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Actinide Removal Process October 2014 Sample Analysis

C. L. Crawford

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EXECUTIVE SUMMARY

Filtration within the Actinide Removal Process (ARP) currently limits the throughput in interim salt processing at the Savannah River Site. In this process, batches of salt solution with Monosodium Titanate (MST) sorbent are concentrated by crossflow filtration. The filtrate is subsequently processed to remove cesium in the Modular Caustic Side Solvent Extraction Unit (MCU) followed by disposal in saltstone grout. The concentrated MST slurry is washed and sent to the Defense Waste Processing Facility (DWPF) for vitrification.

During past ARP processing, there has been a degradation of filter performance manifested as the inability to maintain high filtrate flux throughout a multi-batch cycles during Salt Batches 6, 5 and 4. The objectives of the current effort for Salt Batch 7 samples, which are similar to the most recent ARP sample analysis from 2013, were to characterize the feed streams, to determine if solids (in addition to MST) are precipitating and causing the degraded performance of the filters, and to assess the particle size and rheological data to address potential filtration impacts. The samples from the LWPT and LWHT were obtained from several stages of processing of Salt Batch 7B, Cycle 1, Batch 67.

The following are the key results for sample analysis from the ARP process tanks collected over a five day sequence in this study involving 1) the first and second LWPT samples before and after initiation of batch washing, respectively; 2) the LWHT sample obtained upon completion of batch washing and after initiation of chemical cleaning; and 3) the third and fourth LWPT samples collected after filtration.

- At the time of sampling, the first LWPT slurry in the ARP 512-S was 5.5 M Na^+, 1.9 M OH^- and 2.3 M NO_3^- salt solution with 2.09 wt% of insoluble solids. Based on major analyzed species (Al, Na^+, OH^-, NO_3^-, NO_2^-, CO_3^{2-} and SO_4^{2-}) subsequent samples relative to the first LWPT sample were found to be 10.5-times, 2.3-times, 12.3-times and 1.2-times dilute, respectively, for the second LWPT, the LWHT and the third and fourth LWPT samples.

- The oxalate concentration of the first LWPT sample is very similar to the expected oxalate concentration from blend evaluation calculations for SB7b, whereas in previous analysis the LWPT-1 oxalate concentration was ~ 2.7-times lower than the analyzed Tank 49H oxalate concentration.

- Higher sodium concentrations are observed between the previous set of samples for Tank 49H and LWPT-1, with Na at 6.03±0.03M and 6.57±0.15 M, respectively, and the current initial LWPT sample (Na = 5.52±0.00 M) and calculated blend (Na = 6.22 M).

- For both the third and fourth LWPT samples the oxalate concentrations are greater than expected based on the dilution factors of other major components. This is expected since oxalate is introduced during oxalic acid chemical cleaning (before the third and fourth LWPT samples were collected).

- Comparison of the filtrate anion species with those determined from water-weighted dilutions of the slurry indicate no significant solid species/salts dissolve upon dilution with water.

- The second and third LWPT samples are similar based on major filtrate components, dissolved slurry analysis and measured solids, to the PRFT samples reported in earlier studies from the DWPF.

- The insoluble solids contain primarily titanium and are consistent with the MST added during processing. Based on slurry analysis for titanium, the first through fourth LWPT samples have 2.09 wt%, 0.55 wt%, 0.23 wt% and 0.04 wt% MST, respectively. These MST values for the two highest MST containing samples fall within the range encompassed by separate insoluble solids measurements.

- A very small quantity of sodium aluminosilicate in the range of 0.12 wt% to 1.45 wt% of washed and dried solids may also be present in the insoluble solids, based on the detection of both Al and Si present in the solids isolated from all LWPT samples. For instance the total NAS solids in the third LWPT would be only 5.4E-3 wt% insoluble NAS solids on a slurry basis (1.45 wt% of 0.37 wt% total insoluble solids).

- The primary species identified in all the washed and dried solids by XRD is the MST. No evidence of crystalline sodium aluminosilicate species or aluminum containing gibbsite was observed in the XRD.

- Sodium oxalate (natroxalate) was identified in the fourth LWPT solid and was not detectable in the other LWPT solids. Since the fourth LWPT sample contains new material transferred in from 241-96H, this suggest that the sodium oxalate is present at the start of processing but is removed after processing 67 batches.

- Based on the quantity of iron in the solids from the first two LWPT samples, High-Level Waste sludge also appears to be present as a minor fraction of the solids in the ARP slurries, with sludge present at roughly 1/38th the mass of MST present.

- The ARP 512-S slurries exhibited Newtonian flow behavior, with the higher concentration slurries (first and fourth LWPT samples) having a viscosity of 2.9 to 3.2 cP and the LWHT solution at ~ 2X lower concentration having a viscosity of 1.7 cP at 25 ºC. The second and third LWPT slurries below 1M Na^+ showed viscosities in the range of 0.7 to 1.1 cP. Flow behavior observed for the samples would not appear to challenge the filtration system outside of the expected salt feeds.

- Particle size distribution analysis of the four LWPT slurries shows that these solids are very similar to the starting MST slurries that contain a bi-modal distributions centered around a lower 1.4 micron range and a higher 6.5 micron range.

- Microscopy data obtained for the washed solids shows that they are primarily MST of similar particle size with that expected from the PSD data. Some minor 50 to 100 micron sized fragments enriched in either Al, Fe or Zn/Cl were identified in the first LWPT solids.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CI	Confidence Interval
CVAA	Cold Vapor Atomic Absorption
DS	Dissolved Solids
DWPF	Defense Waste Processing Facility
HF	Hydrofluoric acid
HLW	High-Level Waste
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
LPPP	Low Point Pump Pit
IS	Insoluble Solids
LWHT	Late Wash Hold Tank (in ARP 512-S) LWPT Late Wash Pump Tank (in ARP 512-S)
MCU	Modular Caustic Side Solvent Extraction Unit
MST	Monosodium Titanate
n	Number of replicate measurements
N/A	Not applicable
NAS	Sodium Aluminosilicate
PRFT	Precipitate Reactor Feed Tank
SB	Salt Batch
SEM	Scanning Electron Microscopy
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TS	Total Solids
TTQAP	Task Technical and Quality Assurance Plan
TTR	Task Technical Request
XRD	X-ray Diffraction

1.0 Introduction

1.1 Process Background

Savannah River Remediation (SRR) operates a set of closely coupled facilities that process salt waste, removing strontium, actinides, and cesium prior to disposal of decontaminated salt solution in saltstone. One facility that limits operating rates due to processing difficulties is the filtration portion of the Actinide Removal Process (ARP), located in 512-S. A review team including Savannah River National Laboratory (SRNL) personnel documented observations and recommendations for improved operation, increased throughput, and greater reliability of the ARP in 2013.[1] ARP process samples were subsequently characterized at SRNL to improve understanding of the process chemistry and physical properties of the material being filtered.[2] Process descriptions of the ARP process shown in the simplified process diagram of Figure 1-1 are given in the 2013 SRNL study.[2]

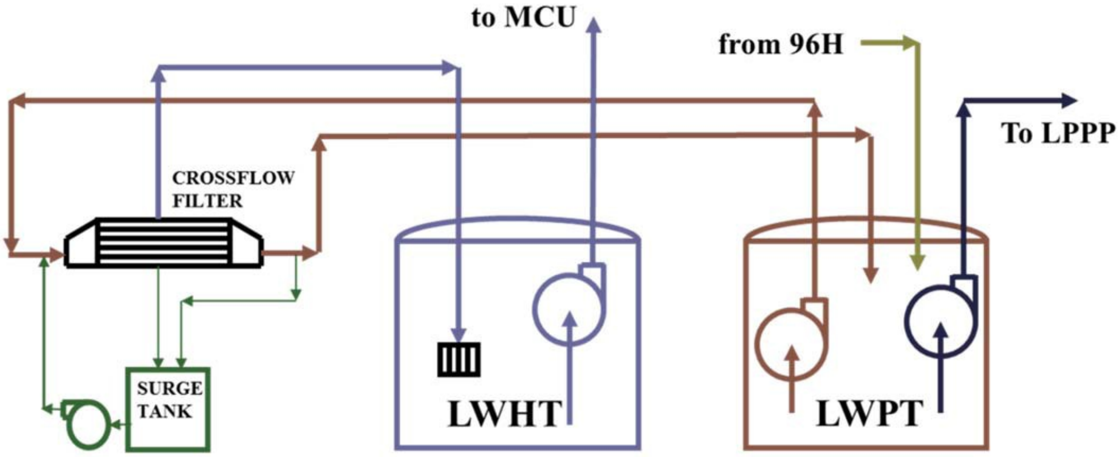


Figure 1-1. Simplified diagram of the 512-S portion of ARP

1.2 Task Objectives

Current sample analyses are a continuation effort involving a series of samples that were taken from the Late Wash Precipitate Tank (LWPT) and Late Wash Hold Tank (LWHT) after completion of cycle 1 of Salt Batch (SB) 7B in early October 2014 to improve understanding of the process chemistry and physical properties of the material being filtered.

The goals of this effort for SB 7B samples, as similarly specified in the 2013 analyses of SB 6D samples, are to characterize the feed streams, to determine if solids (in addition to MST) are precipitating and to apply the particle size and rheological data to confirm the applicability of the filtration process. The current analyses involve an additional LWPT sample (relative to those sampled in 2013) that was taken following the addition of feed to the residual heel of the cleaning solution.

Task activities include extensive physical and chemical analysis of the LWPT and LWHT samples, followed by completion of evaluations utilizing the data collected from the samples. SRR DWPF

Engineering issued a Task Technical Requests (TTR) for this task.[3] A Task Technical and Quality Assurance Plan (TTQAP) contains the work scope for ARP sample characterization.[4]

1.3 Samples

SRR collected five sets of samples from the ARP 512-S facility tanks and sent them to SRNL for analysis. Samples were collected after completion of processing for SB 7B, Cycle 1, Batch 67. Table 1-1 describes the naming convention of the samples, the stage of processing at which the samples were collected, and lists the tank volume and temperature at the time of sampling. Vessels were under continuous agitation just prior to sampling. Four of the sets were from the LWPT and were slurries containing MST and potentially other undissolved solids. The fifth set was from the LWHT and did not contain visible indication of undissolved solids. The volume of each set of samples was nominally 200 mL in total (i.e., two individual 200 mL samples). The first LWPT sample set arrived at SRNL on October 1, 2014 and the second LWPT sample set arrived at SRNL on October 2, 2014. The LWHT sample set and the final two (third and fourth LWPT) sample sets arrived at SRNL on October 6 and 7, 2014. All samples were placed into B-Block of the Shielded Cells on October 8, 2014.

Upon receipt by SRNL, ARP samples from a set (two individual 200 mL samples) were combined into a single bottle for each set. The cell temperature as ARP samples were unpackaged was at ambient temperature, nominally 23 +/- 2 °C. The pH as determined by pH test strips was roughly 14 for all samples. Figure 1-2 contains photographs of the individual samples within each sample set. While visual differences were noted between the different sample sets, no visual differences were noted between samples within each sample set. All of the LWPT samples were turbid with nearly white solids, consistent with the presence of MST as the major solid phase. The whiteness of the solids indicates that the LWPT samples contain no more than a minor fraction of HLW sludge, which tends to contain dark brown or black solids. The LWHT samples were clear and thus did not have visual indication of insoluble solids. The same camera settings and lighting conditions were used for the photographs of all four sample sets. Sample photographs taken through the Cell 12 window exhibit an overall haze due to the cell window and splash guard.

Table 1-1. ARP 512-S sample set description

Process Step	Samples Obtained	Tank Conditions
Samples collected from the LWPT after completing cycle 1 of Salt Batch 7B, but before initiation of batch washing cycle.	ARP-14-012 = bottle cap #1 ARP-14-013 = bottle cap #2 (First set of LWPT samples)	~1600 gal 25 °C
Samples collected from the LWPT after initiation and completion of batch washing cycle (Procedure SW4-15.116-2.4, Rev.14, 11/4/14).	ARP-14-014 = bottle cap #3 ARP-14-015 = bottle cap #4 (Second set of LWPT samples)	~1700 gal 25 °C
Samples collected from the LWHT after completion of batch washing cycle and initiation of chemical cleaning.	ARP-14-016 = bottle cap #5 ARP-14-017 = bottle cap #6 (LWHT samples)	~1300 gal 25 °C
Samples collected from the LWPT after chemical cleaning and addition of 90 gallons of 50 wt% caustic to the LWHT for heel adjustment.	ARP-14-018 = bottle cap #7 ARP-14-019 = bottle cap #8 (Third set of LWPT samples)	~1800 gal 27 °C
Samples collected from the LWPT after 1) transfer of neutralized chemical cleaning solution to LPPP-PPT; 2) addition of 65 gallons of 50 wt% caustic and 650 gallons of dilute caustic; 3) drain of any heel in backpulse tank and/or crossflow filter to surge tank and 4) receipt of next batch from 241-96H of SB 7B.	ARP-14-020 = bottle cap #9 ARP-14-021 = bottle cap #10 (Fourth set of LWPT samples)	~6000 gal 31 °C

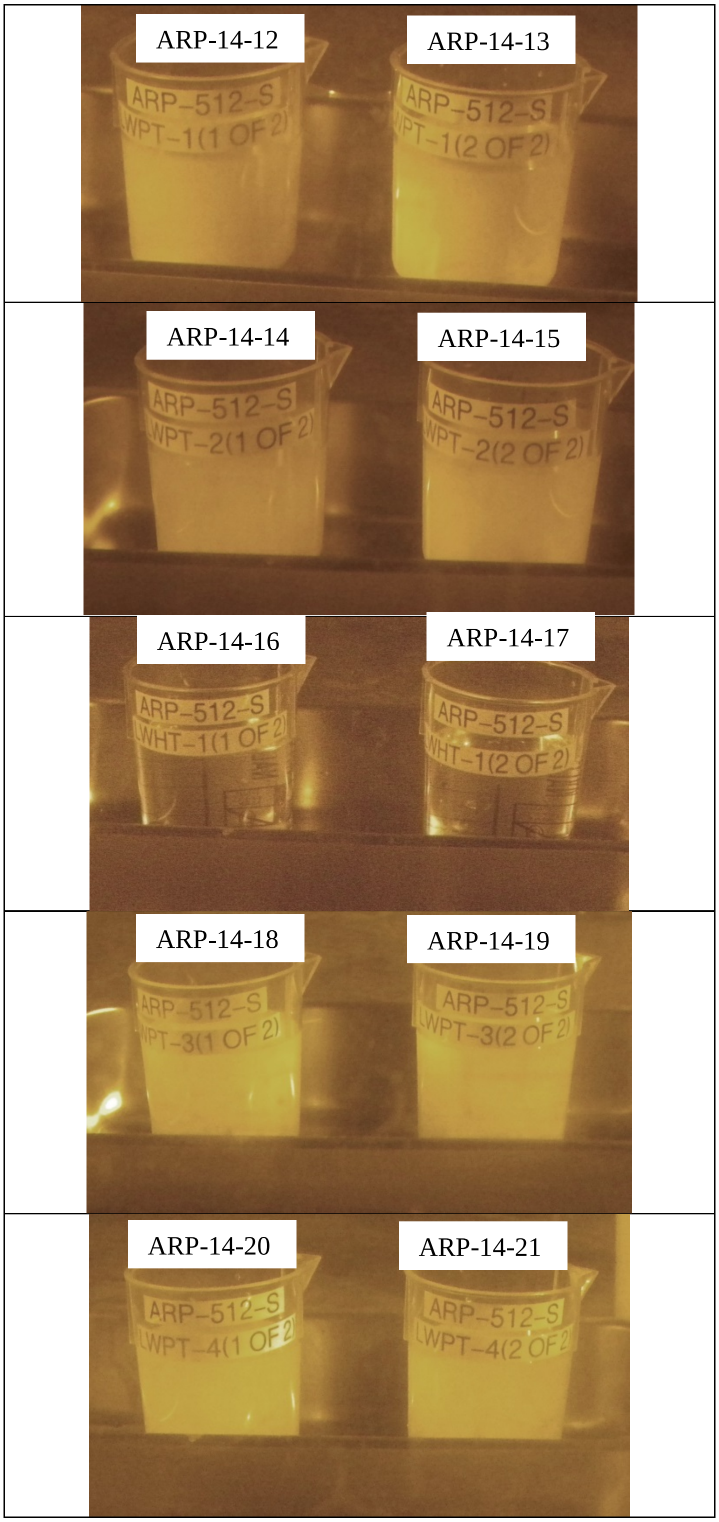


Figure 1-2. APR 512-S samples photographed in SRNL Shielded Cells B-Block Cell 12

2.0 Experimental Procedure

2.1 512-S Sample Analysis

The samples were visually observed and photographed in the SRNL shielded cells.

For the mixed samples (slurries), the following measurements and analyses were performed: pH, solids content (Total Solids (TS), Dissolved (soluble) Solids (DS) and Insoluble Solids (IS)), density, rheology, Particle Size Distribution (PSD) analysis, Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES) for metals, including axial detection for sulfur; Ion Chromatography (IC) for anions; and Total Inorganic Carbon/Total Organic Carbon (TIC/TOC).

For filtrate obtained from the slurries, the following measurements and analyses were performed: ICP-ES for metals, including axial detection for sulfur; IC for anions; TIC/TOC; and Free Hydroxide.

For solids isolated from the slurries, the following measurements and analyses were performed: X-ray Diffraction (XRD), ICP-ES for metals, including axial detection for sulfur, Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS) for certain metals (Pb, Sb and Sn); and Cold Vapor Atomic Absorption (CVAA) for mercury. Scanning Electron Microscopy (SEM) was also performed on the first two samples, LWPT-1 and LWPT-2. The solids in the latter samples LWPT-3 and LWPT-4 were consumed in the above analyses and thus were not analyzed by SEM.

Material for filtrate analysis was obtained from LWPT samples via filtration of the slurries through cup filters with 0.45 µm nylon membranes. These filtrates along with the as-received LWHT solution were submitted directly without any preparations for ICP-ES, IC anions and wet chemical methods.

Preparation of slurries for analyses that require dissolution (ICP-ES) was performed by a mixed acid dissolution method. This method uses a combination of 7 mL of nitric acid and 1.5 mL of hydrofluoric (HF) acid for heated digestion in sealed Teflon® vessels and dilute 0.6 M boric acid for dilution. A nominal 4 g slurry sample was dissolved in a total of 50 mL for these studies. Boric acid is used in these dissolutions to complex any excess HF acid.

Preparation of slurries for analysis by IC for anions and TIC/TOC was accomplished by a 50-times weighted dilution with water. The liquid phase was analyzed by IC and TIC/TOC and would include anions that dissolved during the contact of the slurry with water.

