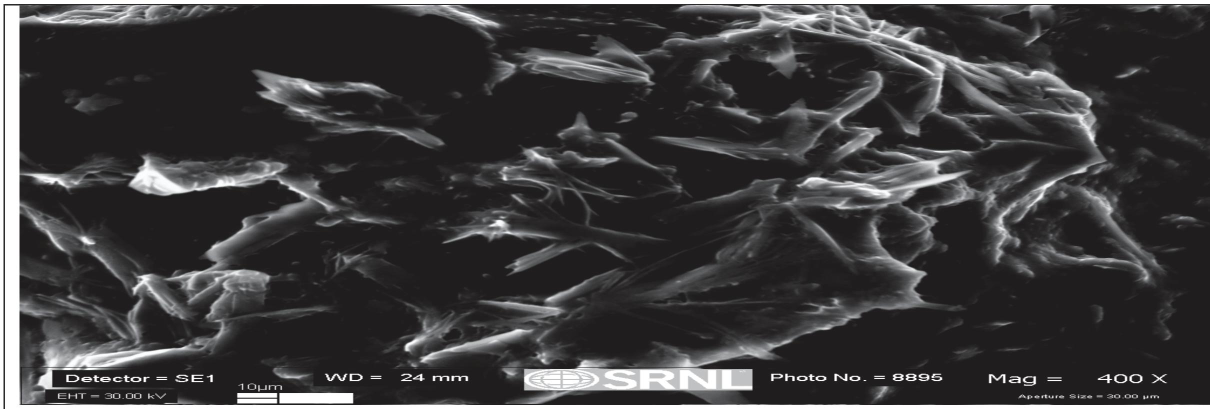
Elements present in the sample also include mainly Na and Ti

Figure 8a. SEM/EDX elemental composition for SWPF Tank 104 washed sample solids

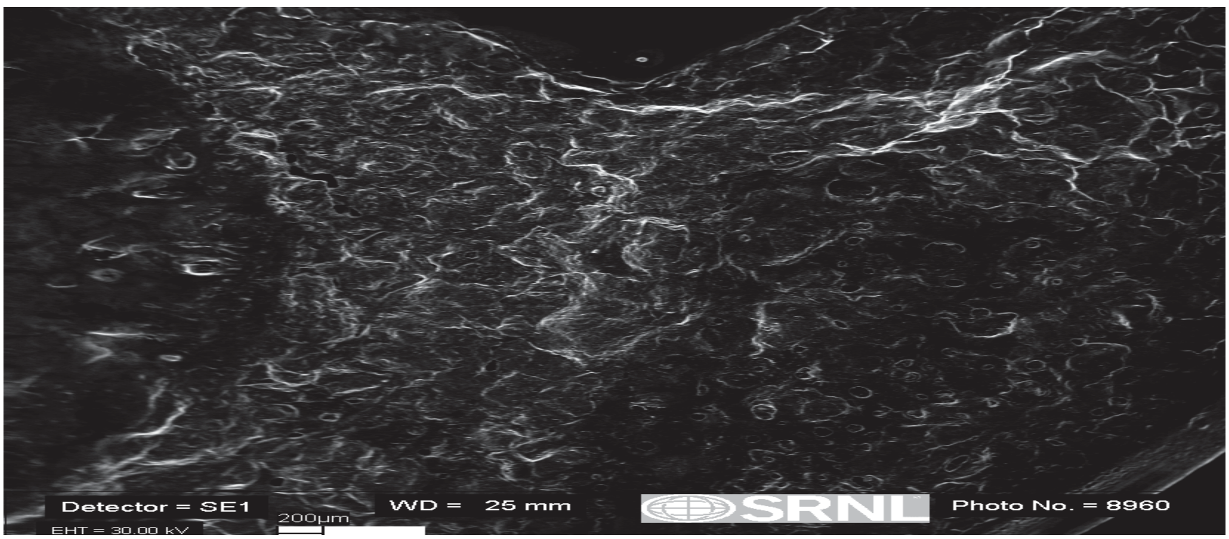
3.3 Tank 101 sample solids

SEM Photo image of representative SWPF Tank 101 solid sample is shown in Figure 9 insert A, and the XRD mineral spectra is shown in Figure 10, insert A. The XRD spectra shows the presence of a few crystalline mineral phases, mainly sodium nitrate or nitratine ($Na(NO_3)$), sodium carbonate ($Na_2CO_3{*}H_2O$), bayerite ($-Al(OH)_3{*}Al(OH)_3$), trona ($Na_3H(CO_3){*}H_2O$), and Sodium aluminum oxide hydroxide hydrate ($Na_2(Al_2O_3(OH_2)_2{*}5H_2O$).

Representative SEM/EDX spectra for the Tank 101 solids sample are presented in Figures 11a-11c.

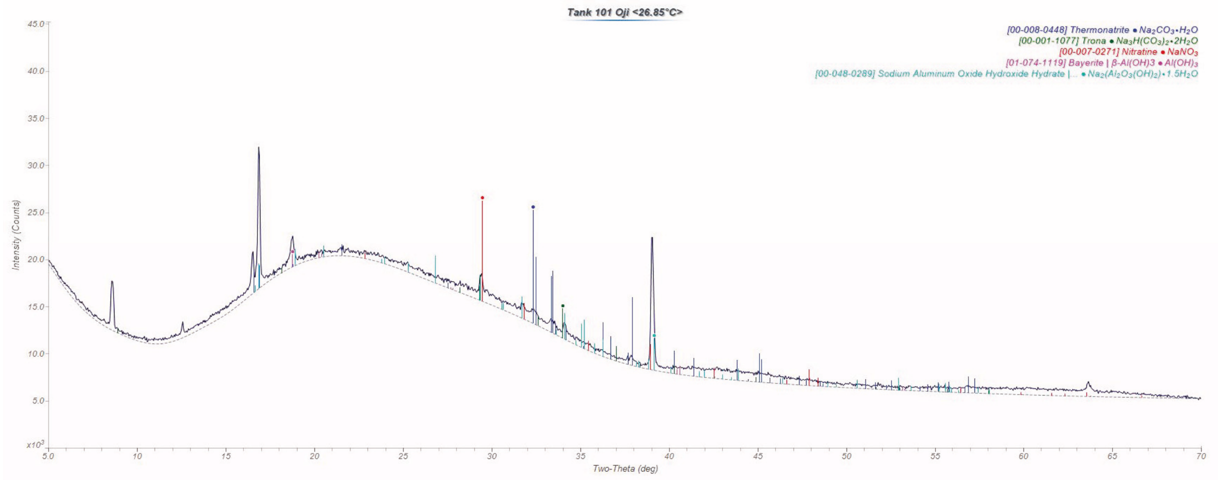


Insert A: SEM photo image of the Tank 101 solid particles (Augusta 09, 2021 sample).