Solids were isolated from the slurries by filtering through 0.45 µm nylon filters and allowing additional air to pass through the filter cake to minimize interstitial liquid inclusion. The solids were then each washed in an equivalent manner with ~ 25 mL of 190 proof (~95%) ethanol in an attempt to remove any remaining interstitial liquid and/or soluble solids. Previous ARP analysis studies did not attempt to wash the solids separated from the LWPT samples.[2] The ethanol was delivered to the solids of the filter cake using a large plastic disposable pipette. The washed solids were air dried in the cells. Ethanol was used instead of water since both sodium carbonate and sodium oxalate are reported to be insoluble in alcohol.[5] Thus if any insoluble sodium carbonate or sodium oxalate solids are present in the separated solids, they would presumably not be washed out as with a solvent in which they are soluble such as water. For instance, a previous study on Precipitate Reactor Feed Tank (PRFT) MST solids showed that water washing of filtered solids rendered only MST with no crystalline solids present as analyzed by XRD.[6]

Prior to the chemical analysis of solids, the material was dried for approximately 12 hours at 120 °C so that the results would be on a dry unwashed solids basis. Preparation of isolated solids for analyses that require dissolution (ICP-ES, ICP-MS, CVAA Hg) was performed by a mixed acid dissolution method in sealed Teflon® vessels. This method uses the same combination (amount and ratio) of nitric and hydrofluoric acids for heated digestion and boric acid for dilution as described above for the acid-dissolved slurries. A nominal 0.2 g solid sample was dissolved in a total of 100 mL for these studies.

Particle size analysis was performed on a Microtrac 100 using scattering mode. LWPT slurry materials of ~ 10 mL were analyzed in 250 mL nonradioactive simulants of sample supernate (based on the results in Section 3.1) that were made from laboratory grade chemicals and filtered with a 0.2 µm nylon filter. The simulated supernate for LWPT-1 and LWPT-4 was 1.9 M NaOH, 2.6 M $NaNO_3$, and 0.66 M $NaNO_2$. The simulated supernate for LWPT-2 was a 10X dilution of LWPT-1 and the simulated supernate for LWPT-3 was a 12X dilution of LWPT-1. Samples were agitated vigorously prior to analysis but samples were not sonicated. Results were obtained for both number and volume distributions. The minimum particle size that the instrument can quantify is 0.7 µm.

The rheological properties of the samples were determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. The shear stress is determined from the torque measurement and is independent of the rheological properties. Based on the expected particle size of the materials in this investigation, the MV 1 rotor was considered most appropriate. Additional details on this instrument and the measurement technique, including calibration, is reported elsewhere.[7]

2.2 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Data obtained during this study are recorded and maintained in SRNL E-Notebook (Production), Savannah River National Laboratory: C. L. Crawford, "ARP 512-S Sample Analysis October 2014", B9108-00026-17.

3.0 Results and Discussion

3.1 Chemical Analysis

Chemical analysis of the various preparations of the LWPT and LWHT samples are reported in Table 3-1 through Table 3-5. Table 3-1 and Table 3-3 contain the analytical results for the portions of the LWPT samples passed through a 0.45 µm nylon filter and the relatively solids free LWHT sample without filtration. Results are reported on a liquid basis. Table 3-2 contains analysis of the LWPT samples including suspended solids, and an additional analysis of the LWHT sample without filtration. Table 3-4 shows the anion results for weighted dilutions of the as-received LWPT slurries. Results are reported on a wet slurry volume basis. Table 3-5 contains the analysis of the solids that were isolated from the slurry by filtration, washed with ethanol and dried. Thus these results are on a dried solids mass basis.

The average values and the relative standard deviations (RSD) are reported where replicate preparations and measurements were performed. Average values are preceded by "<" when the analyte is below the limits of quantification. The RSD values are not reported when all measurements are below the limits of quantification. Average values are preceded by "<="

(meaning “less than or equal to”) when values above the limits of quantification are combined with other values taken at the limits of quantification.

Table 3-1 shows that only limited components (Al, Cr, K, Na and S) were detectable in all the filtered LWPT slurries and the LWHT sample. Most of the elements were determined to be less than the ICP-ES method quantification limits for all five samples as noted at the bottom of Table 3-1. The soluble titanium in the first LWPT sample of 22.6 mg/L decreased to less than detectable amounts in the second LWPT and the third LWPT and was just above detection at 1.6 mg/L in the LWHT sample. Soluble titanium was 5.1 mg/L in the final LWPT filtrate. Similar analysis of filtrates from caustic MST-containing slurries also have shown relatively low amounts of soluble Ti in the range of 7.1 mg/L to 23.8 mg/L in filtered and acid diluted ARP samples.[2]

Data in Tables 3-1 and 3-3 for the higher concentration components including Al, Na, OH^-, NO_3^-, NO_2^-, CO_3^{2-} and SO_4^{2-} can be used to calculate the change in these sample solution concentrations through the processing steps. The second LWPT sample is 10.5-times dilute as the first LWPT sample and the LWHT solution is 2.3-times dilute as the first LWPT sample. These results are expected since the second LWPT sample derives from the so-called ‘feed and bleed’ processing that reduces the sodium molarity of the LWPT to ~ 0.5 M, and the LWHT sample derives from accumulated material during the batch washing cycle. The third LWPT sample is somewhat similar to the second LWPT sample as it is about 12.3-times dilute as the first LWPT sample. The final fourth LWPT sample is similar to the initial LWPT sample as it is only 1.2-times dilute as the first LWPT sample. The Na and hydroxide were not used in these latter calculations since they were added in various amounts as 50 wt% caustic and dilute caustic preceding collection of the third and fourth LWPT samples.

The oxalate concentration is less than detection for the second LWPT sample which is expected from the calculated dilution factor of 10.5X and the IC anion method detection limit of 100 mg/L (1.1E-3M) for oxalate anion. The oxalate concentration for the LWHT sample is as expected from comparison with all the other dilution factors from the major components discussed above. For both the third and fourth LWPT samples the oxalate concentrations are greater than expected based on the dilution factors of other major components. Oxalate is introduced during oxalic acid chemical cleaning (before the third LWPT samples were collected). The oxalate concentration in the LWHT solution of 1.56E-03 M is about 8X higher than recently projected (1.9E-04 M) from modeling calculations[8] involving proposed flushing steps after washing. However, the proposed flushing steps associated with those preliminary evaluations were not performed for this specific cleaning evolution.

Some limited comparisons can be made between the current filtrate data and similar data obtained from the 512-S samples in 2013.[2] Comparison of the Al, Na, NO_3^- and OH^- concentrations from current first LWPT filtrate to the previous LWPT-1 filtrate indicates that all of these major species are lower in the current sample, while the current oxalate concentration is ~ 3.6X higher. The lower Na concentration (1.27×10^5 mg/L, or 5.52±0.00 M vs. 1.51×10^5 mg/L, or 6.57±0.15 M) is likely responsible for the observed higher oxalate concentration since oxalate solubility is a strong function of sodium concentration. Another comparison can be made between the current and previous results for carbonate concentrations in the LWPT samples after chemical cleaning of the filter. The 2013 LWPT-3 sample (after filter cleaning and heel adjustment) indicated an increased level of carbonate vs. that expected from dilution factors of other major species. However for the current LWPT samples obtained after filter cleaning (the third and fourth LWPT samples) the observed carbonate concentrations are in good agreement with that expected from the dilution factor of the other major components.

Comparisons can also be made between the analyzed salt batch feed and the initial LWPT filtrates for the 2013 samples (Tank 49H vs. LWPT-1)[2] and the current samples (Blend Evaluation for Tank 49H[9] vs. first LWPT). As was detailed in the previous work comparison of the major filtrate species (Al, Na, NO_2^-, NO_3^-, SO_4^{2-}, CO_3^{2-} and OH^-) indicates good agreement between the Tank 49H major ions and the LWPT-1, with an average feed:LWPT ratio of 1.05 ± 0.08. One exception was noted as the oxalate concentration in the LWPT-1 sample was observed to be lower (1.11E-03 M) than the Tank 49H feed value of 2.95E-03 M. Similar comparisons of the same species (Al, Na, NO_2^-, NO_3^-, SO_4^{2-}, CO_3^{2-} and OH^-) also indicates good agreement between the Blend Evaluation for Tank 49H major ions and the first LWPT, with an average feed:LWPT ratio of 1.06 ± 0.10. The oxalate concentrations were also similar within experimental uncertainty in the current LWPT analyses vs. the Blend Evaluation Tank 49H in the range of 4.00E-03 to 4.08E-03 M. The range of sodium molar concentrations in the 2013 samples (Tank 49H = 6.03 ± 0.10 M and LWPT-1 = 6.57 ± 0.15 M) were higher than the current Blend Evaluation for Tank 49H (6.22 M) and the initial LWPT = 5.52 M.

Table 3-2 and Table 3-4 contain the analysis of the as-received ARP samples (including solids when present). Because LWHT-1 was relatively free of solids and was already analyzed without filtration (see Table 3-1 and Table 3-3), only a single replicate was processed for LWHT-1 with the slurry analysis preparation. Slurry results, which include the dissolution of solids, are reported on a volume basis for easier comparison with the filtrate results. As noted in Table 3-2, comparable levels of Sb and Sn were observed in the dissolution blank as were detected in the samples, so the detection limits for the water blank are reported for all samples for these two elements. All Si values shown in Table 3-2 are blank corrected due to presence of detectable Si levels in the dissolution blank, which ranged from 70% to 100% of the Si measured in the dissolved samples. Spurious levels of Si can arise from concentrated HF acid used in these dissolutions, interacting with the glass and quartz components in the ICP-ES analysis instrumentation. A trace detectable level of TIC was also detected in the water blank of the weighted dilutions so the carbonate values in Table 3-4 are blank corrected.

Hydroxide was not analyzed on the slurry preparations, so the filtrate data should be used for slurry free hydroxide. From the comparison of the sodium and anion results between the slurry and the filtrate, good agreement was seen. This indicates that the majority of the analytes are primarily present in the soluble phase and there is little or no contribution from the solids for most analytes. The main detectable components for all samples from filtrate analysis from Table 3-1 (Al, Cr, K, Na and S) are also observed as the main components of the dissolved slurry shown in Table 3-2. The first LWPT sample indicates 127 mg/L of Ca and the other samples show trace levels of detectable Ca up to 28 mg/L for the as-received slurries vs. the filtrate data that indicates no detectable Ca present.

As expected, the primary difference between the slurry results and the filtrate results is the much higher titanium content in the slurry due to the inclusion of MST. The average titanium concentrations in the first through fourth LWPT samples are 13,400 mg/L, 2,780 mg/L, 1,150 mg/L and 260 mg/L, respectively. Making an assumption that all of the titanium is in the form of MST with empirical formula $NaHTi_2O_5$, and using the measured densities reported later in Table 3-6, the first through fourth LWPT samples have 2.09 wt%, 0.55 wt%, 0.23 wt% and 0.04 wt% MST, respectively.

The anion results in the as-received slurry shown in Table 3-4 when compared to those of the filtrate shown in Table 3-3 do not indicate any significant increases suggesting that no appreciable salt components are contained in the insoluble solids for the LWPT samples.

Table 3-1. Elemental analysis of filtered LWPT samples and as-received LWHT sample*

element (mg/L)	ARP-14-012/013 (First LWPT)		ARP-14-014/015 (Second LWPT)		ARP-14-016/017 (LWHT)		ARP-14-018/019 (Third LWPT)		ARP-14-020/021 (Fourth LWPT)	
	average	RSD	average	RSD	Average	RSD	average	RSD	average	RSD
Ag	*<1.94E+00*	--	*<1.94E+00*	--	*<1.94E+00*	--	*<1.94E+00*	--	*<1.94E+00*	--
Al	3.51E+03	0.6%	3.19E+02	2.2%	1.70E+03	0.4%	2.62E+02	0.5%	2.72E+03	0.0%
B	5.24E+01	0.3%	*<2.17E+01*	--	2.47E+01	2.9%	*<2.17E+01*	--	4.08E+01	2.3%
Ba	*<1.00E+00*	--	*<1.00E+00*	--	*<1.00E+00*	--	*<1.00E+00*	--	*<1.00E+00*	--
Be	*<1.40E-01*	--	*<1.40E-01*	--	*<1.40E-01*	--	*<1.40E-01*	--	*<1.40E-01*	--
Ca	*<7.20E-01*	--	*<7.20E-01*	--	*<7.20E-01*	--	*<7.20E-01*	--	*<7.20E-01*	--
Cd	*<1.27E+00*	--	*<1.27E+00*	--	*<1.27E+00*	--	*<1.27E+00*	--	*<1.27E+00*	--
Ce	*<1.12E+01*	--	*<1.12E+01*	--	*<1.12E+01*	--	*<1.12E+01*	--	*<1.12E+01*	--
Co	*<1.75E+00*	--	*<1.75E+00*	--	*<1.75E+00*	--	*<1.75E+00*	--	*<1.75E+00*	--
Cr	3.74E+01	0.6%	3.51E+00	1.6%	1.83E+01	0.0%	3.14E+00	6.1%	2.95E+01	0.5%
Cu	*<3.54E+00*	--	*<3.54E+00*	--	*<3.54E+00*	--	*<3.54E+00*	--	*<3.54E+00*	--
Fe	*<1.39E+00*	--	*<1.39E+00*	--	*<1.39E+00*	--	*<1.39E+00*	--	*<1.39E+00*	--
Gd	*<4.44E+00*	--	*<4.44E+00*	--	*<4.44E+00*	--	*<4.44E+00*	--	*<4.44E+00*	--
K	5.07E+02	9.5%	6.56E+01	2.8%	1.66E+02	9.4%	5.57E+01	4.4%	4.26E+02	10.8%
La	*<1.97E+00*	--	*<1.97E+00*	--	*<1.97E+00*	--	*<1.97E+00*	--	*<1.97E+00*	--
Li	1.68E+01	3.4%	<1.27E+01	--	*<1.27E+01*	--	*<1.27E+01*	--	*1.41E+01*	0.5%
Mg	*<8.61E+00*	--	*<8.61E+00*	--	*<8.61E+00*	--	*<8.61E+00*	--	*<8.61E+00*	--
Mn	*<8.00E-01*	--	*<8.00E-01*	--	*<8.00E-01*	--	*<8.00E-01*	--	*<8.00E-01*	--
Mo	*<1.79E+01*	--	*<1.79E+01*	--	*<1.79E+01*	--	*<1.79E+01*	--	*<1.79E+01*	--
Na	1.27E+05	0.0%	1.24E+04	1.1%	6.11E+04	0.2%	1.81E+04	0.4%	1.12E+05	0.6%
Ni	*<4.55E+00*	--	*<4.55E+00*	--	*<4.55E+00*	--	*<4.55E+00*	--	*<4.55E+00*	--
P	1.94E+02	0.4%	*<4.73E+01*	--	9.50E+01	1.9%	*<4.73E+01*	--	1.53E+02	1.4%
Pb	*<1.30E+02*	--	*<1.30E+02*	--	*<1.30E+02*	--	*<1.30E+02*	--	*<1.30E+02*	--
S	3.32E+03	0.6%	2.80E+02	5.3%	1.57E+03	0.5%	2.64E+02	2.7%	2.65E+03	0.5%
Sb	*<4.11E+01*	--	*<4.11E+01*	--	*<4.11E+01*	--	*<4.11E+01*	--	*<4.11E+01*	--
Si	*5.60E+01*	0.1%	*<1.70E+01*	--	2.74E+01	0.8%	*<1.70E+01*	--	4.33E+01	1.1%
Sn	*<9.31E+01*	--	*<9.31E+01*	--	*<9.31E+01*	--	*<9.31E+01*	--	*<9.31E+01*	--
Sr	*<1.28E+01*	--	*<1.28E+01*	--	*<1.28E+01*	--	*<1.28E+01*	--	*<1.28E+01*	--
Th	*<1.16E+01*	--	*<1.16E+01*	--	*<1.16E+01*	--	*<1.16E+01*	--	*<1.16E+01*	--
Ti	2.26E+01	0.3%	*<9.30E-01*	--	1.58E+00	1.3%	*<9.30E-01*	--	5.08E+00	0.1%
U	*<6.99E+01*	--	*<6.99E+01*	--	*<6.99E+01*	--	*<6.99E+01*	--	*<6.99E+01*	--
V	*<6.90E-01*	--	*<6.90E-01*	--	*<6.90E-01*	--	*<6.90E-01*	--	*<6.90E-01*	--
Zn	3.28E+00	3.7%	*<1.06E+00*	--	*<1.06E+00*	--	*<1.06E+00*	--	2.86E+00	1.5%
Zr	*<6.20E-01*	--	*<6.20E-01*	--	*<6.20E-01*	--	*<6.20E-01*	--	*<6.20E-01*	--

*The following elements were not detected above the method quantification limits for all samples: Ag, Ba, Be, Ca, Cd, Ce, Co, Cu, Fe, Gd, La, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Th, U, V and Zr.

Table 3-2. Elemental analysis of as-received samples from acid dissolution of LWPT slurries and LWHT solution

(mg/L)	ARP-14-012/013 (First LWPT)		ARP-14-014/015 (Second LWPT)		ARP-14-016/017 (LWHT)	ARP-14-018/019 (Third LWPT)		ARP-14-020/021 (Fourth LWPT)	
	average	RSD	average	RSD		average	RSD	average	RSD
Ag	*<3.07E+00*	--	*<2.39E+00*	--	*<2.58E+00*	*<2.27E+00*	--	*<2.66E+00*	--
Al	3.70E+03	0.2%	3.32E+02	0.9%	1.66E+03	2.72E+02	1.1%	2.85E+03	0.3%
Ba	9.56E+00	0.9%	1.99E+00	4.1%	*<1.33E+00*	*<1.17E+00*	--	*<1.37E+00*	--
Be	4.83E+00	0.4%	9.33E-01	0.1%	*<1.87E-01*	*3.86E-01*	1.9%	*<1.92E-01*	--
Ca	1.27E+02	0.5%	2.82E+01	3.2%	1.14E+00	1.34E+01	0.6%	3.81E+00	0.5%
Cd	5.53E+01	0.0%	1.13E+01	2.0%	*<1.70E+00*	4.66E+00	0.2%	*<1.74E+00*	--
Ce	2.29E+01	6.4%	*<1.38E+01*	--	*<1.49E+01*	*<1.31E+01*	--	*<1.54E+01*	--
Co	1.87E+01	1.0%	6.49E+00	1.4%	*<2.33E+00*	2.66E+00	2.8%	*<2.40E+00*	--
Cr	3.95E+01	0.5%	4.01E+00	0.4%	1.79E+01	4.29E+00	2.8%	3.05E+01	0.9%
Cu	*<5.60E+00*	--	*<4.35E+00*	--	*<4.72E+00*	*<4.14E+00*	--	*<4.86E+00*	--
Fe	1.38E+02	0.7%	3.38E+01	2.4%	6.33E+00	1.58E+01	8.1%	*<1.91E+00*	--
Gd	*<7.02E+00*	--	*<5.46E+00*	--	*<5.92E+00*	*<5.19E+00*	--	*<6.10E+00*	--
K	4.94E+02	2.4%	*<=4.85E+01*	--	2.23E+02	5.51E+01	4.1%	4.66E+02	1.9%
La	*<3.11E+00*	--	*<2.42E+00*	--	*<2.63E+00*	*<2.31E+00*	--	*<2.71E+00*	--
Li	9.29E+01	4.7%	*<1.56E+01*	--	*<1.70E+01*	*<1.48E+01*	--	*<1.74E+01*	--
Mg	*<1.36E+01*	--	*<1.06E+01*	--	*<1.15E+01*	*<1.01E+01*	--	*<1.18E+01*	--
Mn	*<1.26E+00*	--	*<9.84E-01*	--	*<1.07E+00*	*<9.35E-01*	--	*<1.10E+00*	--
Mo	*<2.84E+01*	--	*<2.21E+01*	--	*<2.39E+01*	*<2.10E+01*	--	*<2.47E+01*	--
Na	1.36E+05	0.7%	1.33E+04	0.0%	6.41E+04	1.89E+04	0.0%	1.17E+05	0.1%
Ni	*<7.19E+00*	--	*<5.60E+00*	--	*<6.06E+00*	*<5.32E+00*	--	*<6.25E+00*	--
P	3.34E+02	3.0%	*<5.82E+01*	--	*8.15E+01*	*<5.53E+01*	--	1.41E+02	1.2%
Pb	1.99E+03	2.8%	*<1.60E+02*	--	*<1.73E+02*	*<1.52E+02*	--	*<1.79E+02*	--
S	3.26E+03	0.8%	2.93E+02	0.8%	1.46E+03	2.69E+02	1.4%	2.58E+03	1.0%
Sb*	*<5.36E+01*	--	*<5.36E+01*	--	*<5.36E+01*	*<5.36E+01*	--	*<5.36E+01*	--
Si**	5.34E+01	3.4%	5.55E+00	--	1.74E+01	*<9.47E+01*	--	2.93E+01	32.7%
Sn*	*<3.97E+02*	--	*<3.97E+02*	--	*<3.97E+02*	*<3.97E+02*	--	*<3.97E+02*	--
Sr	2.78E+00	0.0%	5.37E-01	0.1%	*<1.60E-01*	2.17E-01	1.4%	*<1.65E-01*	--
Th	1.47E+01	5.0%	*<4.48E+00*	--	*<4.85E+00*	*<4.26E+00*	--	*<5.00E+00*	--
Ti	1.30E+04	1.0%	2.78E+03	1.3%	2.24E+00	1.15E+03	0.7%	2.60E+02	1.3%
U	3.81E+02	0.7%	7.85E+01	0.8%	*<4.80E+01*	*<=4.42E+01*	--	*<4.94E+01*	--
V	*<4.90E-01*	--	*<3.81E-01*	--	*<4.13E-01*	*<3.62E-01*	--	*<4.25E-01*	--
Zn	1.58E+01	2.3%	3.35E+00	4.4%	*<1.41E+00*	1.46E+00	1.0%	2.90E+00	7.5%
Zr	*<9.80E-01*	--	9.95E-01	2.2%	*<8.26E-01*	*<7.25E-01*	--	*<8.51E-01*	--

*Blank is of comparable magnitude to samples for Sb and Sn – thus detection limit for blank sample reported for all samples.

** Blank corrected values reported; Only single replicate contained detectable Si for second LWPT; Blank Si detection limit reported for third LWPT sample.

Table 3-3. Ion analysis of filtered LWPT samples and as-received LWHT sample

analyte (M)	ARP-14-012/013 (First LWPT)		ARP-14-014/015 (Second LWPT)		ARP-14-016/017 (LWHT)		ARP-14-018/019 (Third LWPT)		ARP-14-020/021 (Fourth LWPT)	
	average	RSD	average	RSD	Average	RSD	average	RSD	average	RSD
OH^-	1.93E+00	0.7%	1.76E-01	2.0%	8.95E-01	0.2%	3.71E-01	4.0%	2.03E+00	0.3%
NO_3^-	2.31E+00	1.0%	2.25E-01	1.6%	1.04E+00	1.9%	1.85E-01	14.8%	1.96E+00	8.7%
NO_2^-	6.64E-01	0.7%	6.14E-02	1.3%	3.05E-01	0.5%	5.22E-02	5.9%	5.39E-01	0.6%
CO_3^{2-}	2.76E-01	0.4%	2.93E-02	1.0%	1.32E-01	0.4%	2.42E-02	0.7%	2.19E-01	0.8%
SO_4^{2-}	8.28E-02	2.7%	7.85E-03	1.3%	3.38E-02	2.2%	7.20E-03	0.3%	6.42E-02	2.8%
$C_2O_4^{2-}$	4.00E-03	0.8%	*<1.14E-03*	--	1.56E-03	4.1%	6.65E-02	6.0%	9.78E-03	2.6%
PO_4^{3-}	4.67E-03	0.8%	*<1.05E-03*	--	1.75E-03	2.1%	*<1.05E-03*	--	4.51E-03	0.3%
CHO_2^-	1.40E-02	1.5%	*<2.22E-03*	--	5.88E-03	1.9%	*<2.22E-03*	--	1.05E-02	0.4%
F^-	*<5.26E-03*	--	*<5.26E-03*	--	*<5.26E-03*	--	*<5.26E-03*	--	*<5.26E-03*	--
Cl^-	7.53E-03	2.1%	*<2.82E-03*	--	3.29E-03	0.6%	*<2.82E-03*	--	5.77E-03	6.6%
Br^-	*<6.26E-03*	--	*<6.26E-03*	--	*<6.26E-03*	--	*<6.26E-03*	--	*<6.26E-03*	--
AlO_2^-	1.30E-01	0.6%	1.18E-02	2.2%	6.28E-02	0.4%	9.71E-03	0.5%	1.01E-01	0.0%
anions	5.79E+00	--	5.54E-01	--	2.59E+00	--	7.89E-01	--	4.98E+00	--
Na^+	5.52E+00	0.0%	5.39E-01	1.1%	2.66E+00	0.2%	7.85E-01	0.4%	4.85E+00	0.6%
Na^+/anions	0.95	--	0.97		1.03		0.99		0.97	

Table 3-4. Anion analysis of as-received slurries from weighted dilutions of LWPT samples

(M)	ARP-14-012/013 (First LWPT)		ARP-14-014/015 (Second LWPT)		ARP-14-018/019 (Third LWPT)		ARP-14-020/021 (Fourth LWPT)	
	average	RSD	average	RSD	Average	RSD	average	RSD
NO_3^-	2.38E+00	5.1%	2.20E-01	2.7%	1.76E-01	0.8%	1.80E+00	1.2%
NO_2^-	6.79E-01	0.4%	6.37E-02	0.3%	5.17E-02	0.7%	5.33E-01	0.5%
CO_3^{2-} *	2.91E-01	2.6%	3.04E-02	8.5%	2.69E-02	4.6%	2.28E-01	2.7%
SO_4^{2-}	7.68E-02	1.8%	7.32E-03	1.1%	6.01E-03	0.7%	5.89E-02	1.1%
$C_2O_4^{2-}$	*<6.85E-03*	--	*<5.51E-03*	--	5.05E-02	3.1%	8.65E-03	2.9%
PO_4^{3-}	*<6.35E-03*	--	*<5.11E-03*	--	*<5.06E-03*	--	*<5.73E-03*	--
CHO_2^-	*<1.34E-02*	--	*<1.08E-02*	--	*<1.07E-02*	--	*<1.21E-02*	--
F^-	*<3.17E-02*	--	*<2.55E-02*	--	*<2.53E-02*	--	*<2.86E-02*	--
Cl^-	*<1.70E-02*	--	*<1.37E-02*	--	*<1.36E-02*	--	*<1.53E-02*	--
Br^-	*<3.77E-02*	--	*<3.04E-02*	--	*<3.01E-02*	--	*<3.40E-02*	--

*Carbonate values blank corrected.

Table 3-5 contains the chemical analysis of the washed and dried solids isolated from the LWPT samples. Note that mercury analysis may reflect a low bias because the solids were dried at 120 °C for 12 hours prior to analysis, potentially volatilizing a portion of the mercury. The vapor pressure of mercury is known to increase by an order of magnitude going from ambient temperature to the 120 °C drying temperature.[10] A separate test using the original air-dried solids (dissolution and mercury analysis) and accounting for their moisture content would be required in order to obtain the actual amount of mercury associated with the filtered and washed solids. Additionally the slurry and filtrate could also be analyzed for mercury, which was not performed in the current testing, to provide further information about the mercury content of these samples. The reported Si values are also blank-corrected due to the presence of significant amounts of Si from the acid-dissolved blank, which ranged from 20% to 85% of the Si observed in the samples. Due to the low quantity of solids from the third and fourth LWPT samples available for analysis, a single analysis was performed for these solids.

All the washed and dried LWPT solid samples contain primarily sodium, in the range of 9.6 to 16.8 wt%, and titanium in the range of 25.0 to 40.2 wt%. Comparison of the Ti to Na mass levels shows that the Ti to Na ratio is as expected from pure MST ($NaHTi_2O_5$) with a Ti to Na mass ratio of 4.2 for both the second and third LWPT solids. This comparison for the first and fourth LWPT solids indicates an excess of Na not associated with MST as the Ti to Na mass ratio is only 2.7 and 1.5, respectively. Other components in the 0.5 to 1 wt% range for these samples are Fe, U and P. Many trace components detectable up to the 0.5 wt% level for these solids are Si, Cd, S, Al, Li and Ca. All of these elements measured in the washed and dried dissolved solids were also noted in previous dissolved solids analyses in which the solids were not washed.[2] As noted in that study, oxalic acid cleaning of the filter could contribute to the observed ~ 2X higher iron level in the third LWPT sample (1.05 wt%) vs. either of the first two LWPT samples with ~ 0.4 wt% iron. By comparison with the filtrate and slurry analyses, the Fe, U and Ca components are likely from the solids portion rather than the supernate portion of the original slurries.

Aluminum and silicon in the dissolved solids from all LWPT samples suggest the possible presence of sodium aluminosilicates (NAS) such as those previously identified in the SRS tank farm derived from sodalite ($Na_8(Al_6Si_6O_{24})Cl_2$) and cancrinite ($Na_6Ca_2[(CO_3)_2Al_6Si_6O_{24}]\cdot 2H_2O$).[2] Both of these sodium aluminosilicates have equal molar amounts in their empirical formulas. Since the molar mass of Al (26.9815 g/mol) and Si (28.0855 g/mol) are similar, then the aluminosilicates would also contain roughly equal mass amounts of these two components. The first and fourth LWPT samples would be limited in aluminosilicate formation by the very low amounts of Si observed, 0.02 and 0.09 wt% Si, respectively, vs. the much higher reported Al values above 0.2 wt%. The second LWPT sample contains nearly equivalent amounts of both Al (0.052 wt%) and Si (0.049 wt%). The third LWPT sample would be limited in aluminosilicate formation by the Al content of 0.24 wt% vs. the higher 0.49 wt% of Si. Thus it would appear that the third LWPT solids sample could contain 2.5X (0.24 wt% Al in third LWPT/0.09 wt% Si in fourth LWPT) to 10X (0.24 wt% Al in third LWPT/0.02 wt% Si in first LWPT) the amount of aluminosilicates as the other LWPT solids. The maximum amount of aluminosilicates in the washed and dried solids can be estimated using a 6X factor (6X Si for the first, second and fourth LWPT and 6X Al for the third LWPT) for either the sodalite or the cancrinite to give 0.12 wt%, 0.28 wt%, 1.45 wt% and 0.52 wt% for the first through fourth LWPT solids samples, respectively. These are very low amounts on a slurry basis. For instance the highest calculated NAS of 1.45 wt% for the third LWPT would only be 1.45% of 0.37wt% total solids, or about 5.4E-3 wt% NAS insoluble solids on a slurry basis.

The data in Table 3-5 for washed and dried solids isolated from the LWPT samples can be used to form a rough estimate of the relative amount of sludge present in the ARP slurries. From the first LWPT sample, the washed dried solids contained 0.414 wt% iron and 37.4 wt% titanium, respectively. Approximating that typical HLW sludge is 20 wt% iron (assuming mostly PUREX), the washed dried solids from the first LWPT sample contained roughly 78 wt% MST and 2.1 wt% sludge. Thus, the solids in the ARP slurries being filtered contained roughly a 38:1 ratio of MST: sludge. Calculations for the second LWPT solids gives a similar MST: sludge ratio of 36:1. The uranium analysis from these dissolved solids cannot be accurately used to estimate the sludge content of the solids because U is adsorbed onto the MST along with Sr, Pu and Np during processing.[11] Thus the calculated sludge amounts using U would be expected to be biased high. As discussed in the previous ARP analysis report,[2] these calculated MST: sludge ratios using iron data are considerably higher than have been studied in previous crossflow filtration testing from 1999 that used nominal ratios of 1:1 MST to sludge and a maximum of 2:1 MST to sludge.[12] However, current ongoing testing with the crossflow filtration unit in SRNL involves MST-only and testing with MST and other added solids.[13]

Table 3-5. Elemental analysis of washed, dried solids isolated from LWPT samples

(wt %)	ARP-14-012/013 (First LWPT) solids		ARP-14-014/015 (Second LWPT) solids		ARP-14-018/019 (Third LWPT) solids	ARP-14-020/021 (Fourth LWPT) solids
	average	RSD	average	RSD		
Ag	*<3.38E-03*	--	*<3.35E-03*	--	*<4.62E-03*	*<5.97E-03*
Al	2.01E-01	0.0%	5.23E-02	0.4%	2.43E-01	4.70E-01
Ba	2.58E-02	0.3%	2.80E-02	1.0%	4.53E-02	1.93E-02
Be	1.47E-03	1.4%	1.61E-03	1.8%	1.24E-03	8.31E-04
Ca	3.22E-01	1.1%	3.64E-01	2.7%	4.43E-01	2.63E-01
Cd	1.50E-01	0.0%	1.62E-01	1.3%	1.44E-01	9.27E-02
Ce	6.19E-02	0.1%	6.76E-02	0.4%	5.37E-02	4.47E-02
Co	1.52E-02	1.4%	1.61E-02	3.5%	1.97E-02	1.18E-02
Cr	6.46E-03	1.2%	5.38E-03	15.4%	3.98E-02	2.43E-02
Cu	*<6.18E-03*	--	<=6.22E-03	--	1.60E-02	*<1.09E-02*
Fe	4.14E-01	0.7%	4.63E-01	0.6%	1.05E+00	4.70E-01
Gd	8.01E-03	5.2%	8.41E-03	12.1%	*<1.06E-02*	*<1.37E-02*
Hg*	3.64E-02	2.9%	6.24E-02	7.8%	9.07E-02	8.06E-02
K	8.04E-02	1.8%	<=7.33E-02	--	*<9.30E-02*	*<1.20E-01*
La	*<3.44E-03*	--	*<3.40E-03*	--	*<4.69E-03*	*<6.06E-03*
Li	2.23E-01	2.9%	2.18E-01	1.0%	1.81E-01	1.39E-01
Mg	*<1.50E-02*	--	*<1.49E-02*	--	*<2.63E-02*	*<2.65E-02*
Mn	*<1.40E-03*	--	1.68E-03	2.1%	3.92E-02	7.45E-03
Mo	*<3.13E-02*	--	*<3.09E-02*	--	*<4.27E-02*	*<5.52E-02*
Na	1.40E+01	2.5%	9.88E+00	3.2%	9.65E+00	1.68E+01
Ni	*<7.93E-03*	--	*<7.85E-03*	--	1.21E-01	1.67E-02
P	7.45E-01	0.8%	8.08E-01	2.3%	9.45E-01	5.16E-01
Pb**	1.26E-02	3.8%	1.71E-02	4.1%	2.19E-02	2.48E-02
S	1.88E-01	20.3%	*<1.11E-01*	--	*<1.53E-01*	3.95E-01
Sb**	*<8.90E-04*	--	*<8.73E-04*	--	2.25E-03	*<1.57E-03*
Si***	1.95E-02	18.1%	4.95E-02	47.1%	4.89E-01	9.10E-02
Sn**	*<=1.18E-02*	--	*<3.67E-03*	--	*<5.11E-03*	*<=8.45E-03*
Sr	8.22E-03	0.7%	8.97E-03	1.0%	1.03E-02	5.71E-03
Th	2.53E-02	4.2%	2.64E-02	0.5%	5.22E-02	2.22E-02
Ti	3.74E+01	0.6%	4.02E+01	1.2%	3.54E+01	2.50E+01
U	9.93E-01	0.8%	1.07E+00	2.0%	9.34E-01	6.49E-01
V	*<5.41E-04*	--	*<5.35E-04*	--	*<7.38E-04*	*<9.54E-04*
Zn	3.64E-02	0.2%	4.60E-02	2.8%	7.49E-02	2.34E-02
Zr	2.48E-02	0.0%	2.71E-02	1.3%	2.48E-02	1.52E-02

* Hg values may reflect low bias due to drying of solids before dissolution.

** Pb values determined from ICPMS using mass numbers 206, 207 and 208; Sb values from ICPMS using mass numbers 121, 123; Sn values from ICPMS using mass numbers 117,118.

*** Si values blank corrected.

3.2 Physical and Solids Analysis

Table 3-6 contains the results for the weight percent solids and density analyses for the LWPT slurries and LWHT solution samples. For most data, the averages are reported along with the standard deviations (st. dev.) and the number of measurements (n). Two sided 95% confidence intervals (CI) are reported for the insoluble solids because it is a calculated value that requires propagation of uncertainty. Both the TS and DS are measured values and the IS is calculated from these data.

The progression of measured total solids and densities for the five ARP samples qualitatively follow the general dilution and concentration changes observed from the earlier filtrate data. Based on the analyzed titanium results in Table 3-2, the calculated insoluble MST solids in the first through fourth LWPT samples are 2.09 wt%, 0.55 wt%, 0.23 wt% and 0.04 wt%, respectively. The first and second LWPT calculated MST values fall within the range encompassed by the insoluble solids measurement reported in Table 3-6 suggesting no other significant solid species are present in these slurries. In addition, the insoluble solids content of the first LWPT sample as determined by calculation from Ti analysis and conversion to MST (2.09 wt%) or the range measured from solids analysis (2.23 ± 0.27 wt%) is in the expected range from nominal processing in the LWPT. That is a nominal 0.2 g/L MST strike would result in a calculated 1.04 wt% MST after 67 cycles at full volume using the measured density for the first LWPT sample shown in Table 3-6. Further concentation from nominally 4,500 gallons down to 1,600 gallons would produce a 2.92 wt% MST slurry.

The calculated MST value for the third LWPT sample is lower than the average insoluble solids measurements by ~ 40% and lower than the lower bound insoluble solids by ~ 20% (0.23 wt% calculated MST vs. 0.37 +/- 0.08 wt% measured insoluble solids). However there are no other potential solids from either the weighted dilution data vs. filtrate data, nor the dissolved washed and dried solids that could quantitatively account for this difference. It is possible that the uncertainty associated with measuring insoluble solids content of slurries with this low of an insoluble solids content (~ 0.37 wt%) is quite high, and thus is inaccurate vs. the calculated MST solids derived from actual Ti analysis. Given the fourth LWPT sample low measured insoluble solids of only 0.09 wt% and associated uncertainty it is likely that the calculated MST value can account for these solids. Data for the second and third LWPT samples in Table 3-6 as well as slurry and filtrate data presented earlier for the second and third LWPT samples show that these two samples are similar to the previously characterized PRFT samples from the DWPF.[6] For instance the quantitative solids data for the second and third LWPT sample from Table 3-6 show TS, DS and IS as 4.3/3.8/0.6 wt%, respectively and 5.4/5.0/0.4 wt%, respectively vs. the PRFT sample with 6.0/5.4/0.6 wt%, respectively. This is to be expected since both of these samples were obtained just prior to transfer of LWPT samples to the LPPP-PPT.