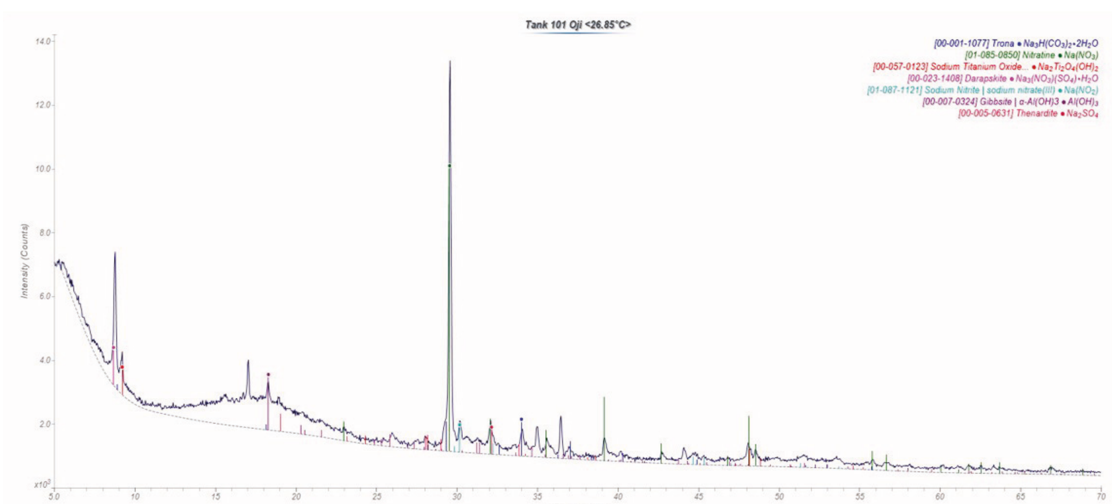


Insert B: SEM photo image of the Tank 101 solid particles (September 24, 2021 re-sample).

Figure 9. SEM photo image of the Tank 101 solid particles (Augusta and September 2021 samples).



Insert A: XRD Spectra for the Tank 101 solid sample (Augusta 09, 2021 sample)



Insert B: XRD Spectra for the Tank 101 solid re-sample (September 24, 2021 sample)

Figure 10. **XRD Spectra for the Tank 101 solid samples (Augusta and September 2021 samples)**

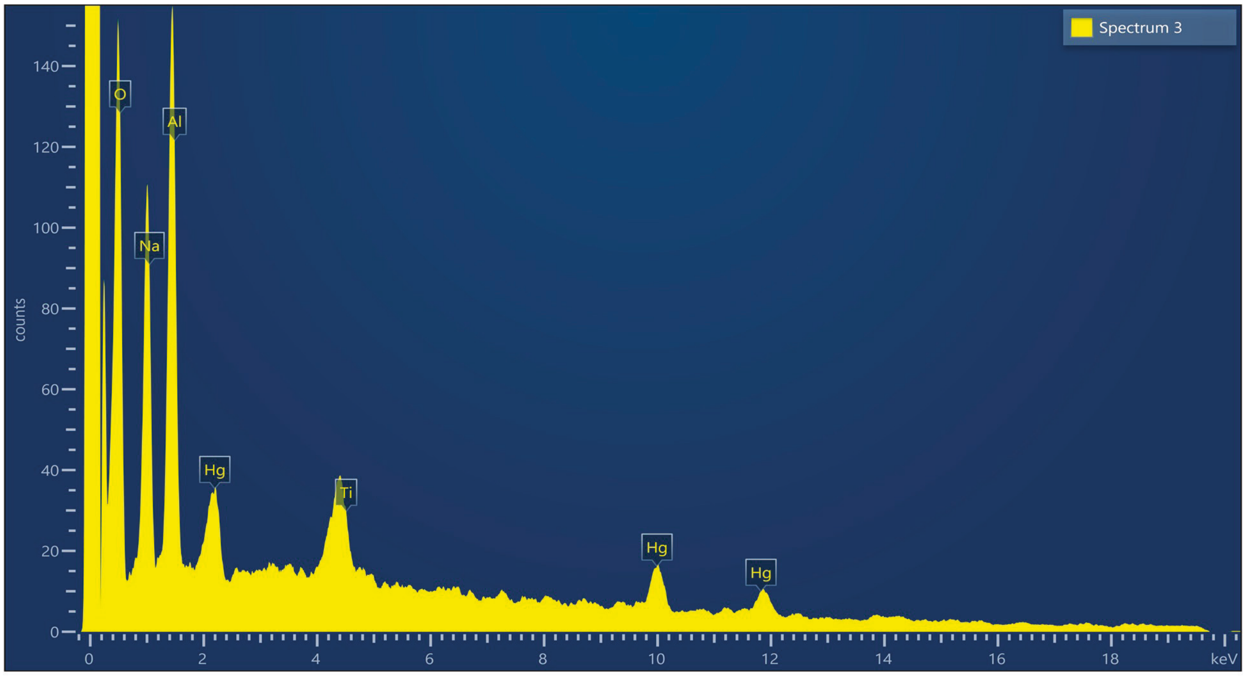


Figure 11a. SEM/EDX elemental composition for SWPF Tank 101 sample solids

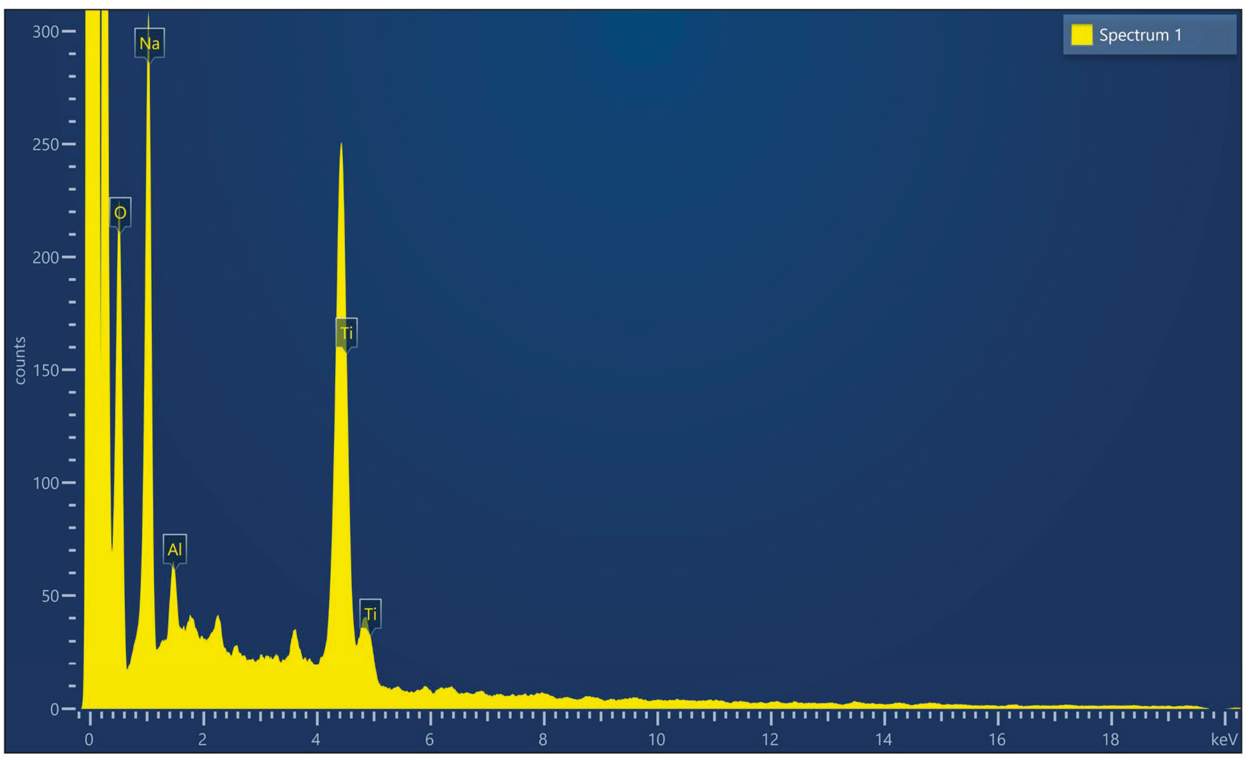


Figure 11b. SEM/EDX elemental composition for SWPF Tank 101 sample solids

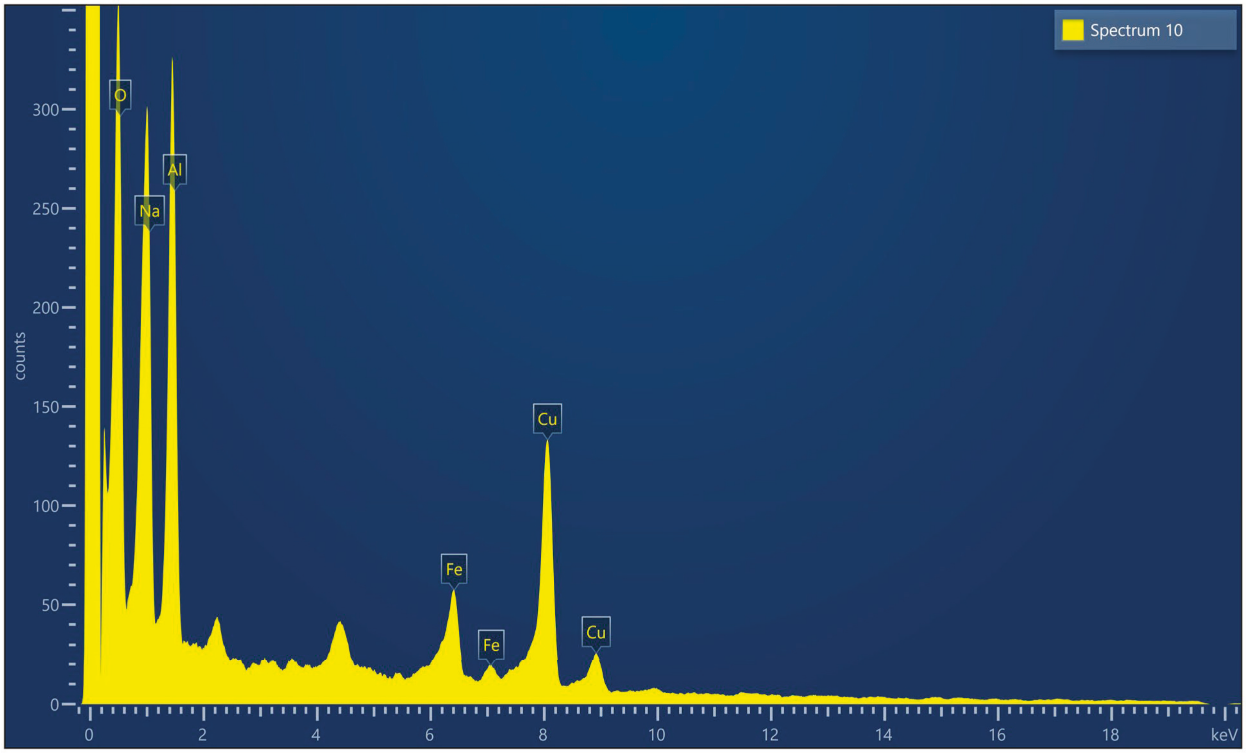


Figure 11c. SEM/EDX elemental composition for SWPF Tank 101 sample solids

The elemental composition for the Tank 101 solids includes mainly Na, Al, Hg, Ti, Cu, and Fe as shown in Figures 11a-11c. Image analysis of the photo images for the Tank 101 solids shows that the particles sizes are in the range of 2-4 microns.

The most recent analytical results for the SWPF feed tank solution chemistry reports the free-OH concentration as 2.4 molar[1]. Based on previous SRS tank farm solution chemistry experience[2] for the supernatant liquid phases, the formation of mineral solids, especially the insoluble aluminosilicates, seem to be more prevalent only when the free-OH concentration in the tank liquid phases is less than 2.0 molar. In other words, the more caustic the feed solution is the higher the solubility of insoluble salts like bayerite and gibbsite minerals. Therefore, it is not expected that the SWPF tank farm feed solutions, with a free-OH concentration above 2.0 molar, will contain appreciable amounts of solids, especially the insoluble solids like zeolite A, sodalite, and cancrinite.

The XRD mineralogy results for the Tank 104 pre-wash solids and that of the Tank 101 solids [Figure 4 insert A, and Figure 10, insert A] are mainly small amounts of sodium minerals as discussed above with the presence of bayerite (-$Al(OH)_3$*$Al(OH)_3$ (or gibbsite) in the Tank 101 sample being the main mineral difference between these two samples.

The semiquantitative data summarized in Table 1, is the average weight percent data for the elements identified at various locations on the SEM/EDX photographs for Tank 101, Tank 104-pre-wash and Tank 104- washed samples. The average weight percent data for the principal elements in each of the three samples are summarized for sodium (Na), aluminum (Al) and titanium (Ti). The other trace elements occur within the sample solids at one or two locations on the bulk of the solids.

Table 1. Average Weight Percent Elemental Distribution for Tank 101, Tank 104 Pre-wash, and Tank 104 Washed Samples-July 26, 2021, August 09, 2021 Samples

Elemental components	Tank 101, wt.%	%RSD	Tk 104 pre-wash, wt.%	%RSD	Tk 104 washed, wt.%	%RSD
Sodium (Na)	27.3	30.0	38.6	6.0	9.3	18.3
Aluminum (Al)	12.6	90.5	3.0	27.0	MDL	-
Titanium (Ti)	11.1	60.4	11.7	30.0	32.0	23.4
Other trace elements occasionally seen once or twice in the SEM/EDX spectrographs.						
	Tank 101, wt.%	**%RSD**	**Tk 104 pre-wash, wt.%**	**%RSD**	**Tk 104 washed, wt.%**	**%RSD**
Calcium (Ca)	MDL	-	MDL	-	MDL	-
Iron (Fe	**2.2**	-	13.4	-	MDL	-
Copper (Cu)	**5.7**	43.4	MDL	-	MDL	-
Silicon (Si)	MDL	-	7.3	-	MDL	-
Chromium (Cr)	MDL	-	**2.7**	-	MDL	-
Nickel (Ni)	MDL	-	**4.2**	-	MDL	-
Uranium (U)	MDL	-	MDL	-	MDL	-
Mercury (Hg)	**8.3**	-	MDL	-	MDL	-

MDL = Below instrument measurement limits. STDEV = one sigma standard deviation.

The average wt.% sodium (Na), as shown in Table 1, for Tank 101 is 27.3 ± 8.2 wt.% (30 %RSD) and that for the Tank 104 pre-wash sample is 38.6 ± 2.3 wt.% (6.0 %RSD). This relative increase in sodium content of the Tank 104 pre-wash is not expected because sodium concentration is normally lowered from the Tank 101 to Tank 104 and the sodium molarity goes from 6.4 to 5.6 molar. This observation in sodium concentration in the two tanks cannot be attributed to caustic adjustment from the addition of sodium as sodium hydroxide. However, the semiquantitative sodium concentration data for the Tank 101 with a 30 %RSD may be the source of this unexpected reversal of the sodium concentration order in the Tank 104 pre-wash sample. There is also a significant drop in the sodium concentration down to 9.3 ± 1.7 wt.% (18.3 %RSD) in the Tank 104 washed sample possibly because of the leaching out of soluble sodium.