Table 3-6. Solids quantitative analysis and density measurement of ARP 512-S samples

Sample	Total Solids (TS) (wt% in slurry)			Dissolved Solids (DS) (wt% in filtrate)			Insoluble Solids (IS) (wt% in slurry)		Density (g/mL) (Slurry(LWPT) or Solution (LWHT)	
	avg.	st.dev.	n	avg.	st.dev.	n	avg.	95% C.I.	avg.	st.dev.
ARP-14-012/013 (First LWPT)	33.3	0.1	3	31.7	0.1	3	2.23	0.27	1.292	0.003
ARP-14-014/015 (Second LWPT)	4.3	0.01	3	3.8	0.02	3	0.60	0.06	1.048	0.022
ARP-14-016/017 (LWHT)	16.1	0.1	3	see Total Solids			NA	NA	1.138	0.007
ARP-14-018/019 (Third LWPT)	5.4	0.02	3	5.0	0.03	3	0.37	0.08	1.062	0.029
ARP-14-020/021 (Fourth LWPT)	26.8	0.1	3	26.7	0.1	3	0.09	0.46	1.233	0.003

Figure 3-1 shows an example of the flow curves obtained during rheology measurements. Other flow curves obtained are presented in Appendix A. No yield stress (positive intercept in the flow curve slope) was noted for these samples. Newtonian viscosities (with units of cP) can be estimated from 1000-times the slope of the plot of the shear stress (in Pa) versus the shear rate (in1/s). Table 3-7 contains a compilation of the viscosities of the samples measured at the nominal cell temperature of approximately 25 °C.

The measured viscosities of the three highest concentrated samples are in the range of 1.7 to 3.2 cP for the first LWPT, the LWHT and the fourth LWPT samples that were 5.5M Na^+, 2.6M Na^+ and 4.8M Na^+, respectively. The lower concentrated second LWPT and third LWPT have lower measured viscosities in the range of 0.72 to 1.1 cP.

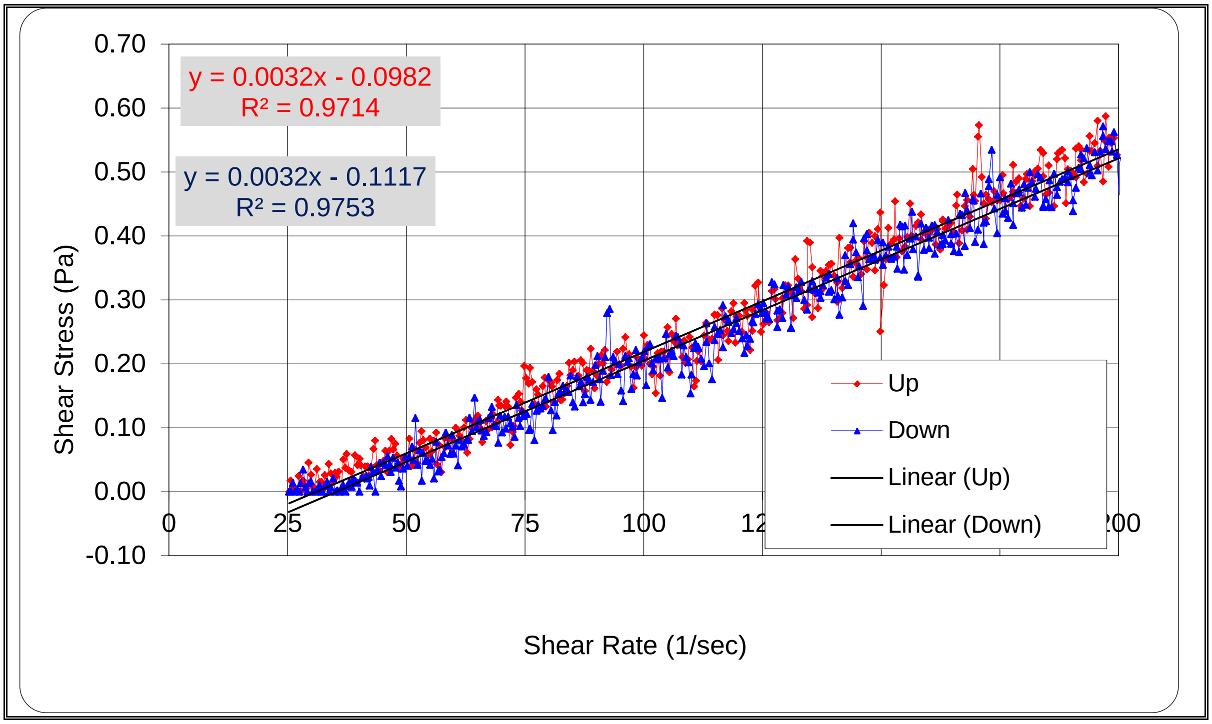


Figure 3-1. Example flow curve (rheology measurement) of first LWPT slurry

Table 3-7. Viscosity measurements of ARP 512-S samples (at approx. 25 °C)

sample	Average (cP)	(RSD)
First LWPT	3.2	0%
Second LWPT	1.1	6.7%
LWHT	1.7	13%
Third LWPT	0.72	14%
Fourth LWPT	2.9	12%

Figure 3-2 contains a plot of the particle size volume distributions of the four LWPT samples. Appendix B contains the instrument results for the particle size measurements, including both the volume and the number distributions. All four samples were similar, with the first, second and fourth LWPT samples being almost identical. The third LWPT post filter cleaning sample contained a small amount of large particles (possibly due to aggregation or another phenomenon) and had slight elongations of the distributions on the high end of the larger particles. Similar results were observed for the post filter cleaning LWPT sample in the previous study.[2] The volume distributions for the LWPT samples were bimodal with the major fractions centered around 6.5 µm and the minor fraction centered around 1.4 µm. The particle size measurements are consistent with the measurements made on the Harrell Industries MST feed materials, which are characterized upon receipt.[6] Differences can be attributed to the different instruments used for the measurements of the MST feeds.

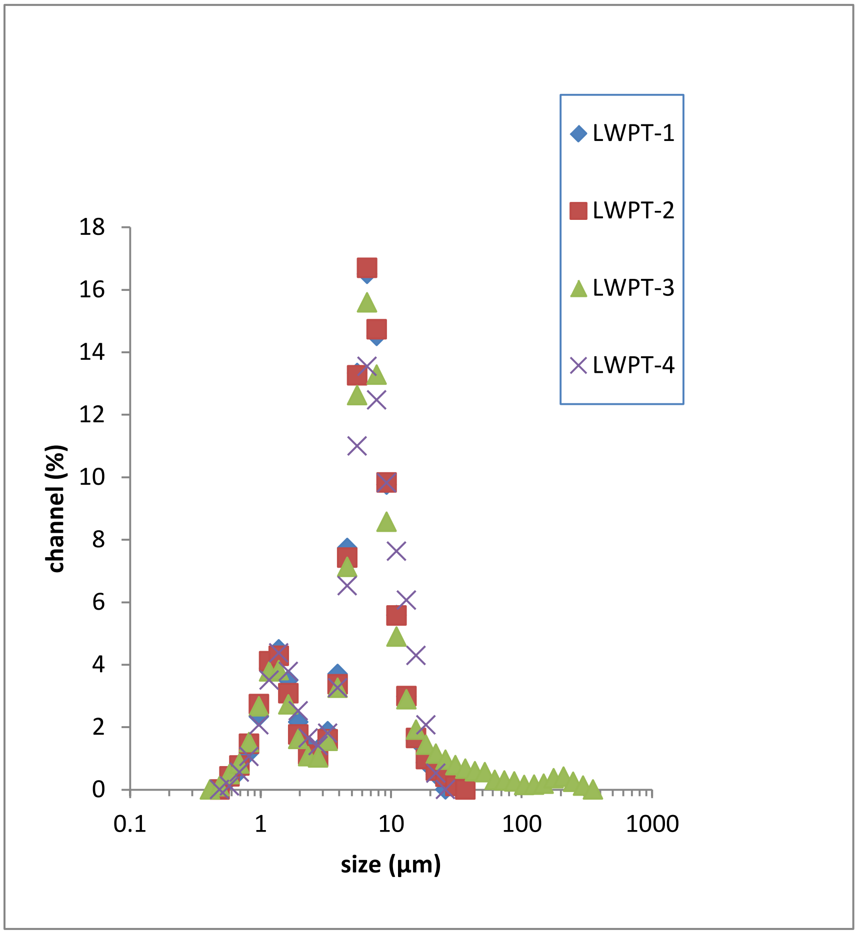


Figure 3-2. Particle size volume distributions of LWPT samples

Figure 3-3, Figure 3-4, Figure 3-5 and Figure 3-6 contain the XRD results for the washed solids isolated from the first, second, third and fourth LWPT sample, respectively. All four samples matched a sodium titanium oxide hydroxide compound ($Na_2Ti_2O_4(OH)_2$). The peaks are somewhat broad and poorly defined and weak, consistent with the XRD results from the previous ARP sample analysis[2] and the PRFT sample analysis.[6] This compound is the XRD instrument library match that is seen when analyzing "as-received" MST ($NaHTi_2O_5$). The origin of the XRD peak profile assignment is not from MST, but is from another related material. The presence of these solids indicate that there is MST present in the samples.

The ethanol washed solids from the first through the third LWPT samples do not show any other peaks, which is similar to previous water washed PRFT solids.[6] A detectable amount of sodium oxalate ($Na_2C_2O_4$) is observed in the fourth LWPT solid sample shown in Figure 3-6, which suggests that the sodium oxalate is present as a solid in this final LWPT slurry. Similar detectable amounts of sodium oxalate were also reported for the LWPT-1 and LWPT-2 samples from 2013.[2] The fourth LWPT sample, unlike the first through third LWPT sample, had more processing steps (transfer of neutralized chemical cleaning solution, addition of caustic and dilute caustic, draining of heal in backpulse tank and/or crossflow filter to surge tank, and receipt of next batch) involved before it was collected. This particular fourth LWPT sample also had the lowest Ti to Na ratio of 1.5 from the solids analysis from Table 3-5 suggesting other Na-containing solids may be present beyond the expected MST.

A generalized comparison of the previous LWPT samples vs. the current LWPT samples can be made with respect to the observed presence of sodium oxalate. The previous LWPT-1 and LWPT-2 were both much higher in sodium concentration compared to either the first or second LWPT samples from this work. They were also obtained from earlier in the processing (cycle 16 vs. cycle 67). The previous LWPT-3 sample and the current third LWPT sample were both collected from the LWPT after filter cleaning and pH adjustment. Neither of these 'heel' samples showed detectable amounts of sodium oxalate. The fourth LWPT sample from this study, which does show detectable sodium oxalate, represents material collected from the LWPT after a new batch from 241-96H was received, and thus is not comparable to any of the previous LWPT samples. Considering the current LWPT samples, the data suggests that insoluble sodium oxalate is present at the start of processing, but is not present after 67 cycles of processing.