The semiquantitative result for aluminum in the Tank 101 averaged 12.6 ± 11.4 wt.% (90.5 %RSD), which drops to an average low of 3.0 ± 0.8 wt.% (27.0 %RSD) in the Tank 104 pre-wash sample. This drop in aluminum concentration in the Tank 104 pre-wash sample may be due to dilutions and the reaction /dissolution of aluminum minerals because of the addition of caustic solution. ~~Of course, the aluminum concentration in the Tank 104 washed sample is expected to be less than instrument detection limits because most of the aluminum had been leached out of the solids during washing.~~

The average wt.% titanium [11.1 ± 6.7 wt.% (60.4 %RSD)] in the Tank 101 sample seems to be about the same order of magnitude as the average wt.% titanium in the Tank 104 pre-wash sample at 11.7 ± 3.5 (30.0 %RSD). These semiquantitative data for titanium in the solids cannot be correct because the Tank 101 sample was taken before any adjustment or MST addition, in other words, the Tank 101 sample was material directly transferred from Tank 49. Again, with a 60 %RSD for the titanium data the high uncertainty in the Tank 101 sample titanium data may be the reason for this inconsistent result.

The relatively large increase in the average wt.% titanium in the Tank 104 washed sample [32.0 ± 7.5 wt.% (23.4 %RSD)] is due to the absence of all other solids which had dissolved and washed out during the washing process. Other trace elements occasionally seen at one of two places in the bulk of the SEM photographic images, especially in the Tank 104 pre-wash sample solids, include iron, silicon, chromium, and nickel. A measurable amount of total mercury and copper is also present in the Tank 101 sample.

3.4 Tank 101 and Tank 104 Washed Sample Solids- September 24, 2021 Re-Samples

As earlier mentioned, it was necessary to re-sample the Tank 101 sample and Tank 104 washed sample to confirm the initial XRD and SEM characterization results with a larger sample size.

The measured particle sizes for the Tank 101 re-sample solids material now ranged from 4 to 11 microns, with an average of about 8 microns. This is considerably larger than the particle sizes for the August 09, 2021 Tank 101 solids sample, with particle sizes in the ranged of 1-4 microns and 2-3 microns being the most common diameters.

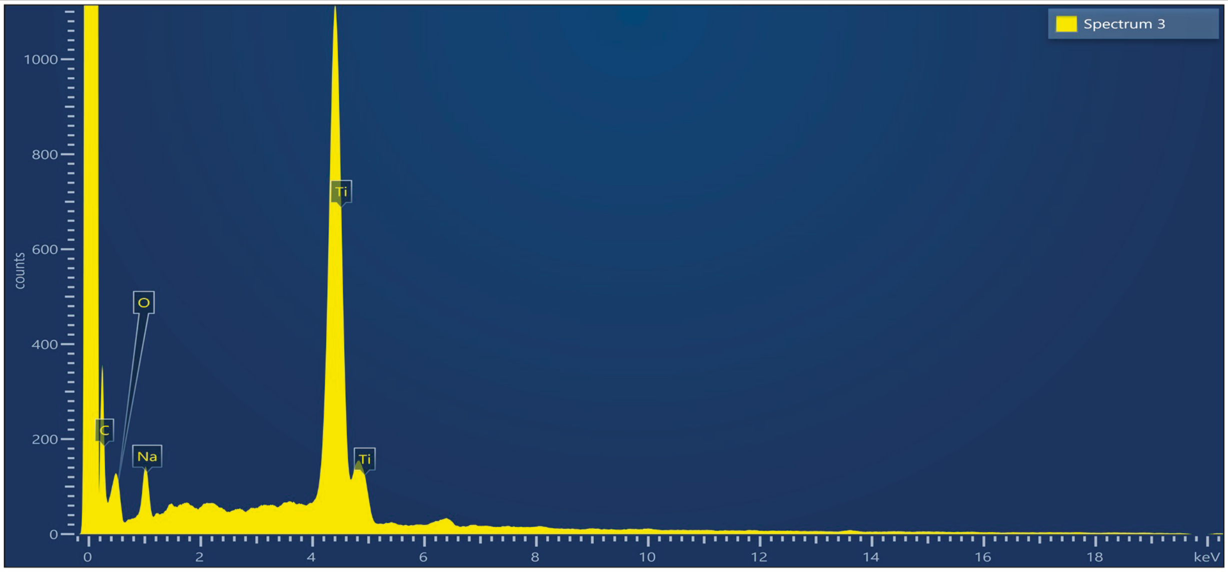
The particle size distribution for the Tank 104 washed re-sample, on the other hand, now ranged from 0.5 to 2.5 microns with an average diameter of 1 micron. This particle sizes are considerably smaller than the particle sizes of the initial August 09, 2021 Tank 104 washed sample, which ranged from 1-4 microns with 2-3 microns being the most common diameters.

For comparison, the SEM image photographs, and XRD spectra for the re-sampled Tank 104 washed sample and re-sampled Tank 101 solids have been put under the same figures with those from the August 09, 2021 Tank 104 washed sample and the Tank 101 solids sample as shown in Figure 6 (SEM images), Figure 7 (XRD spectra), 9 (SEM images), and 10 (XRD spectra).

The SEM image photographs for the two Tank 104 washed solids, at different magnifications, are shown in Figure 6, insert A, for the August 09, 2021 Tank 104 washed solids, and insert B for the re-sampled Tank 104 washed solids. Apart from the different image background lighting and particle size difference for these Tank 104 washed solids (re-sampled and original August 09, 2021 Tank 104 washed samples) the two samples look alike.

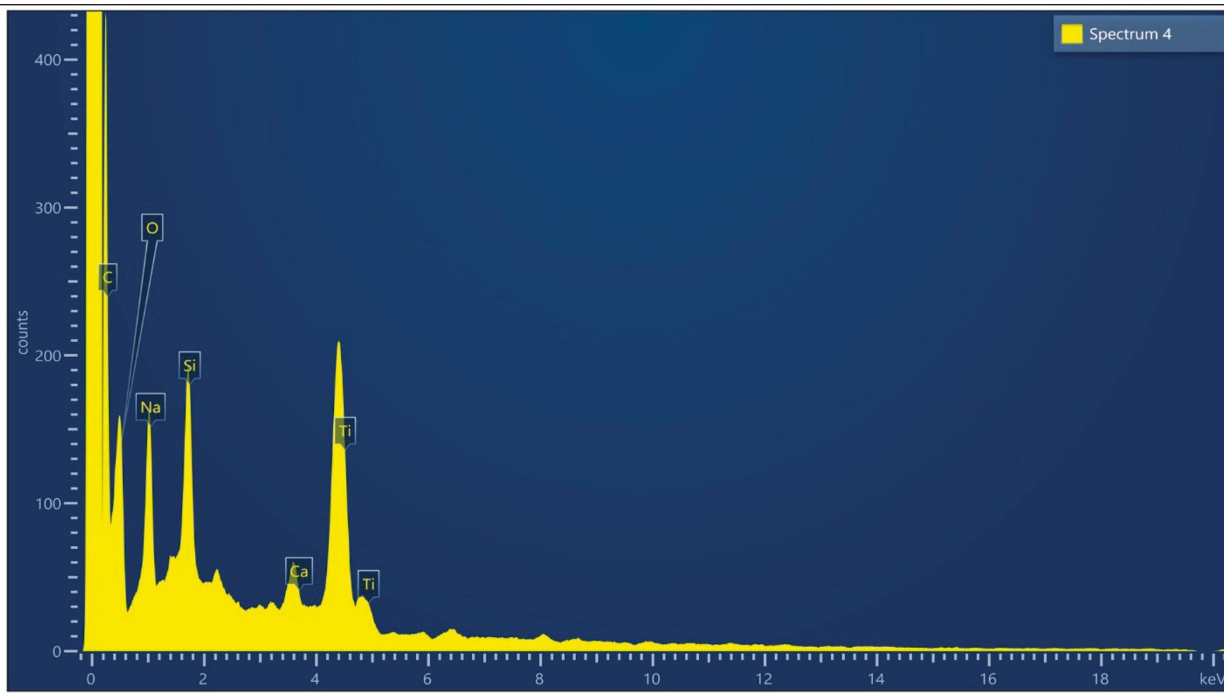
The XRD spectra, as shown in Figure 7, for these two samples are similar although a larger background shift is seen in the August 09, 2021 Tank 104 washed sample (Figure 7, insert A). This is due to the smaller sample size used for this XRD characterization, which means a higher background shift contribution from the sample holder. Both samples show the same predominant sodium and titanium minerals and elemental composition of sodium and titanium after the washing process (Figure 7, 8a,12a, and 12 b). As shown in Figure 12b and Table 2, other trace elements seen in the Tank 104 washed re-sample include calcium, iron, and silicon.

The SEM images for the two Tank 101 solids (re-sample and August 09, 2021 samples) are shown in Figure 9, inserts A and B. These two images are similar and show the presence of interstitial salts. Please note that the images are taken at different magnifications. As earlier mentioned, the particle sizes for these two samples are different, possibly due to different sample weights, sampling locations/sampling and settling issues.



The predominant elements present in the Tank 104 washed sample are mainly Na and Ti.

Figure 12a. SEM/EDX Major Elemental Compositions for Tank 104 Washed Re-sample

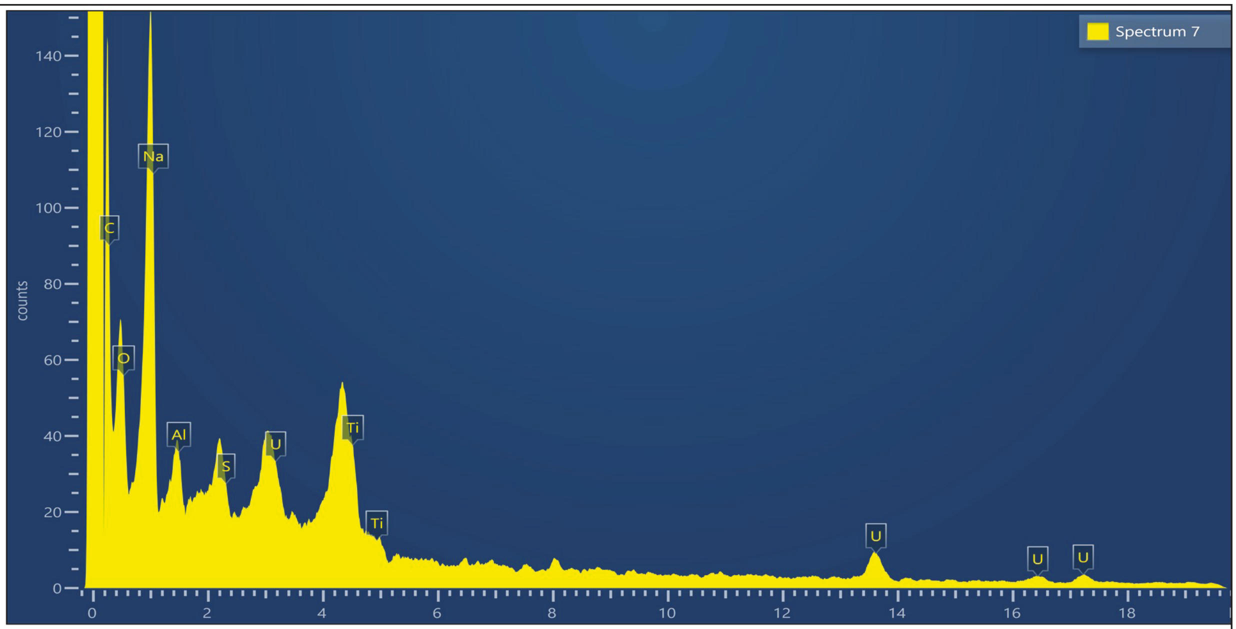


Other trace elements occasionally seen in the Tank 104 washed re-sample solids include traces of silicon and calcium

Figure 12 b. SEM/EDX Major elemental compositions for the Tank 104 Washed Re-sample

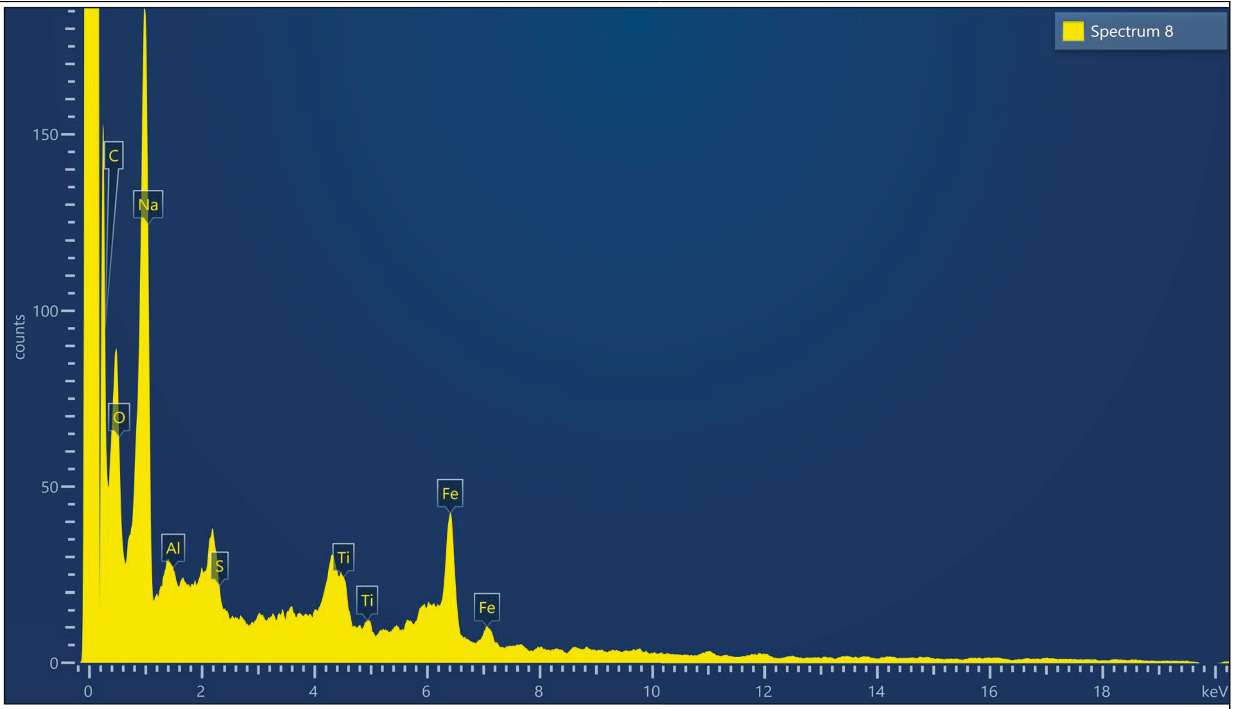
The XRD mineral content for these two Tank 101 solids (re-sample and August 09, 2021 sample), shown in Figure 10, inserts A and B have three minerals in common and these are trona ($Na_3H(CO_2)_2{*}2H_2O$, nitratine ($NaNO_3$), and bayerite/ gibbsite (a-$Al(OH)_3{*}Al(OH)_3$. The differences in the mineralogy of these two samples collected at different times is the presence of a sodium sulfate mineral called darapskite ($Na_3(NO_3)(SO_4){*}H_2O$), sodium titanium oxide ($Na_3TiO_4(OH)_2$), sodium nitrite ($NaNO_2$), and thenardite/sodium sulfate (Na_2SO_4), which are present in the Tank 101 re-sample but not in the August 09, 2021 Tank 101 sample. The Tank 101 re-sample does not contain the aluminum oxide hydroxide phase ($Na_2(Al_2O_3(OH_2){*}H_2O$), and thermonatrite ($Na_2CO_3{*}H_2O$) found in the August 09, 2021 Tank 101 sample.