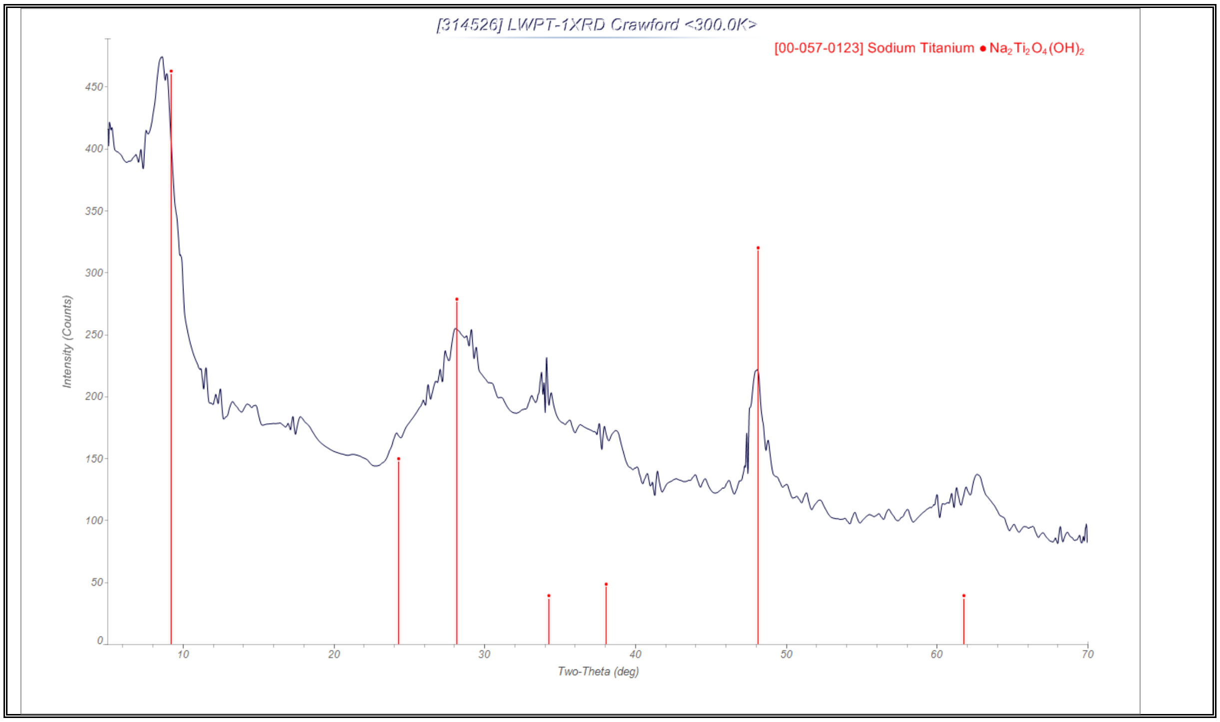


Figure 3-3. XRD analysis of washed solids isolated from the first LWPT sample

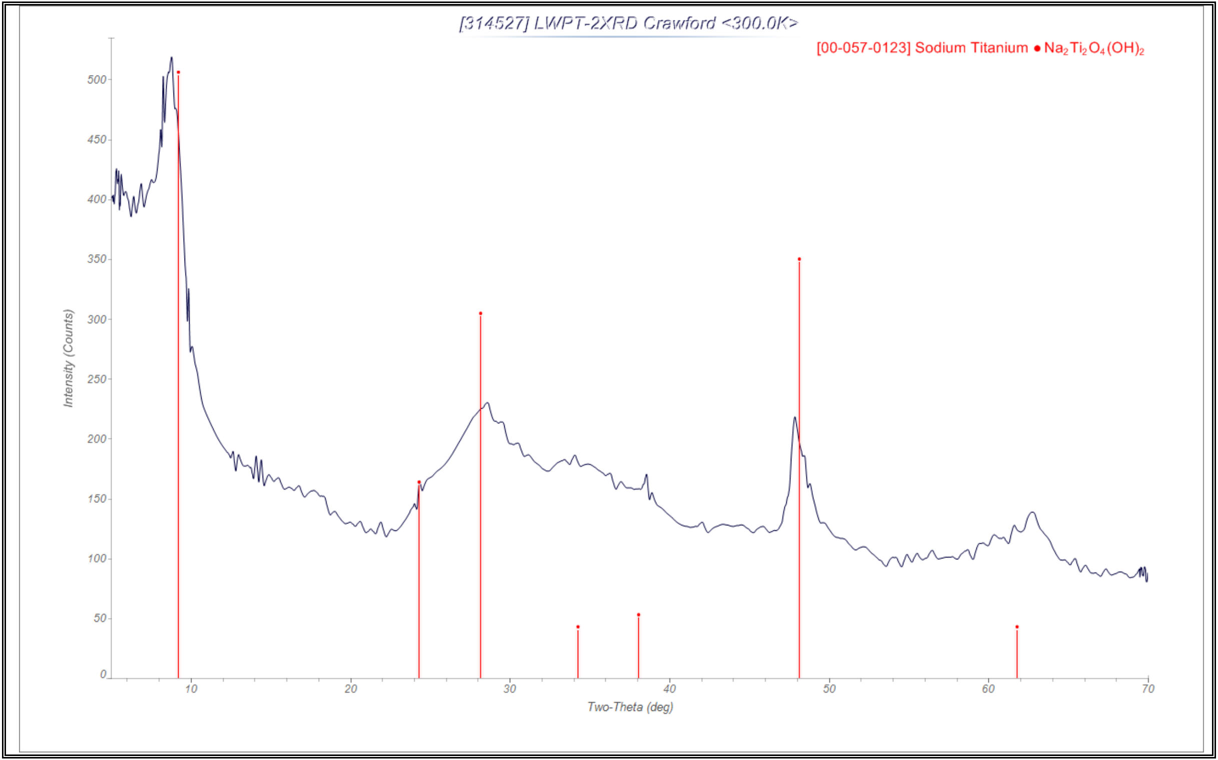


Figure 3-4. XRD analysis of washed solids isolated from the second LWPT sample

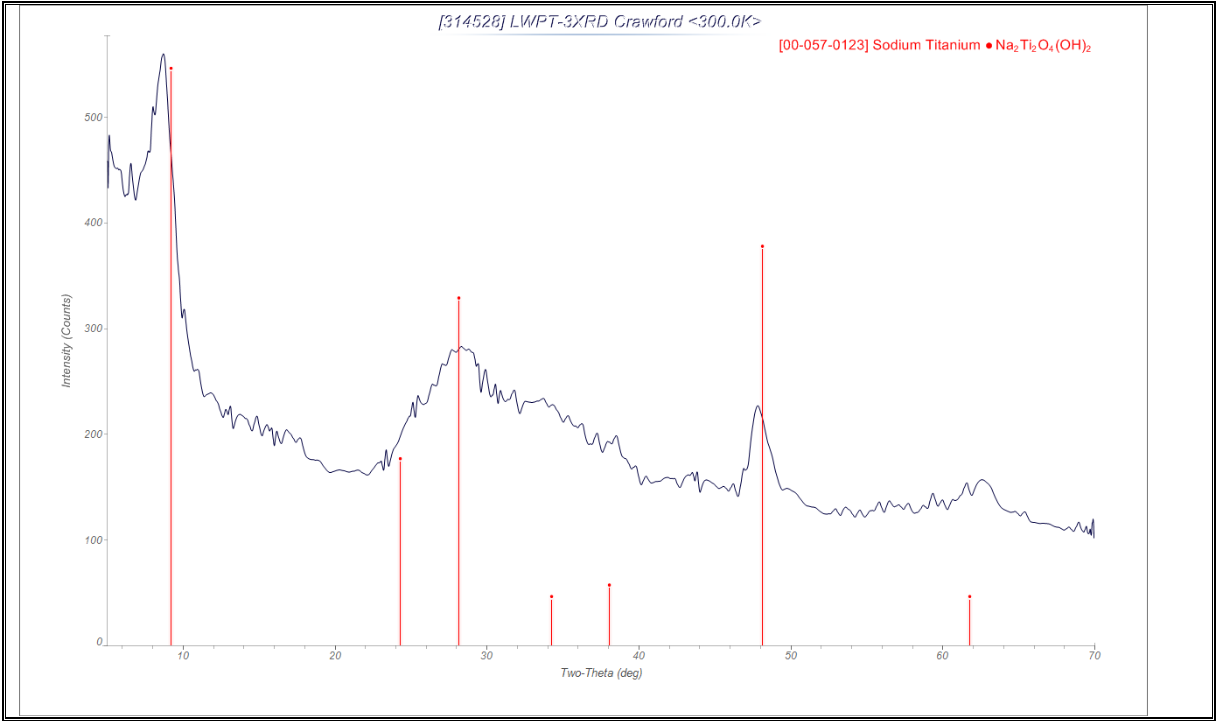


Figure 3-5. XRD analysis of washed solids isolated from the third LWPT sample

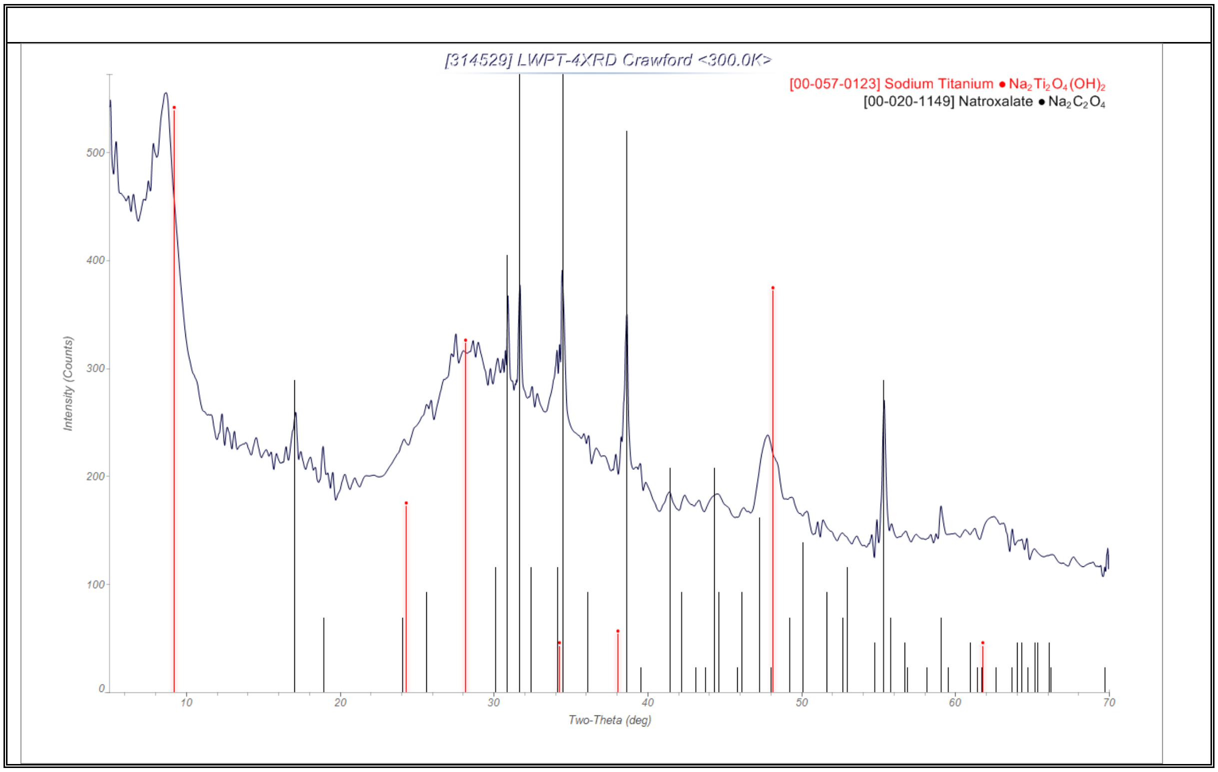
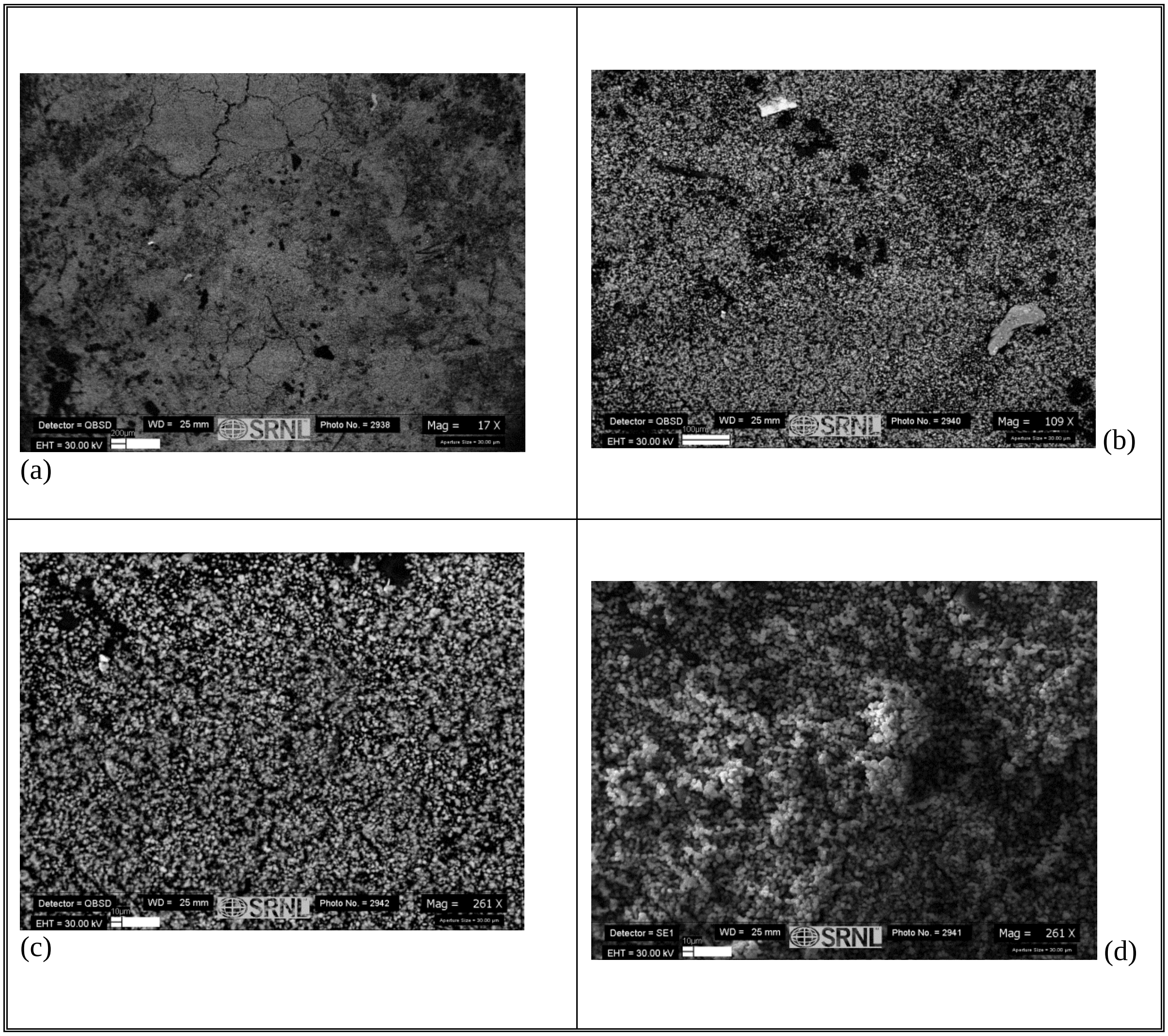


Figure 3-6. XRD analysis of washed solids isolated from the fourth LWPT sample

Figure 3-7 contains example SEM images from washed solids isolated from the first LWPT sample and Figure 3-8 shows washed solids data from the second LWPT sample. These images show increasing magnification levels going from (a) low magnification to (b) mid-range magnification to (c) highest magnification, respectively, of the SEM backscattered electrons for each sample. In the backscattering mode of the SEM electrons from the incident beam are analyzed. The final SEM image in both figures (d) shows an image obtained by analyzing the secondary electrons emitted from atoms on the surface of the samples at the highest magnification. The higher magnification images show roughly spherical MST solids with particle sizes that appear consistent with the PSD analysis, i.e., on the order of < 10 microns. The magnification for each image is shown in the lower right portion. The scale bars for these figures are visible in the lower left portion of the micrograph as 200, 100 and 10 microns for Figure 3-7 (a), (b) and (c), respectively, and 200, 20 and 30 microns for Figure 3-8 (a), (b) and (c), respectively. Appendix C contains additional SEM images for the LWPT samples along with Energy Dispersive Spectroscopy (EDS) elemental analysis for several locations within each image. Figure C- 1 through Figure C- 7 show data from the first LWPT sample and Figure C- 8 through Figure C- 12 show data from the second LWPT sample. Data from the first LWPT sample indicate that the solids are primarily MST with a few localized spots or fragments associated with either Al, Fe or Zn/Cl. These particles appear to be on the order of 50 to 100 microns. Trace levels of Al, Si, S, U, K, Ca and Fe are also observed in the EDS spectra. All of the second LWPT sample data show primarily MST with trace levels of Si, U, K, Ca and Fe present. No Al, Fe or Zn/Cl rich fragments were identified in these second LWPT samples. One possible explanation for the absence of these fragments in the second LWPT solids could be that they are dissolved during the washing process that dilutes the first LWPT slurries by ~ 10.5X.



(a)

(b)

(c)

(d)

Figure 3-7. Example SEM images from first LWPT sample

(a) 17-X magnification; (b) 109-X magnification; (c) 261-X magnification and (d) 261-X magnification; Images a-c obtained from backscattering mode and image d obtained in secondary electron mode.

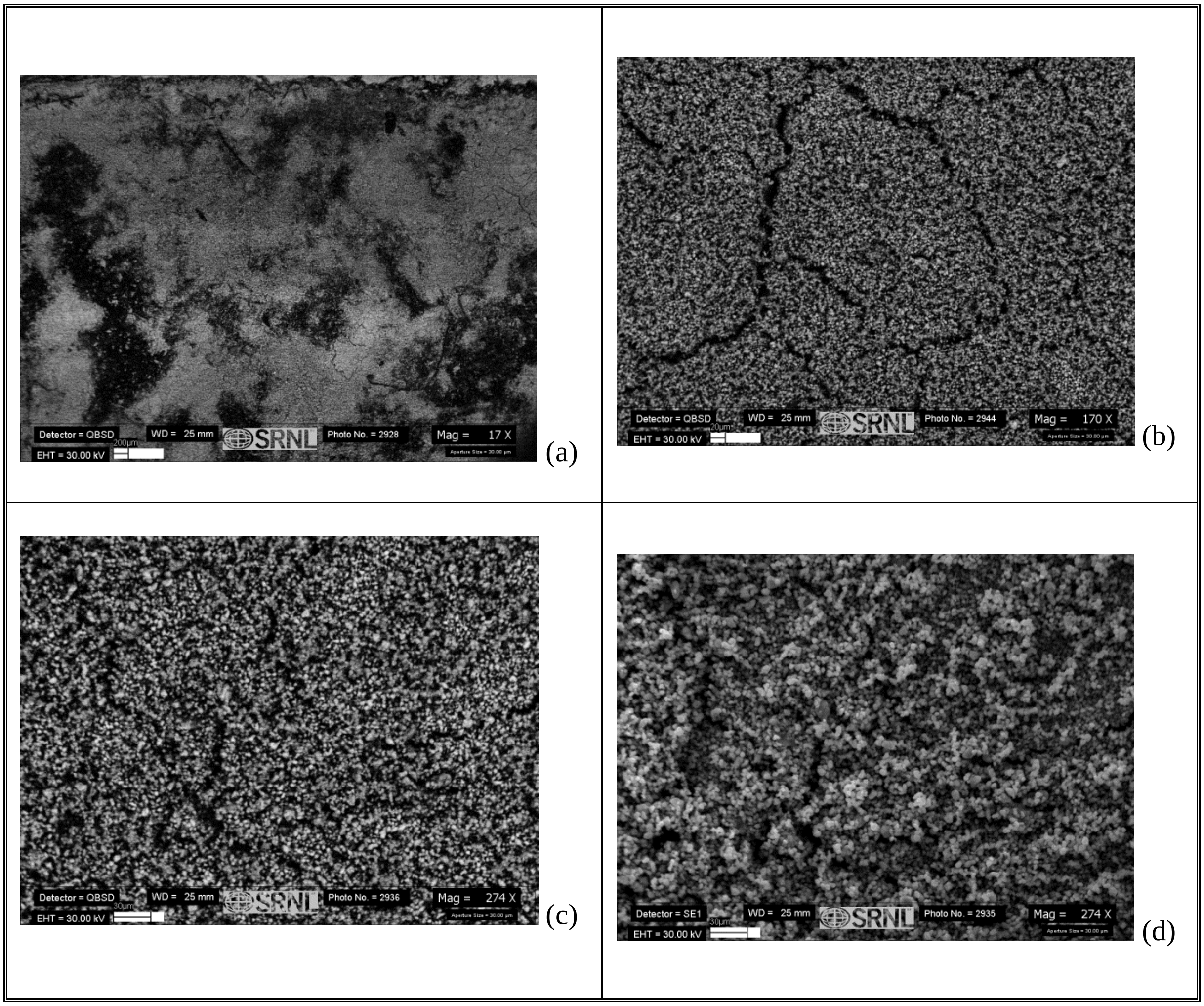


Figure 3-8. Example SEM images from second LWPT sample

(a) 17-X magnification; (b) 170-X magnification; (c) 274-X magnification and (d) 274-X magnification. Images a-c obtained from backscattering mode and image d obtained in secondary electron mode.

4.0 Conclusions

The following are the key results for sample analysis from the ARP process tanks collected over a five day sequence in this study involving 1) the first and second LWPT samples before and after initiation of batch washing, respectively; 2) the LWHT sample obtained upon completion of batch washing and after initiation of chemical cleaning; and 3) the third and fourth LWPT samples collected after filtration.

- At the time of sampling, the first LWPT slurry in the ARP 512-S was 5.5 M Na^+, 1.9 M OH^- and 2.3 M NO_3^- salt solution with 2.09 wt% of insoluble solids. Based on major analyzed species (Al, Na^+, OH^-, NO_3^-, NO_2^-, CO_3^{2-} and SO_4^{2-}) subsequent samples relative to the first LWPT sample were found to be 10.5-times, 2.3-times, 12.3-times and 1.2-times dilute, respectively, for the second LWPT, the LWHT and the third and fourth LWPT samples.

- The oxalate concentration of the first LWPT sample is very similar to the expected oxalate concentration from blend evaluation calculations for SB7b, whereas in previous analysis the LWPT-1 oxalate concentration was ~ 2.7-times lower than the analyzed Tank 49H oxalate concentration.

- Higher sodium concentrations are observed between the previous set of samples for Tank 49H and LWPT-1, with Na at 6.03±0.03M and 6.57±0.15 M, respectively, and the current initial LWPT sample (Na = 5.52±0.00 M) and calculated blend (Na = 6.22 M).

- For both the third and fourth LWPT samples the oxalate concentrations are greater than expected based on the dilution factors of other major components. This is expected since oxalate is introduced during oxalic acid chemical cleaning (before the third and fourth LWPT samples were collected).

- Comparison of the filtrate anion species with those determined from water-weighted dilutions of the slurry indicate no significant solid species/salts dissolve upon dilution with water.

- The second and third LWPT samples are similar based on major filtrate components, dissolved slurry analysis and measured solids, to the PRFT samples reported in earlier studies from the DWPF.

- The insoluble solids contain primarily titanium and are consistent with the MST added during processing. Based on slurry analysis for titanium, the first through fourth LWPT samples have 2.09 wt%, 0.55 wt%, 0.23 wt% and 0.04 wt% MST, respectively. These MST values for the two highest MST containing samples fall within the range encompassed by separate insoluble solids measurements.

- A very small quantity of sodium aluminosilicate in the range of 0.12 wt% to 1.45 wt% of washed and dried solids may also be present in the insoluble solids, based on the detection of both Al and Si present in the solids isolated from all LWPT samples. For instance the total NAS solids in the third LWPT would be only 5.4E-3 wt% insoluble NAS solids on a slurry basis (1.45 wt% of 0.37 wt% total insoluble solids).

- The primary species identified in all the washed and dried solids by XRD is the MST. No evidence of crystalline sodium aluminosilicate species or aluminum containing gibbsite was observed in the XRD.

- Sodium oxalate (natroxalate) was identified in the fourth LWPT solid and was not detectable in the other LWPT solids. Since the fourth LWPT sample contains new material transferred in from 241-96H, this suggest that the sodium oxalate is present at the start of processing but is removed after processing 67 batches.

- Based on the quantity of iron in the solids from the first two LWPT samples, High-Level Waste sludge also appears to be present as a minor fraction of the solids in the ARP slurries, with sludge present at roughly 1/38th the mass of MST present.

- The ARP 512-S slurries exhibited Newtonian flow behavior, with the higher concentration slurries (first and fourth LWPT samples) having a viscosity of 2.9 to 3.2 cP and the LWHT solution at ~ 2X lower concentration having a viscosity of 1.7 cP at 25 ºC. The second and third LWPT slurries below 1M Na^+ showed viscosities in the range of 0.7 to 1.1 cP. Flow behavior observed for the samples would not appear to challenge the filtration system outside of the expected salt feeds.

- Particle size distribution analysis of the four LWPT slurries shows that these solids are very similar to the starting MST slurries that contain a bi-modal distributions centered around a lower 1.4 micron range and a higher 6.5 micron range.

- Microscopy data obtained for the washed solids shows that they are primarily MST of similar particle size with that expected from the PSD data. Some minor 50 to 100 micron sized fragments enriched in either Al, Fe or Zn/Cl were identified in the first LWPT solids.

5.0 Recommendations

- Equilibrium modelling with OLI Analyzer™ and OLI ESP™ could be performed in the manner of previous ARP analysis[2] or recent preliminary evaluations pertaining to solids precipitation in the ARP LWHT with sump residue in surge tank[8] to determine chemical components at risk of precipitation and to simulate the ARP process for these Salt Batch 7B samples.

- Future analysis of 512-S samples should incorporate ICP-MS analysis along with ICP-ES to obtain accurate and low detection limits for trace components such as Pb, Sb and Sn since ICP-MS analysis is known to have better detection limits for these trace components vs. ICP-ES.

- Although the mixed acid dissolution method appears adequate for the complete dissolution of MST slurries and solids, additional dissolution methods involving alkali fusion could also be applied to the LWPT slurries and solids in attempts to measure the Si concentrations without background interference from the HF acid used in mixed acid dissolutions.