The major elemental compositions for the Tank 101 re-sample, as shown in representative Figures 13a-13c include Na, Al, Ti, U, S, and Fe. These elements were also present in the August 09, 2021 Tank 101 sample (Figures 11a-11c). However, this Tank 101 re-sample SEM EDX characterization results do not seem to contain measurable amounts of traces of Cu, which was present in the August 09, 2021 Tank 101 sample solids. The Tank 101 re-sample also contained detectable amounts of sulfur, which was not seen in the August 09, 2021 Tank 101 sample (Table 2).



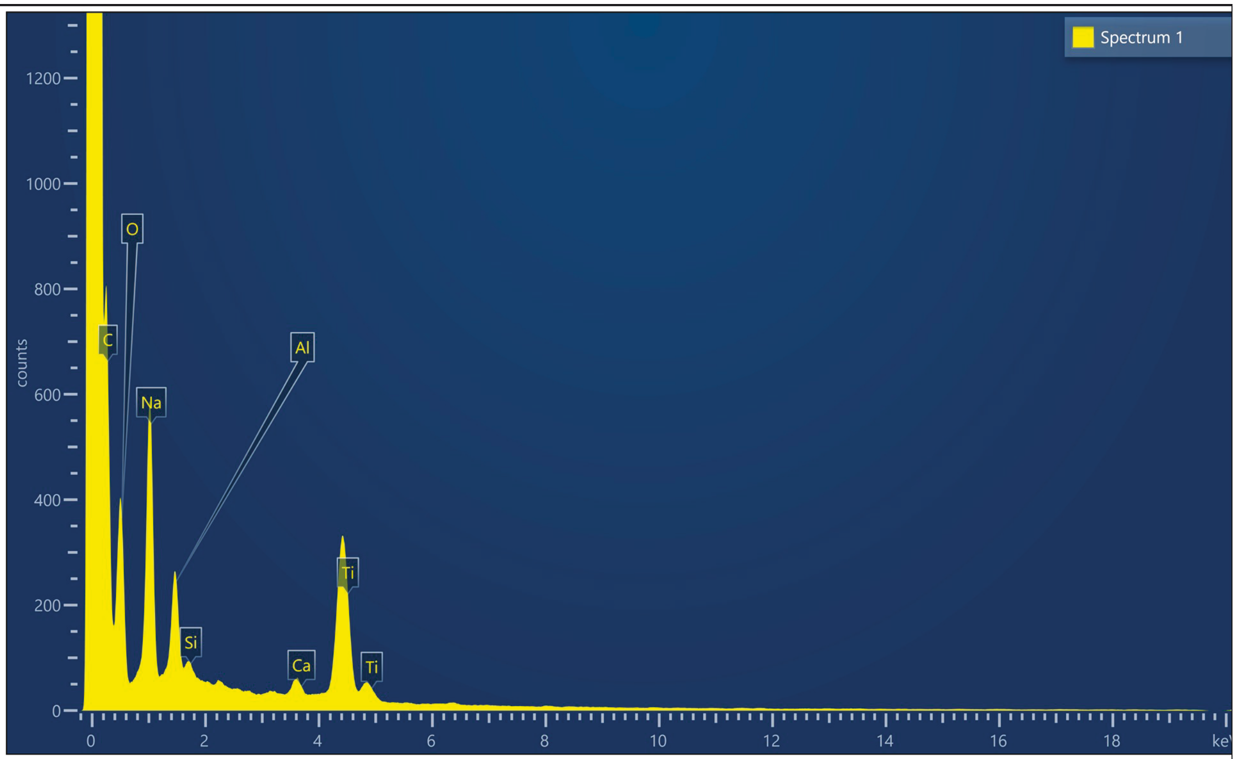
Na, Al, S, Ti, U,

Figure 13a. SEM/EDX Elemental Compositions for Tank 101 Re-sample



Na, Al, Ti, S, Fe

Figure 13b. SEM/EDX Elemental Compositions for Tank 101 Re-sample



Na, Si, Al, Ca, Ti

Figure 13c. SEM/EDX Elemental Compositions for Tank 101 Re-sample

As earlier mentioned, the re-sampling of the Tank 101 and Tank 104 washed samples for these XRD and SEM/EDX characterizations increased the representative sample weights from 12 and 25 mg to 140 and 450 mg, respectively, which is more than a 10-fold increase in characterization sample size.

The effect of this larger sample size on the Tank 101 re-sample was more significant in terms of XRD spectral quality and number of minerals in identified. As outlined above, a total of seven minerals were identified in the Tank 101 re-sample spectra compared to a total of five minerals the August Tank 101 sample. On the other hand, the relative XRD spectra intensities for these minerals were more definite in the Tank 101 re-sample (Figure 10, insert B) because of the minimal baseline background shift when compared with the original Tank 101 characterization (Figure 10, insert A, Augusta 09, 2021 sample).

The XRD spectra for this Tank 101 re-sample (Figure 10, insert B), shows that sodium nitrate mineral or nitratine ($Na(NO_3)$), spectral signal peak is the most pronounced peak of all the crystalline minerals identified. The peaks for some of the other minerals are barely above the signal-to-noise ratio for the XRD spectra baseline. This means that soluble $Na(NO_3)$ is qualitatively the most abundant mineral in the Tank 101 sample or in other words, the Tank 101 sample solids contain mostly $Na(NO_3)$ mineral with smaller amounts of other crystalline minerals present as well.