- It was noted that mercury analysis of the solids may reflect a low bias because the solids were dried at 120 °C for 12 hours prior to analysis, potentially volatilizing a portion of the mercury. A separate test using the original air-dried solids (dissolution and mercury analysis) and accounting for their moisture content would be required in order to obtain the actual amount of mercury associated with the filtered and washed solids. Additionally the slurry and filtrate could also be analyzed for mercury, which was not performed in the current testing, to provide further information about the mercury content of these samples.

6.0 References

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Appendix A. Rheology Measurements of 512-S Samples

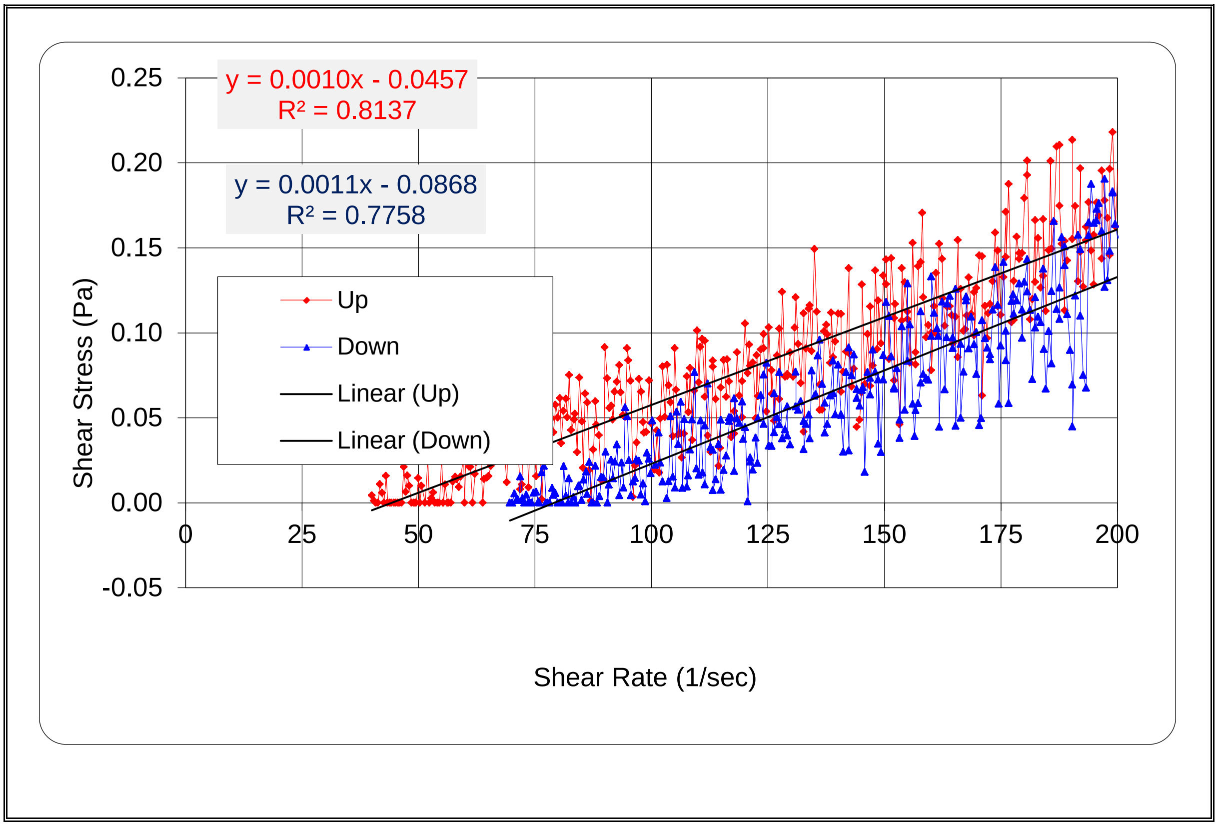


Figure A -1. Flow curves of successive measurements from second LWPT sample

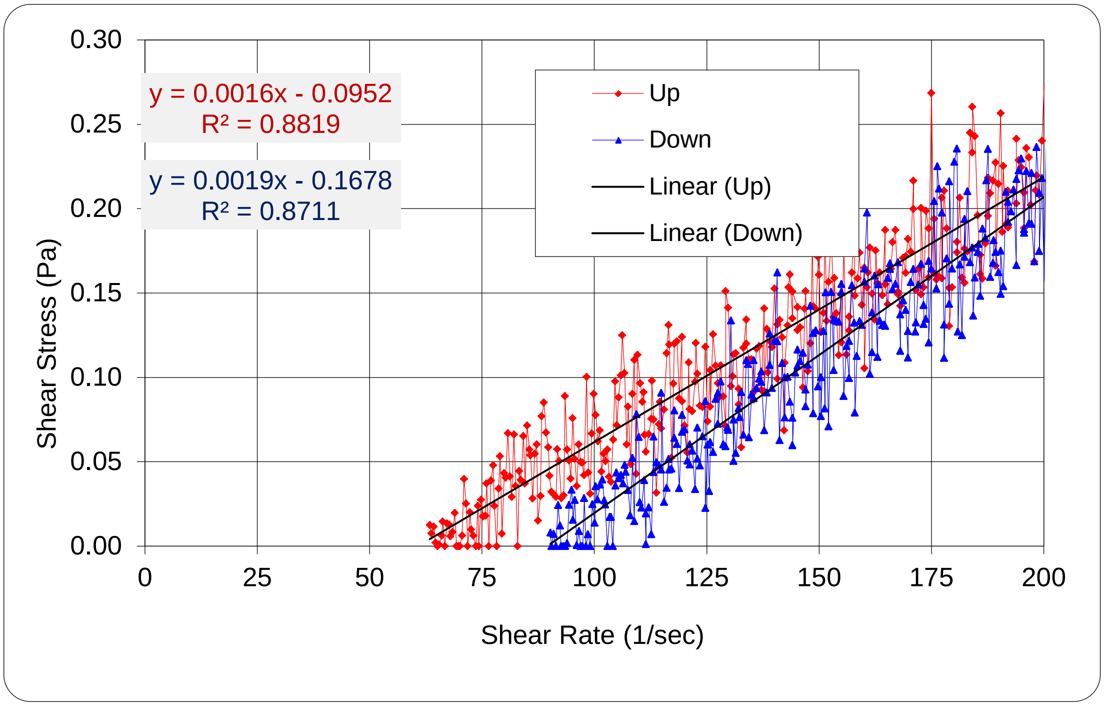


Figure A-2. Flow curves of successive measurements from LWHT sample

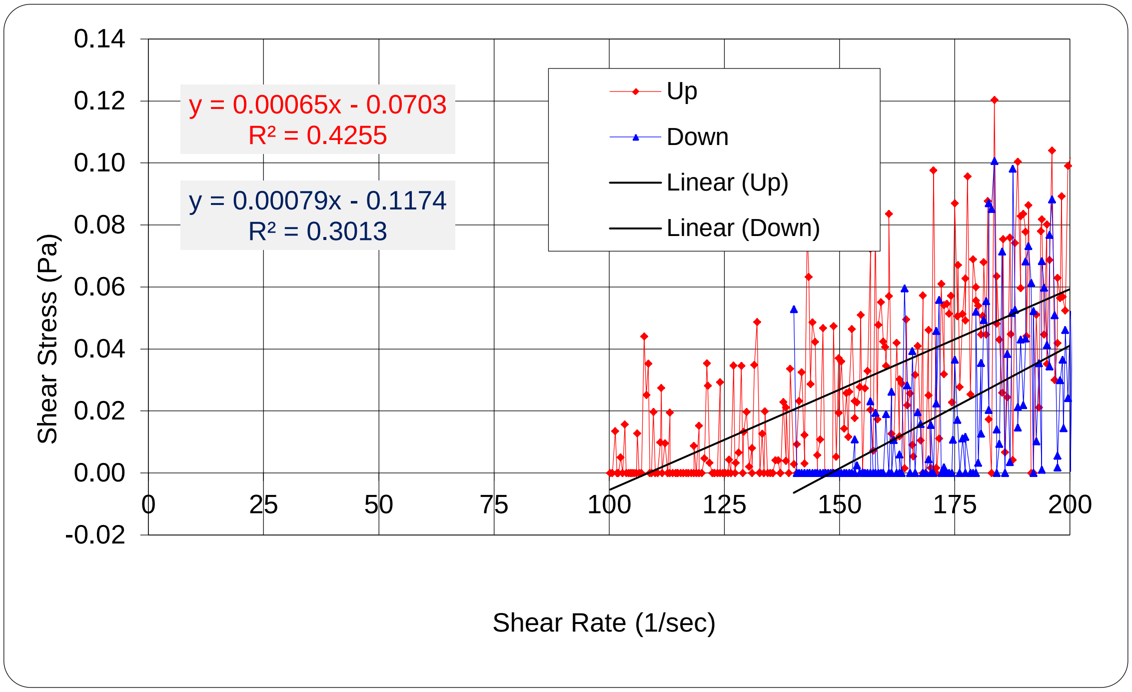


Figure A-3. Flow curves of successive measurements from third LWPT sample

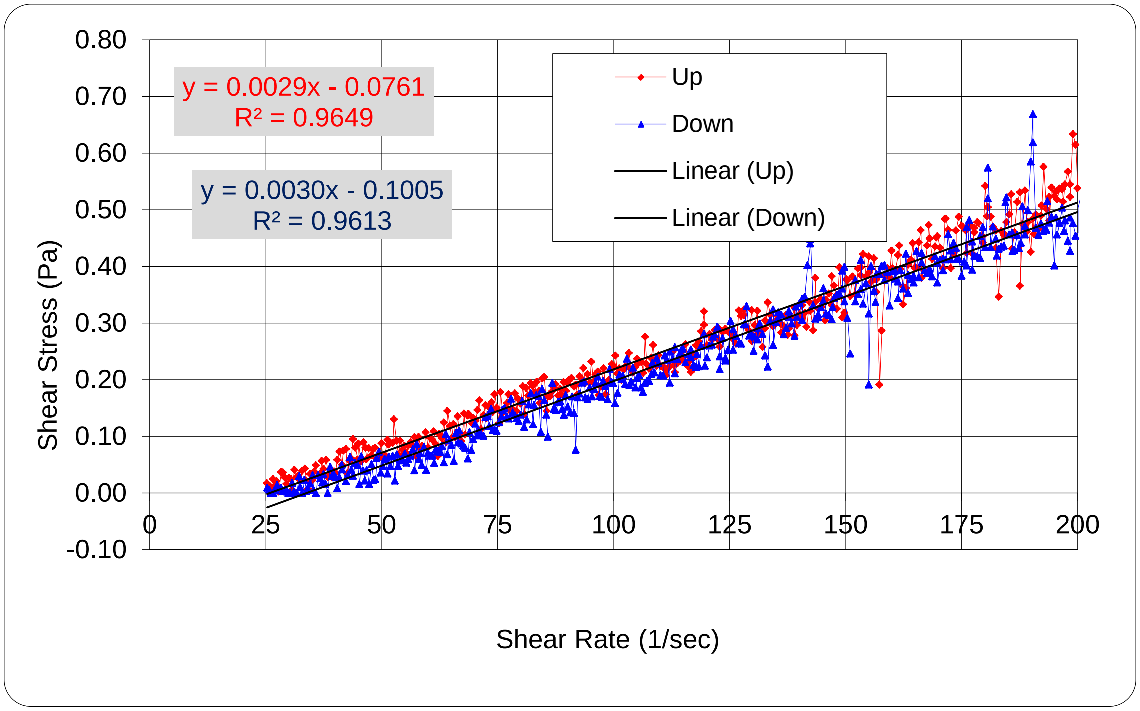
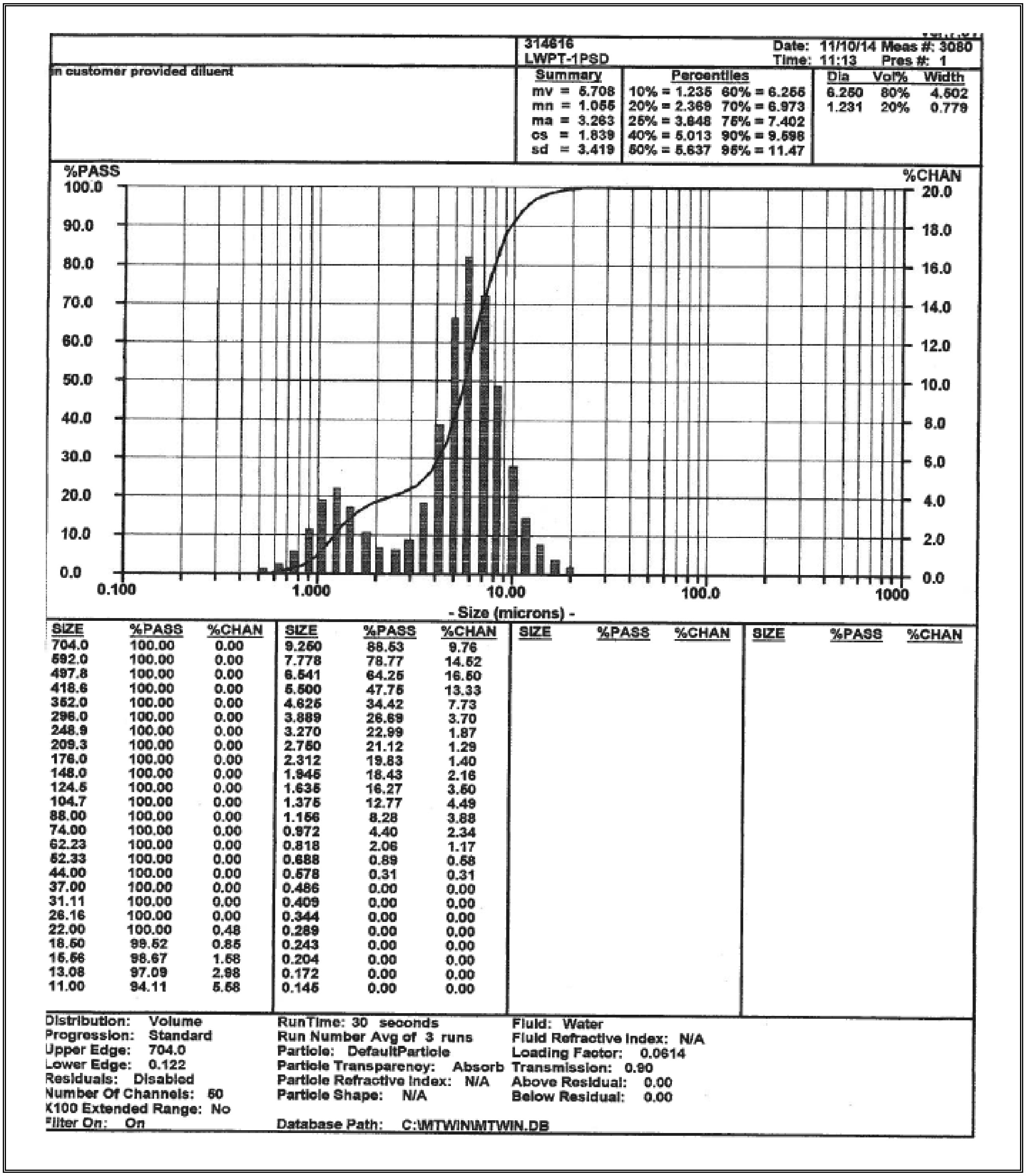


Figure A-4. Flow curves of successive measurements from fourth LWPT sample

Appendix B. Particle Size Distribution Measurements of LWPT Samples



314616
LWPT-1PSD

Date: 11/10/14 Meas #: 3080
Time: 11:13 Pres #: 1

n customer provided diluent

Summary	Percentiles		Dia	Vol%	Width
mv = 5.708	10% = 1.235	60% = 6.256	6.250	80%	4.502
mn = 1.055	20% = 2.369	70% = 6.973	1.231	20%	0.779
ma = 3.263	25% = 3.848	75% = 7.402			
cs = 1.839	40% = 5.013	90% = 9.598			
sd = 3.419	50% = 5.637	95% = 11.47			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	88.53	9.76						
592.0	100.00	0.00	7.778	78.77	14.52						
497.8	100.00	0.00	6.541	64.25	16.50						
418.6	100.00	0.00	5.500	47.75	13.33						
352.0	100.00	0.00	4.625	34.42	7.73						
296.0	100.00	0.00	3.889	26.69	3.70						
248.9	100.00	0.00	3.270	22.99	1.87						
209.3	100.00	0.00	2.750	21.12	1.29						
176.0	100.00	0.00	2.312	19.83	1.40						
148.0	100.00	0.00	1.945	18.43	2.16						
124.5	100.00	0.00	1.635	16.27	3.50						
104.7	100.00	0.00	1.375	12.77	4.49						
88.00	100.00	0.00	1.156	8.28	3.88						
74.00	100.00	0.00	0.972	4.40	2.34						
62.23	100.00	0.00	0.818	2.06	1.17						
52.33	100.00	0.00	0.688	0.89	0.58						
44.00	100.00	0.00	0.578	0.31	0.31						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.48	0.289	0.00	0.00						
18.50	99.52	0.85	0.243	0.00	0.00						
15.56	98.67	1.58	0.204	0.00	0.00						
13.08	97.09	2.98	0.172	0.00	0.00						
11.00	94.11	5.58	0.145	0.00	0.00						

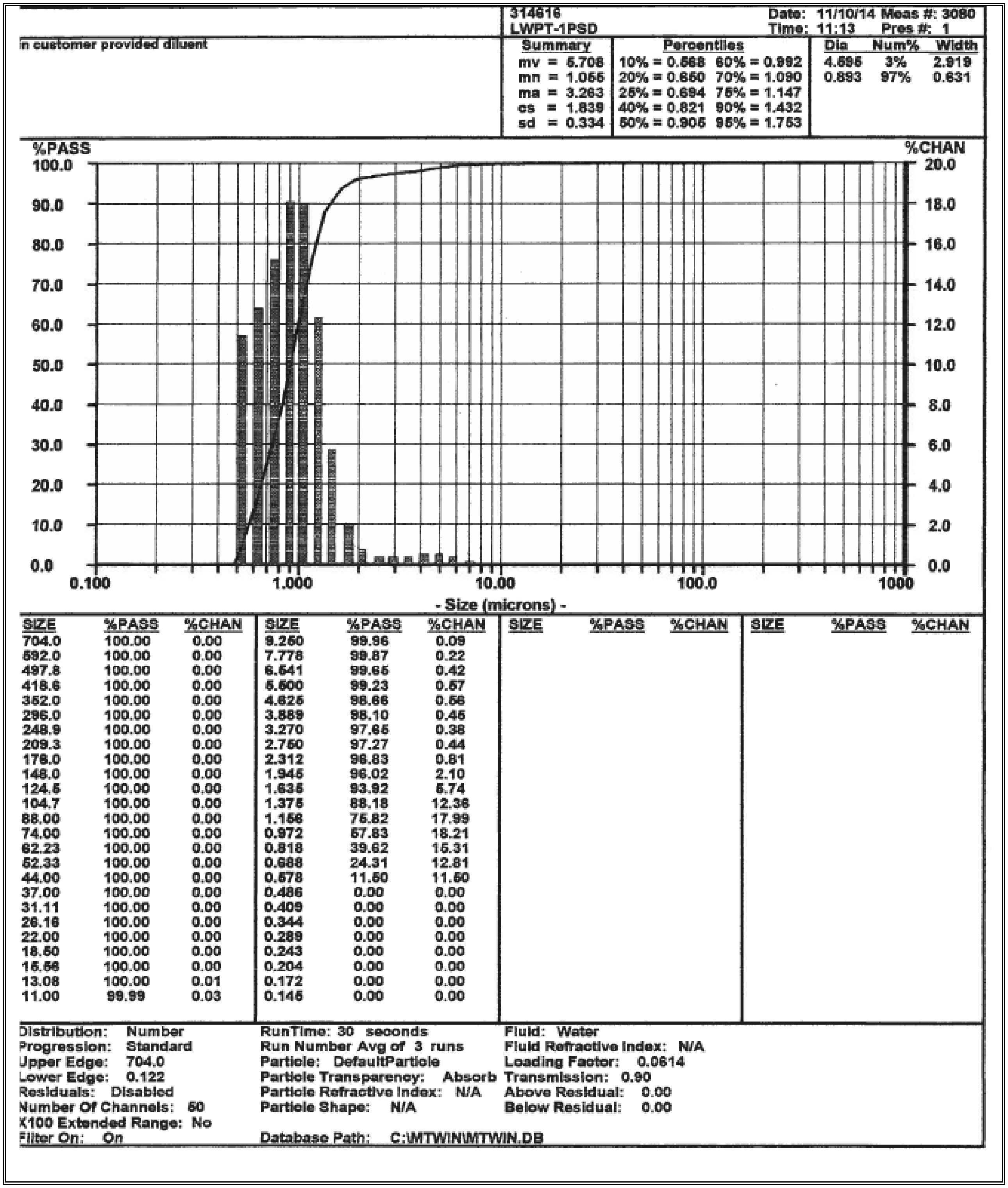
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0614
Transmission: 0.90
Above Residual: 0.00
Below Residual: 0.00

Figure B-1. First LWPT particle size volume distribution measured in salt simulant



314616
LWPT-1PSD

Date: 11/10/14 Meas #: 3080
Time: 11:13 Pres #: 1

n customer provided diluent

Summary	Percentiles		Dia	Num%	Width
mv = 5.708	10% = 0.568	60% = 0.992	4.695	3%	2.919
mn = 1.055	20% = 0.650	70% = 1.090	0.893	97%	0.631
ma = 3.263	25% = 0.694	75% = 1.147			
cs = 1.839	40% = 0.821	90% = 1.432			
sd = 0.334	50% = 0.905	95% = 1.753			

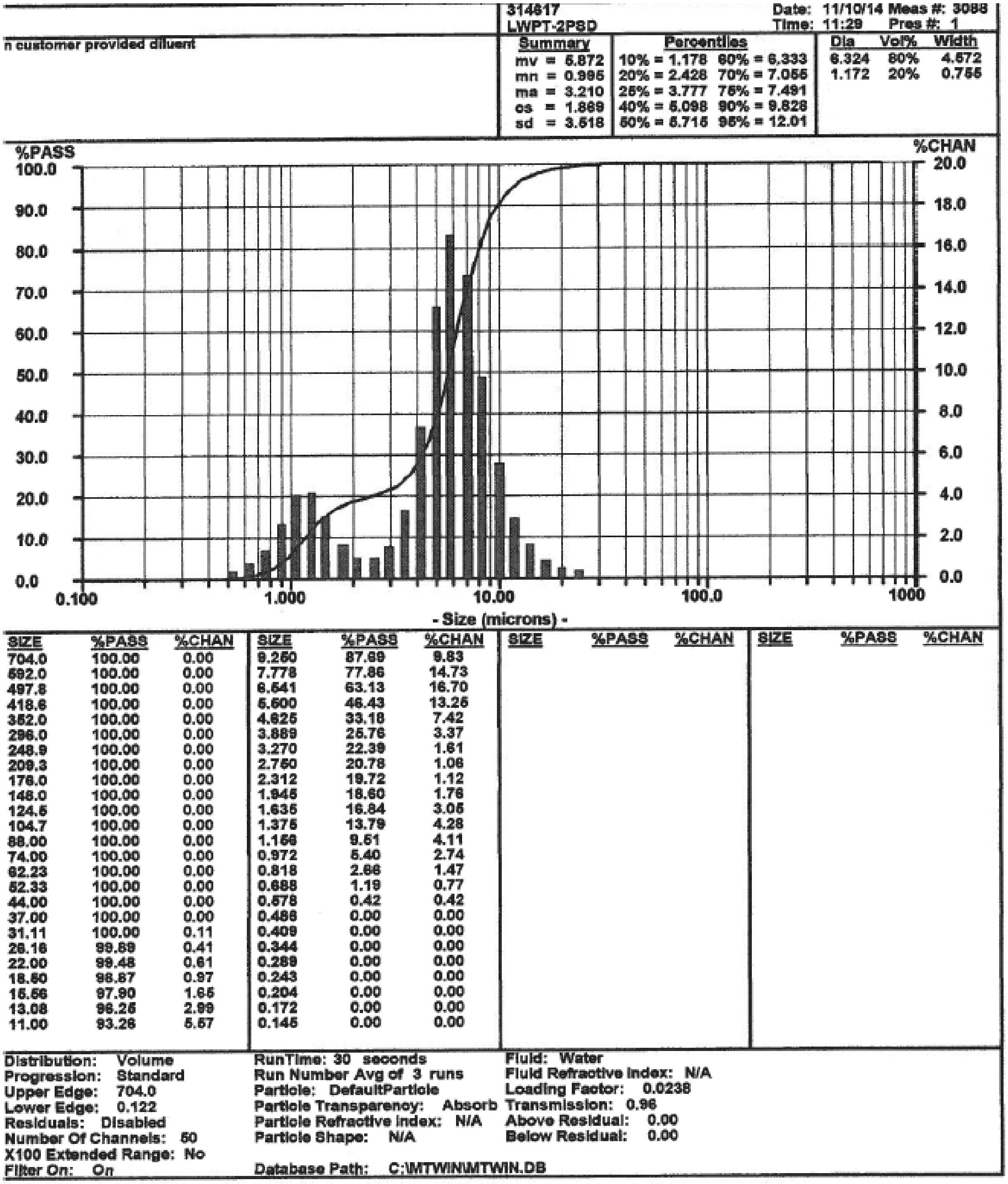
SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.96	0.09						
592.0	100.00	0.00	7.778	99.87	0.22						
497.8	100.00	0.00	6.541	99.65	0.42						
418.6	100.00	0.00	5.500	99.23	0.57						
352.0	100.00	0.00	4.625	98.66	0.56						
296.0	100.00	0.00	3.889	98.10	0.45						
248.9	100.00	0.00	3.270	97.65	0.38						
209.3	100.00	0.00	2.750	97.27	0.44						
176.0	100.00	0.00	2.312	96.83	0.81						
148.0	100.00	0.00	1.945	96.02	2.10						
124.5	100.00	0.00	1.635	93.92	5.74						
104.7	100.00	0.00	1.375	88.18	12.36						
88.00	100.00	0.00	1.156	75.82	17.99						
74.00	100.00	0.00	0.972	57.83	18.21						
62.23	100.00	0.00	0.818	39.62	15.31						
52.33	100.00	0.00	0.688	24.31	12.81						
44.00	100.00	0.00	0.578	11.50	11.50						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.03	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0614
Transmission: 0.90
Above Residual: 0.00
Below Residual: 0.00

Figure B-2. First LWPT particle size number distribution measured in salt simulant



314617
LWPT-2PSD

Date: 11/10/14 Meas #: 3088
Time: 11:29 Pres #: 1

n customer provided diluent

Summary	Percentiles		Dia	Vol%	Width
mv = 5.872	10% = 1.178	60% = 6.333	6.324	80%	4.572
mn = 0.995	20% = 2.428	70% = 7.055	1.172	20%	0.755
ma = 3.210	25% = 3.777	75% = 7.491			
cs = 1.869	40% = 5.098	90% = 9.828			
sd = 3.518	50% = 5.716	95% = 12.01			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	87.69	9.83						
592.0	100.00	0.00	7.778	77.86	14.73						
497.8	100.00	0.00	6.541	63.13	16.70						
418.6	100.00	0.00	5.500	46.43	13.25						
352.0	100.00	0.00	4.625	33.18	7.42						
296.0	100.00	0.00	3.889	25.76	3.37						
248.9	100.00	0.00	3.270	22.39	1.61						
209.3	100.00	0.00	2.750	20.78	1.06						
176.0	100.00	0.00	2.312	19.72	1.12						
148.0	100.00	0.00	1.945	18.60	1.76						
124.5	100.00	0.00	1.635	16.84	3.05						
104.7	100.00	0.00	1.375	13.79	4.28						
88.00	100.00	0.00	1.156	9.51	4.11						
74.00	100.00	0.00	0.972	5.40	2.74						
62.23	100.00	0.00	0.818	2.68	1.47						
52.33	100.00	0.00	0.688	1.19	0.77						
44.00	100.00	0.00	0.578	0.42	0.42						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.11	0.409	0.00	0.00						
26.16	99.89	0.41	0.344	0.00	0.00						
22.00	99.48	0.61	0.289	0.00	0.00						
18.50	98.87	0.97	0.243	0.00	0.00						
15.56	97.90	1.65	0.204	0.00	0.00						
13.08	96.25	2.99	0.172	0.00	0.00						
11.00	93.26	5.57	0.145	0.00	0.00						

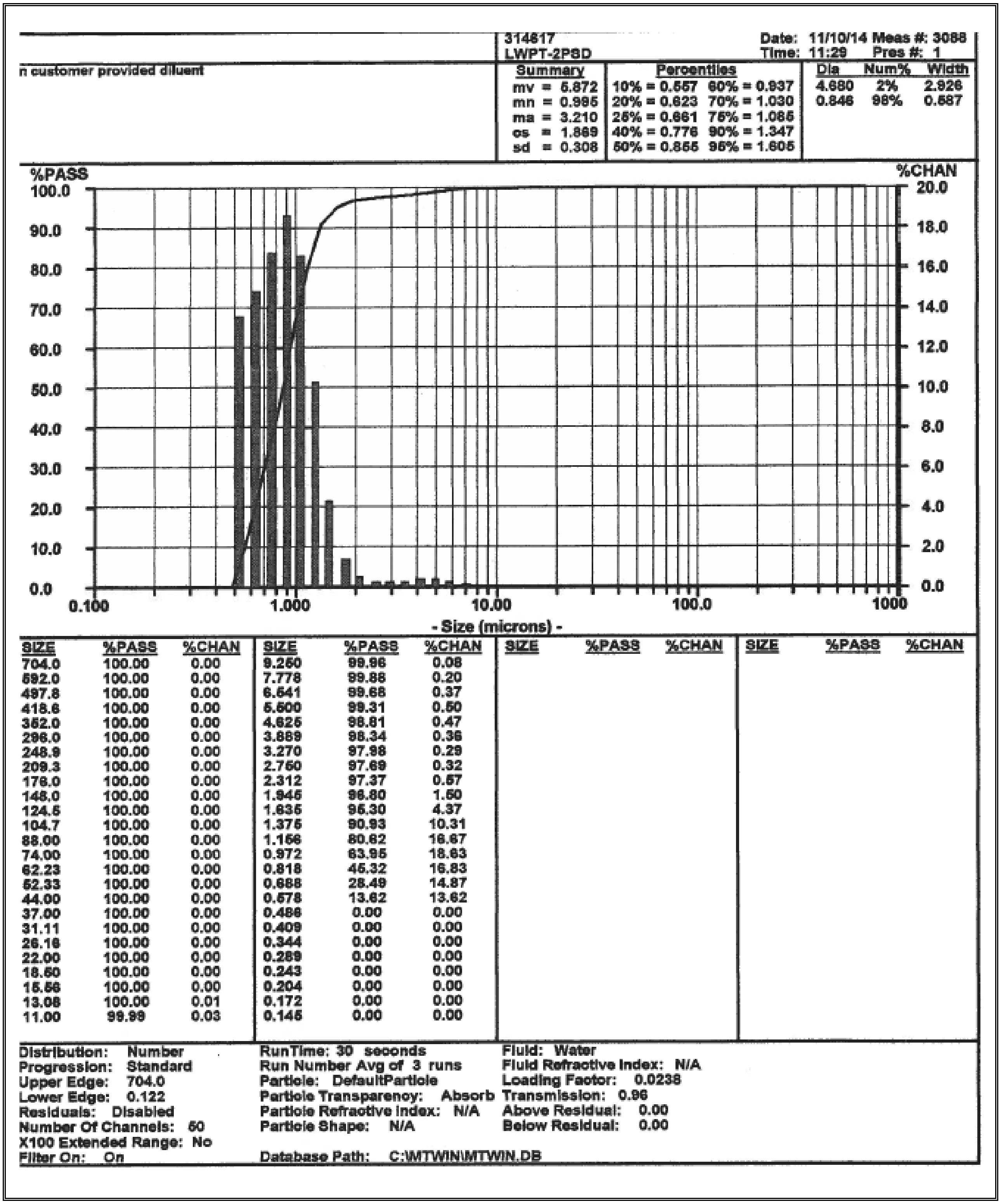
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0238
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Figure B-3. Second LWPT particle size volume distribution measured in salt simulant



314617
LWPT-2PSD

Date: 11/10/14 Meas #: 3088
Time: 11:29 Pres #: 1

n customer provided diluent

Summary
mv = 5.872
mn = 0.995
ma = 3.210
cs = 1.869
sd = 0.308

Percentiles
10% = 0.557 60% = 0.937
20% = 0.623 70% = 1.030
25% = 0.661 75% = 1.085
40% = 0.776 90% = 1.347
50% = 0.855 95% = 1.605

Dia	Num%	Width
4.680	2%	2.926
0.846	98%	0.587

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.96	0.08						
592.0	100.00	0.00	7.778	99.88	0.20						
497.8	100.00	0.00	6.541	99.68	0.37						
418.6	100.00	0.00	5.500	99.31	0.50						
352.0	100.00	0.00	4.625	98.81	0.47						
296.0	100.00	0.00	3.889	98.34	0.36						
248.9	100.00	0.00	3.270	97.98	0.29						
209.3	100.00	0.00	2.750	97.69	0.32						
176.0	100.00	0.00	2.312	97.37	0.57						
148.0	100.00	0.00	1.945	96.80	1.50						
124.5	100.00	0.00	1.635	95.30	4.37						
104.7	100.00	0.00	1.375	90.93	10.31						
88.00	100.00	0.00	1.156	80.62	16.67						
74.00	100.00	0.00	0.972	63.95	18.63						
62.23	100.00	0.00	0.818	45.32	16.83						
52.33	100.00	0.00	0.688	28.49	14.87						
44.00	100.00	0.00	0.578	13.62	13.62						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.03	0.145	0.00	0.00						

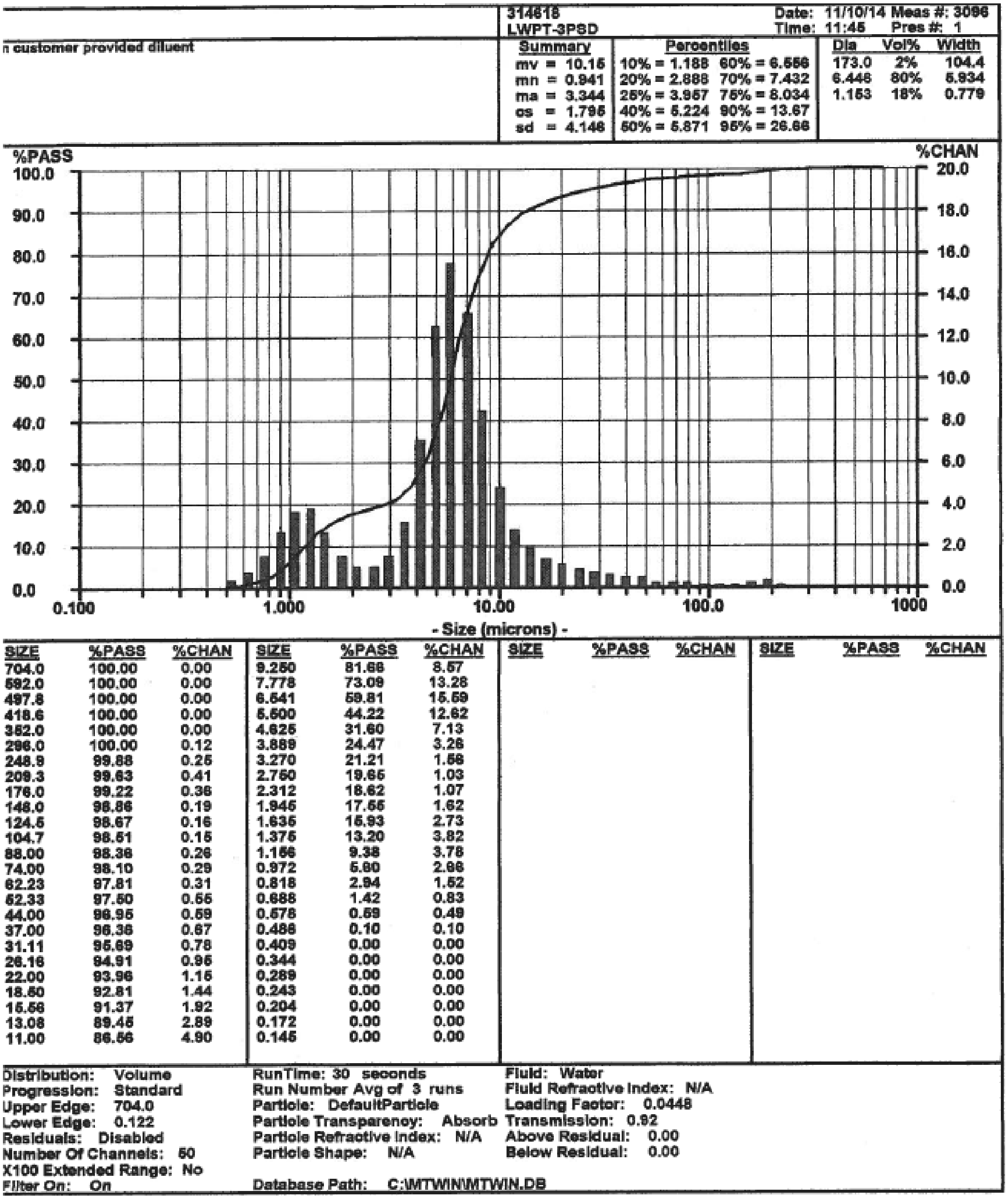
Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0238
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Figure B-4. Second LWPT particle size number distribution measured in salt simulant