Table 2. Average Weight Percent Elemental Distribution for Tank 101, and Tank 104 Washed Samples-September 2021 Re-Sample

Elemental components	Tank 101, wt%	%RSD	Tk 104 pre-wash, wt%	%RSD	Tk 104 washed, wt%	%RSD
Sodium (Na)	43.9	9.8	No Re-sample	na	**17.6**	*13.1*
Aluminum (Al)	5.3	41.5	No Re-sample	na	MDL	-
Titanium (Ti)	9.0	67.8	No Re-sample	na	**34.4**	*18.3*
Other trace elements occasionally seen once or twice in the SEM/EDX spectrographs.						
Other elements	**Tank 101, wt%**	**%RSD**	**Tk 104 pre-wash, wt%**	**%RSD**	**Tk 104 washed, wt%**	**%RSD**
Calcium (Ca)	MDL	-	No Re-sample	-	1.61	-
Iron (Fe)	10.0	na	No Re-sample	-	0.61	-
Copper (Cu)	MDL	-	No Re-sample	-	MDL	-
Silicon (Si)	2.5	na	No Re-sample	-	0.68	-
Chromium (Cr)	MDL	-	No Re-sample	-	MDL	-
Nickel (Ni)	MDL	-	No Re-sample	-	MDL	-
Uranium (U)	MDL	-	No Re-sample	-	MDL	-
Mercury (Hg)	7.9	10.1	No Re-sample	-	MDL	-
Sulfur (S)	3.2	25.0	No Re-sample	-	MDL	-

The average weight percent composition for the main elemental and other trace elemental components for the Tank 101 re-sample, and the Tank 104 washed re-sample are shown in Table 2. The average weight percent elemental sodium in the Tank 101 re-sample at 43.9 wt% (9.8 %RSD) is relatively higher than the average weight percent sodium in the August 09, 2021 Tank 101 sample solids, which was 27.3 wt% (30 %RSD) as shown in Table 1. Both aluminum and titanium average weight percent concentrations in the Tank 101 re-sample [5.3 wt% (41.5 %RSD) and 9.0 wt% (67.8 %RSD), respectively], are relatively smaller than their corresponding concentrations in the Tank 101 August 09, 2021 solids samples, which averaged 12.6 wt% (90.5 %RSD) and 11.1 wt% (60.4 %RSD), respectively.

On the other hand, the average weight percent composition for elemental titanium in the Tank 104 washed re-sample (34.4 wt% (18.3 %RSD) is statistically the same as the composition for elemental titanium in the August 09, 2021 Tank 104 washed sample, which averaged 32.0 wt% (23.4 %RSD). However, the average weight percent sodium in the August 09, 2021 Tank 104 washed sample (9.3 wt% (18.3 %RSD) was only about one-half the average sodium concentration in the Tank 104 washed re-sample, which was 17.6 wt% 13.1 %RSD.

The reason for all these concentration differences for Na, Al, and Ti concentrations in the two Tank 101 and Tank 104 washed samples may be due to sample acquisition differences, such as the analytical sampling amounts.

A review of the feed solution chemistry for the Modular Caustic Side Solvent Extraction Unit (MCU),[3] a predecessor of the current Salt Waste Process Facility (SWPF) process, and OLI* modeling results [4, 5, 6] for the MCU feed solution chemistry component [sodium salts and the corresponding anions, ranging from sodium carbonate, sodium chloride, sodium oxalate, sodium nitrate and nitrite, sodium phosphate, sodium sulfate and sodium fluoride and cations including aluminum, sodium, silicon, potassium and chromium ability to form solids during operations at 25 ^{0}C shows that there is little formation of solids when the feed solution used in the modelling includes silicon and aluminum feed components[4]. The OLI models show that the weight percent solids are normally in the range of 0.003 - 0.015 wt%. Even when the OLI model considers the addition of components known to generate insoluble solids like carbonate (Na_2CO_3), the model still predicts the formation of very small weight percent insoluble solids, which leads to an increase to about 0.061 wt% insoluble solids.

Overall, the OLI model predicted the formation of a very small amounts of insoluble solids with these typical MCU feeds salt solutions.

4.0 Conclusions

The SWPT Tank 101 and two Tank 104 solids (pre-wash and washed solids) samples, have been characterized using XRD and SEM/EDX techniques to address the concerns for the existence of insoluble solids, which may interfere with the operational performance of the SWPF crossflow filters.

The key results from the Tank 101 and Tank 104 XRD and SEM characterizations include the following.

- XRD data for the Tank 104 pre-wash sample show the presence of crystalline sodium compounds, mainly sodium nitrate $Na(NO_3)$, which are likely formed from the evaporation of salt solution.
- Insoluble titanium material [sodium titanium oxide ($Na_2Ti_2O_4(OH)_2$], possibly coming from added monosodium titanate (MST), sodium aluminum hydroxide ($NaAl(OH)_4$), and sodium carbonate ($Na_2CO_3.H_2O$) were also observed in this Tank 104 pre-wash sample.
- Only a titanium mineral phase (sodium titanium oxide, $Na_2Ti_2O_4(OH)_2$) is observed in the Tank 104 washed sample; the other minerals had been washed/leached out.
- Although the presence of small amounts of silicon and aluminum elements were observed in the SEM/EDX data, no insoluble aluminosilicate mineral such as zeolite A, sodalite or cancrinite minerals were confirmed by XRD.
- There is no evidence for the existence of insoluble crystalline mineral oxalate in the XRD results.

* OLI System Inc, Environmental Simulation Program (ESP), Version 9.0.14.

- The washing process mainly took out the sodium salts, elemental mercury, copper, nickel, uranium, silicon, and iron based on comparison of the pre-washed and washed Tank 104 solids; possibly because these elements are not part of the crystalline minerals identified in both the washed Tank 104 sample solids and the pre-wash Tank 104 solids.
- The XRD mineral phases identified in the Tank 101 solids include sodium nitrate or nitratine $Na(NO_3)$, sodium carbonate ($Na_2CO_3*H_2O$), bayerite ($-Al(OH)_3*Al(OH)_3$), trona ($Na_3H(CO_3)*H_2O$), and Sodium aluminum oxide hydroxide hydrate ($Na_2(Al_2O_3(OH_2)_2*5H_2O$), and sodium titanium oxide ($Na_2TiO_4(OH)_2$).
- Traces of the mineral bayerite ($-Al(OH)_3*Al(OH)_3$) or its polymorph (gibbsite) is the only crystalline mineral solids of aluminum identified in the Tank 101 solids.
- The semiquantitative average weight percent amounts of sodium (Na) in the three SWPF tank solids (Tank 101, Tank 104 pre-wash and Tank 104 washed sample) are 27.3 wt% (30.0 %RSD), 38.6 wt% (6.0 %RSD), and 9.3 wt% (18.3 %RSD), while the average weight percent titanium (Ti) for these same SWPF tank solids are 11.1 wt% (60.4 %RSD), 11.7 wt% (30.0 %RSD), and 32.0 wt% (23.4 %RSD), respectively.
- The relative amounts of aluminum (Al) in Tank 101, Tank 104 pre-wash and Tank 104 washed sample are 12.6 wt% (90.5 %RSD, 3.0 wt% (27.0 %RSD), and below instrument detection limits, respectively.
- The particle sizes of these Tank 101, Tank 104 pre-wash and Tank 104 washed solids are in the range of 1-4 microns, while Tank 101 re-sample and Tank 104 washed re-sample particle sizes ranged from 4 to 11 microns, and 0.5 to 2.5 microns, respectively.
- The XRD spectra for the Tank 104 washed re-sample and the original Tank 104 washed samples are essentially the same; both samples show the same predominant sodium-titanium mineral composition.
- The re-sampling and characterization of the Tank 101 solids and the Tank 104 washed sample solids confirm the initial mineralogy results for these two tank solids and did not produce any unexpected results.
- A total of seven XRD minerals were identified in the Tank 101 re-sample and five minerals were identified in the August 09, 2021 Tank 101 sample. Both samples had three minerals in common [trona (Na3H(CO3)*H2O, nitratine (NaNO3), and bayerite/ gibbsite (*a*-Al(OH)3*Al(OH)3].
- The solid sample major elemental components were like those of the initial Tank 101 solids and Tank 104 washed sample solids with slight differences in the semiquantitative weight percent elemental compositions.
- The Tank 101 sample solids contain mostly soluble sodium nitrate $Na(NO_3)$ mineral with relatively smaller amounts of other crystalline minerals present as well.
- The SEM/EDX semiquantitative data for particle size and weight percent elemental compositions for these SWPF solid samples, show significant uncertainties and variations in the results and so may not be reliable enough to account for elemental compositions and particle size changes in the SWPF tank samples.

The chemical make-up of the solids in the SWPF feed tank process chemistry is in line with the OLI model predictions, which indicate that relatively small amounts of soluble and insoluble solids are formed. Above all, there was nothing unexpected, in terms of mineral or solids content, found in the Tank 101, Tank 104 pre-wash, and Tank 104 washed solids samples.

Based on these characterization results for the three SWPF tank samples, the SWPF filtration process performance issues cannot wholly be attributed to the presence of these identified solids. There may be other technical reasons responsible for the poor performance of the SWPF filtration process. Apart from other SWPF operational parameters, it may be necessary to investigate the behavior of the accumulation of salt fines from the salt batches as well as the accumulation of small MST fines, which are normally present in the added MST bulk material.

To obtain more useful analytical results for the elemental compositions and particle size distribution for these SWPF tank solids, it is also recommended that tank solids be acid digested and directly characterized for elemental compositions by inductively coupled plasma atomic emission spectroscopy (ICP-AES) instead of relying on SEM/EDX semiquantitative results. A suspension of SWPF tank solids in a typical SWPF tank simulant solution and characterization for particle size distributions is also recommended.

5.0 Quality Assurance

The Task Technical and Quality Assurance Plan (TTQAP) details the planned activities and associated quality assurance implementing procedures for the characterization of the Analysis of Salt Waste Process Facility Tank Samples: Solvent Hold Tank ESS Tests and Tanks 101,102, 104 Solids Characterizations[7]. The documents referenced in the TTQAP include the following: L. N. Oji: ELN: L5575-00080-11 (Electronic Notebook (Production); SRNL, Aiken, SC 29808 (2014) and various ARD notebooks contain the analytical data. Other relevant QA documents include the Technical Task Request (TTR); see Attachment A.

The TTR (SLA-SRNS-00177 Item x; see attachment A) requested a production support (PS) classification; a Safety Significant Class does not apply to this work. Equipment with a General Service functional classification comprises the analytical measurement systems used to collect data for these characterizations. Standards used to calibrate these systems were purchased at level 2 with a certificate of analysis. Chemicals and reagents used in testing and sample preparation are purchased at levels 2 or 3 and standards are uniquely identified and traceable to NIST or equivalent per 1Q, 2-7 section 5.2.3.

To match the requested PS classification, the reports, calculations, and technical memoranda issued from this testing received technical review by design verification (E7 Manual Procedure 2.60, Section 5.2). This document, including all calculations, was reviewed by Design Verification by Document Review 8, 9. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. The experimental work, the analyses, and peer checks all comply with the customer quality assurance (QA) requirements.

6.0 References

(1) T. B. peters, "SWPF4 Short Term Results, LW-AD-PROJ-210624-2."

(2) L.N. Oji, "Determination of the Crystalline Structure of Scale Solids from the 16H Evaporator Gravity Drain Line to Tank 38H," SRNL-STI-2015-00457, Rev. 0, October 2015.

(3) T. B. peters," Analysis of Strip Effluent Hold Tank and Contactor Drain Tank Sample", SRNL-3100-2014-00088, Rev. 0, May 2014.

(4) M. S. Hay, "OLI Modelling of Strip Effluent Hold Tank and Contactor Drain Tank Solutions," SRNL-L3100-00092, May 2014.

(5) C. J. Martino, D. T. Herman, J. A. Pike and T. B. peters, "Actinide Removal Process Sample Analysis, Chemical Modelling, and Filtration Evaluation, SRNL-STI-2013, 00700, Rev. 0, June 2014.

(6) C. L. Crawford, "Actinide Removal Process October 2014 Sample Analysis," SRNL-STI-2014, 00609, Rev. 0, April 2014

(7) L. N. Oji, and T. B. Peters, "Task Technical and Quality Assurance Plan for the Analysis of Salt Waste Process Facility Tank Samples: Solvent Hold Tank ESS Tests and Tanks 101,102, 104 Solids Characterizations", SRNL-RP-2021-04764, Rev. 0, September 2021.

(8) "Technical Reviews", Manual E7, Procedure 2.60, Revision 18, December 2, 2019.

(9) "Savannah River National Laboratory Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Revision 2, August 2004.

Attachment A: SWPF Tank Sample Analysis-SLA-SRNS-00177-Item X

Analysis Scope:

SRNL to analyze SWPF Samples (4) upon delivery as described.

1) *Solvent Sample from the Solvent Hold Tank – SWPF will collect a sample from the Solvent Hold Tank and transfer the sample to SRNL. Then SRNL would complete an Extraction, Scrub, Strip (ESS) test on the solvent to validate solvent performance in the CSSX process. Analysis is similar to the ESS testing that SRNL has completed for SWPF previously to validate solvent performance. The testing would utilize radiocesium as the tracer.*

2) *TK-101 – SWPF will collect a sample and 'pre-concentrate' the solids in the SWPF hot cell (e.g., on centrifuge filter). Intent is the resultant solids would be low enough dose the samples could be contact handled. SRNL (Sam Fink Retired) indicated a sample of ~10 mg should be sufficient to do characterization and should be contact handled. The solids would be characterized by SRNL using SEM/XRD for both semi-quantitative size and composition.*

3) *TK-102 – SWPF will collect a sample of the concentrated retentate from the cross-flow filters (CFF). SWPF will decant the solid fraction from the bulk of the residual liquid portion of the with the intent that the resultant solids would be low enough dose that the samples could be contact handled (i.e., ~10mg solids). The solids would be characterized by SRNL using SEM/XRD for both semi-quantitative size and composition.*

4) *TK-104 – SWPF will collect a sample of the washed sludge. SWPF will decant the solid fraction from the bulk of the residual liquid portion of the with the intent that the resultant solids would be low enough dose that the samples could be contact handled (i.e., ~10mg solids). The solids would be characterized by SRNL using SEM/XRD for both semi-quantitative size and composition.*

SRNL to provide analytical service, data interpretation, Technical Report or Reports, and residues disposal and administrative documentation.

o Analysis Reports, one for Solvent Hold Tank Analysis and an Analysis Report for Tanks 101, 102 and 104.
  Reports may be combined if acceptable to customer (Parsons) schedule.

SRNL Estimate Assumptions:

- Samples are from SWPF process tanks.
- Sample collection and concentration by SWPF (Parsons).
- Sample transportation by Savannah River Remediation (SRR).
- The QA level for this work is at a production support (PS) level. However, Parsons will provide detailed statement of work to SRNL prior to initiating sampling to ensure the required analysis is understood.
- SRNL will provide a Task Technical Quality Assurance Plan (TTQAP) response document.
- Samples are radioactive but dose rates will be less than contact handing limits, but not high risk regulatory or also-known-as "pink dot" to SRNL (high Hg, F listed, etc.)
- Analytical sample residues will be managed and disposed by SRNL, unused sample will be returned to the liquid waste system through the HAD or as solid waste (Pending EEC (Environmental Evaluation Checklist) approval.

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