314618
LWPT-3PSD

Date: 11/10/14 Meas #: 3096
Time: 11:45 Pres #: 1

n customer provided diluent

Summary
mv = 10.15
mn = 0.941
ma = 3.344
cs = 1.795
sd = 4.146

Percentiles	
10% = 1.188	60% = 6.556
20% = 2.888	70% = 7.432
25% = 3.957	75% = 8.034
40% = 5.224	90% = 13.67
50% = 5.871	95% = 26.66

Dia	Vol%	Width
173.0	2%	104.4
6.446	80%	5.934
1.153	18%	0.779

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	81.66	8.57						
592.0	100.00	0.00	7.778	73.09	13.28						
497.8	100.00	0.00	6.541	59.81	15.59						
418.6	100.00	0.00	5.500	44.22	12.62						
352.0	100.00	0.00	4.625	31.60	7.13						
296.0	100.00	0.12	3.889	24.47	3.26						
248.9	99.88	0.25	3.270	21.21	1.56						
209.3	99.63	0.41	2.750	19.65	1.03						
176.0	99.22	0.36	2.312	18.62	1.07						
148.0	98.86	0.19	1.945	17.55	1.62						
124.5	98.67	0.16	1.635	15.93	2.73						
104.7	98.51	0.15	1.375	13.20	3.82						
88.00	98.36	0.26	1.156	9.38	3.78						
74.00	98.10	0.29	0.972	5.60	2.66						
62.23	97.81	0.31	0.818	2.94	1.52						
52.33	97.50	0.55	0.688	1.42	0.83						
44.00	96.95	0.59	0.578	0.59	0.49						
37.00	96.36	0.67	0.486	0.10	0.10						
31.11	95.69	0.78	0.409	0.00	0.00						
26.16	94.91	0.95	0.344	0.00	0.00						
22.00	93.96	1.15	0.289	0.00	0.00						
18.50	92.81	1.44	0.243	0.00	0.00						
15.56	91.37	1.92	0.204	0.00	0.00						
13.08	89.45	2.89	0.172	0.00	0.00						
11.00	86.56	4.90	0.145	0.00	0.00						

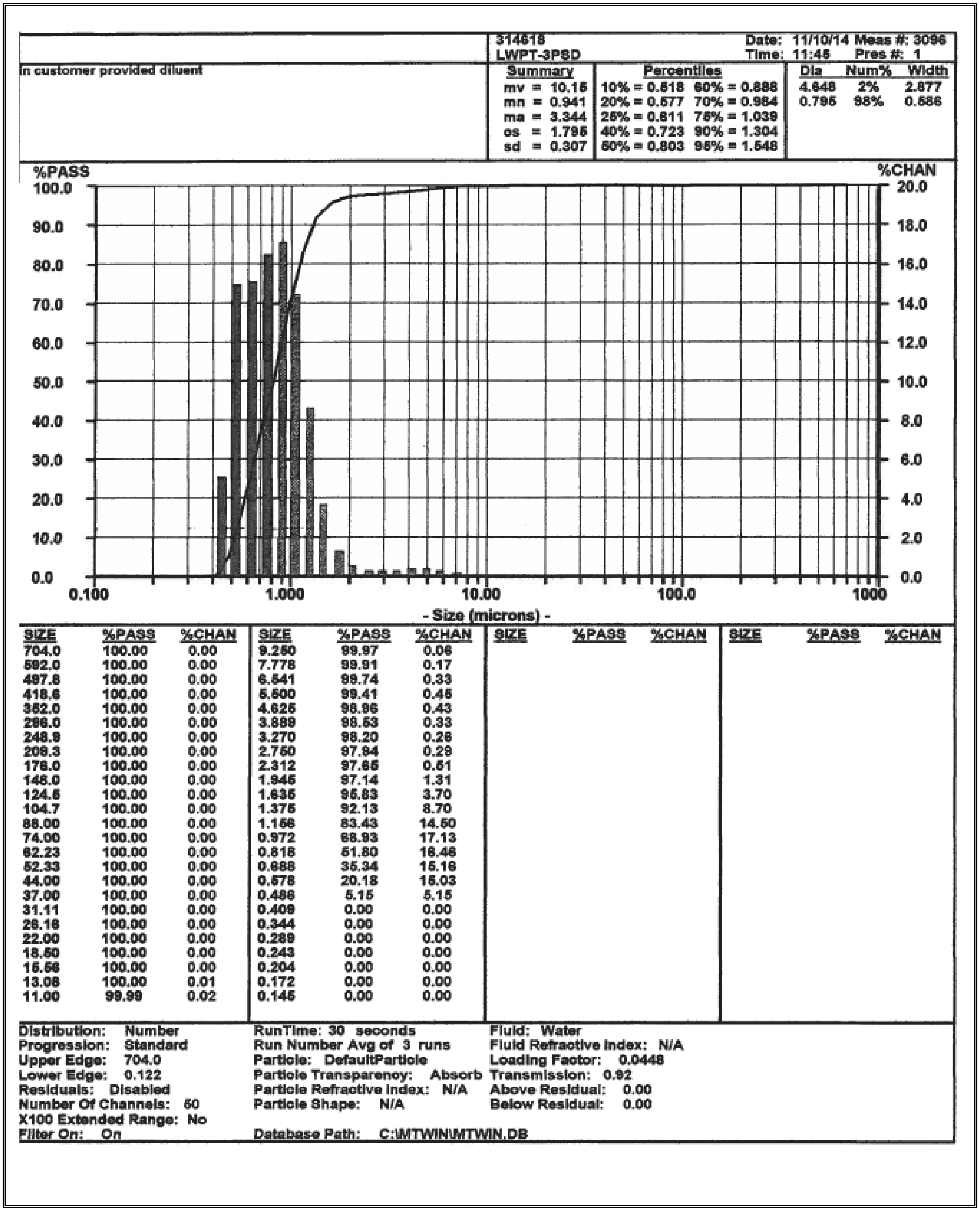
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0448
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

Figure B-5. Third LWPT particle size volume distribution measured in salt simulant



314618 Date: 11/10/14 Meas #: 3096
LWPT-3PSD Time: 11:45 Pres #: 1

in customer provided diluent

Summary	Percentiles		Dia	Num%	Width
mv = 10.15	10% = 0.518	60% = 0.888	4.648	2%	2.877
mn = 0.941	20% = 0.577	70% = 0.984	0.795	98%	0.586
ma = 3.344	25% = 0.611	75% = 1.039			
cs = 1.795	40% = 0.723	90% = 1.304			
sd = 0.307	50% = 0.803	95% = 1.548			

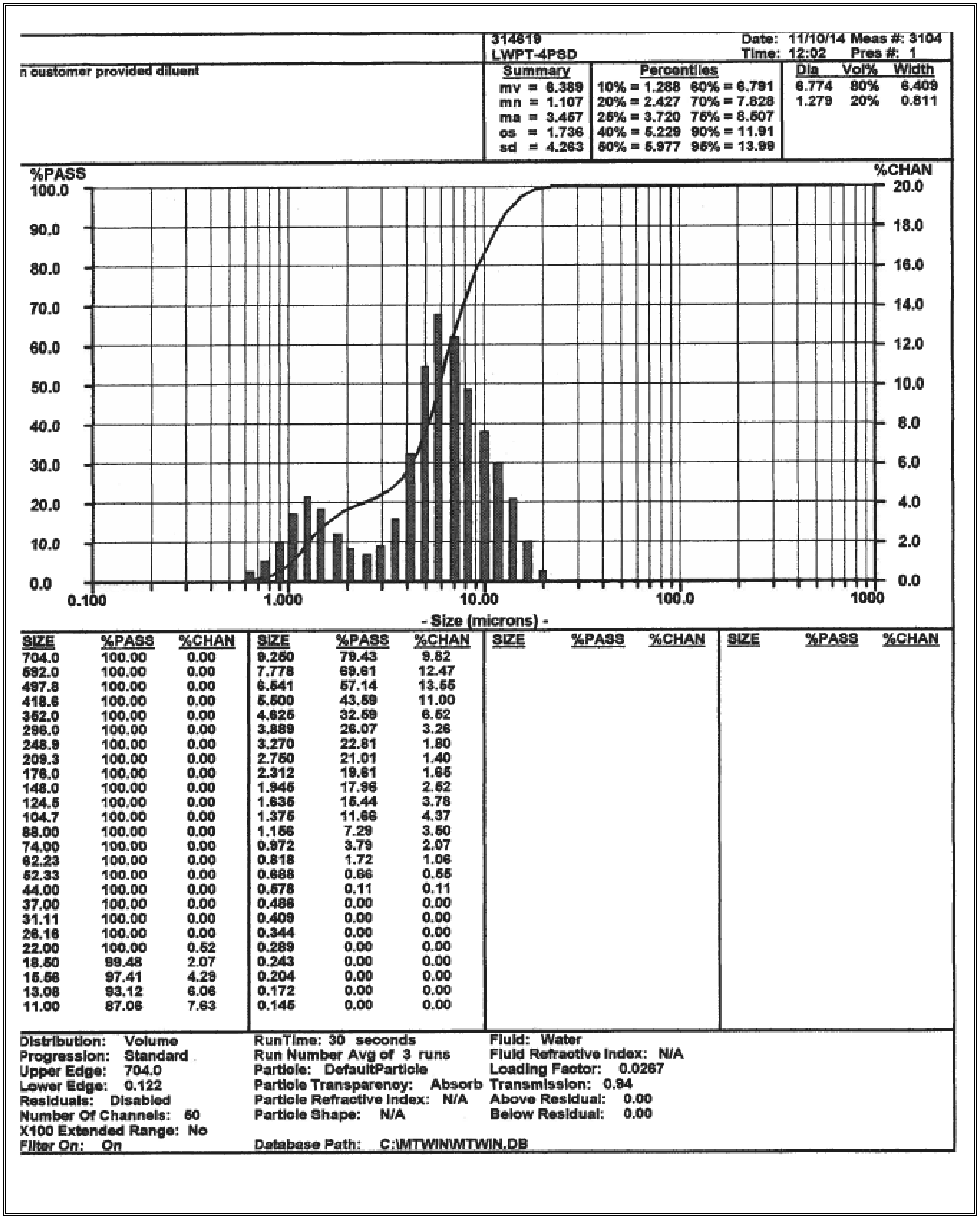
SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.97	0.06						
592.0	100.00	0.00	7.778	99.91	0.17						
497.8	100.00	0.00	6.541	99.74	0.33						
418.6	100.00	0.00	5.500	99.41	0.45						
352.0	100.00	0.00	4.625	98.96	0.43						
296.0	100.00	0.00	3.889	98.53	0.33						
248.9	100.00	0.00	3.270	98.20	0.26						
209.3	100.00	0.00	2.750	97.94	0.29						
176.0	100.00	0.00	2.312	97.65	0.51						
148.0	100.00	0.00	1.945	97.14	1.31						
124.5	100.00	0.00	1.635	95.83	3.70						
104.7	100.00	0.00	1.375	92.13	8.70						
88.00	100.00	0.00	1.156	83.43	14.50						
74.00	100.00	0.00	0.972	68.93	17.13						
62.23	100.00	0.00	0.818	51.80	16.46						
52.33	100.00	0.00	0.688	35.34	15.16						
44.00	100.00	0.00	0.578	20.18	15.03						
37.00	100.00	0.00	0.486	5.15	5.15						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.02	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0448
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

Figure B-6. Third LWPT particle size number distribution measured in salt simulant



n customer provided diluent

314619
LWPT-4PSD

Date: 11/10/14 Meas #: 3104
Time: 12:02 Pres #: 1

Summary	Percentiles	Percentiles	Dia	Vol%	Width
mv = 6.389	10% = 1.288	60% = 6.791	6.774	80%	6.409
mn = 1.107	20% = 2.427	70% = 7.828	1.279	20%	0.811
ma = 3.457	25% = 3.720	75% = 8.507			
cs = 1.736	40% = 5.229	90% = 11.91			
sd = 4.263	50% = 5.977	95% = 13.99			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	79.43	9.82
592.0	100.00	0.00	7.778	69.61	12.47
497.8	100.00	0.00	6.541	57.14	13.55
418.6	100.00	0.00	5.500	43.59	11.00
352.0	100.00	0.00	4.625	32.59	6.52
296.0	100.00	0.00	3.889	26.07	3.26
248.9	100.00	0.00	3.270	22.81	1.80
209.3	100.00	0.00	2.750	21.01	1.40
176.0	100.00	0.00	2.312	19.61	1.65
148.0	100.00	0.00	1.945	17.96	2.52
124.5	100.00	0.00	1.635	15.44	3.78
104.7	100.00	0.00	1.375	11.66	4.37
88.00	100.00	0.00	1.156	7.29	3.50
74.00	100.00	0.00	0.972	3.79	2.07
62.23	100.00	0.00	0.818	1.72	1.06
52.33	100.00	0.00	0.688	0.66	0.55
44.00	100.00	0.00	0.578	0.11	0.11
37.00	100.00	0.00	0.486	0.00	0.00
31.11	100.00	0.00	0.409	0.00	0.00
26.16	100.00	0.00	0.344	0.00	0.00
22.00	100.00	0.52	0.289	0.00	0.00
18.50	99.48	2.07	0.243	0.00	0.00
15.56	97.41	4.29	0.204	0.00	0.00
13.08	93.12	6.06	0.172	0.00	0.00
11.00	87.06	7.63	0.145	0.00	0.00

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN

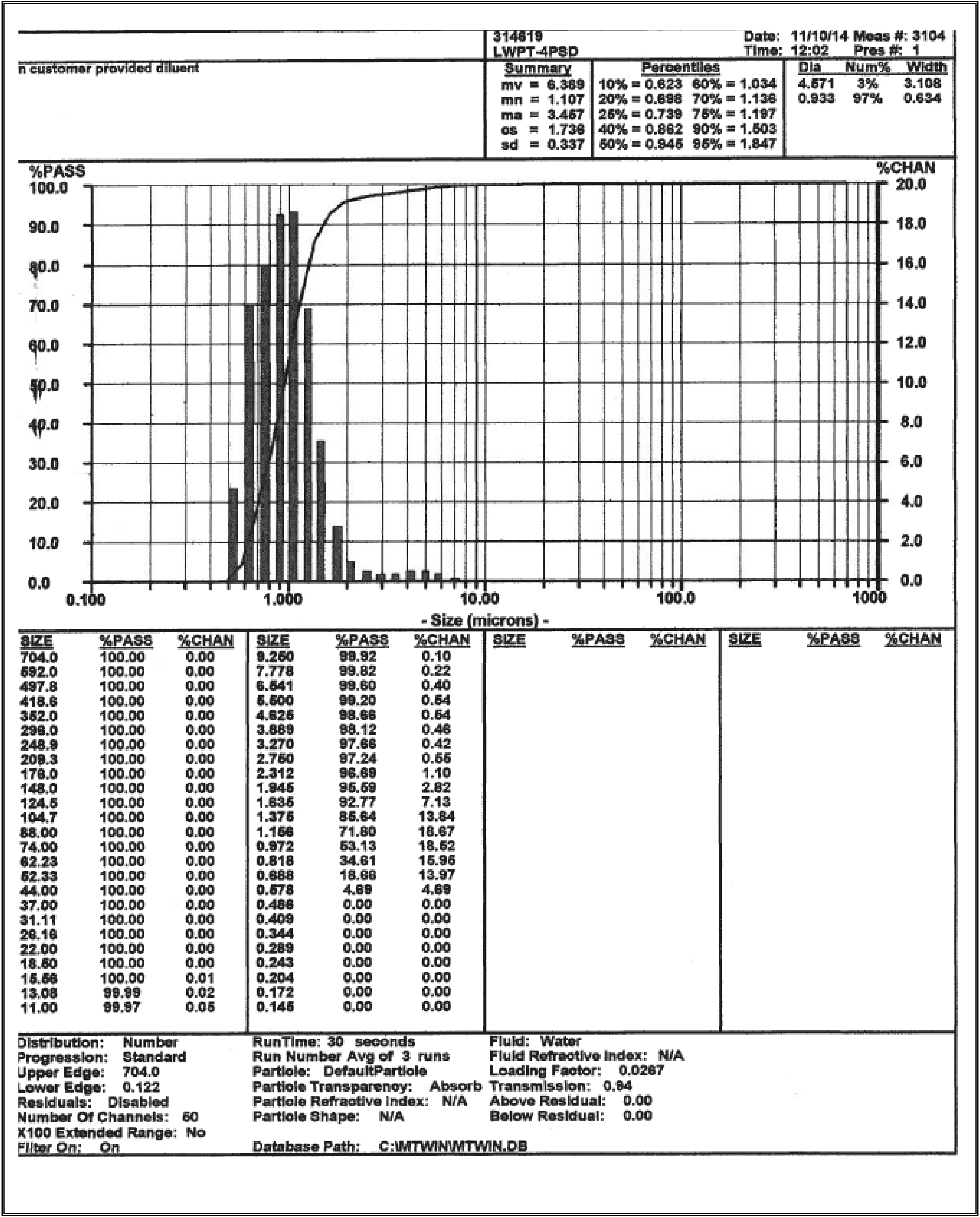
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0267
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Figure B-7. Fourth LWPT particle size volume distribution measured in salt simulant



314619
LWPT-4PSD

Date: 11/10/14 Meas #: 3104
Time: 12:02 Pres #: 1

n customer provided diluent

Summary	Percentiles		Dia	Num%	Width
mv = 6.389	10% = 0.623	60% = 1.034	4.571	3%	3.108
mn = 1.107	20% = 0.698	70% = 1.136	0.933	97%	0.634
ma = 3.457	25% = 0.739	75% = 1.197			
cs = 1.736	40% = 0.862	90% = 1.503			
sd = 0.337	50% = 0.946	95% = 1.847			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.92	0.10						
592.0	100.00	0.00	7.778	99.82	0.22						
497.8	100.00	0.00	6.541	99.60	0.40						
418.6	100.00	0.00	5.500	99.20	0.54						
352.0	100.00	0.00	4.625	98.66	0.54						
296.0	100.00	0.00	3.889	98.12	0.46						
248.9	100.00	0.00	3.270	97.66	0.42						
209.3	100.00	0.00	2.750	97.24	0.55						
176.0	100.00	0.00	2.312	96.69	1.10						
148.0	100.00	0.00	1.945	95.59	2.82						
124.5	100.00	0.00	1.635	92.77	7.13						
104.7	100.00	0.00	1.375	85.64	13.84						
88.00	100.00	0.00	1.156	71.80	18.67						
74.00	100.00	0.00	0.972	53.13	18.52						
62.23	100.00	0.00	0.818	34.61	15.95						
52.33	100.00	0.00	0.688	18.66	13.97						
44.00	100.00	0.00	0.578	4.69	4.69						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.01	0.204	0.00	0.00						
13.08	99.99	0.02	0.172	0.00	0.00						
11.00	99.97	0.05	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.122
Residuals: Disabled
Number Of Channels: 50
X100 Extended Range: No
Filter On: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: DefaultParticle
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\MTWIN.DB

Fluid: Water
Fluid Refractive Index: N/A
Loading Factor: 0.0267
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Figure B- 8. Fourth LWPT particle size number distribution measured in salt simulant

Appendix C. Microscopy Data

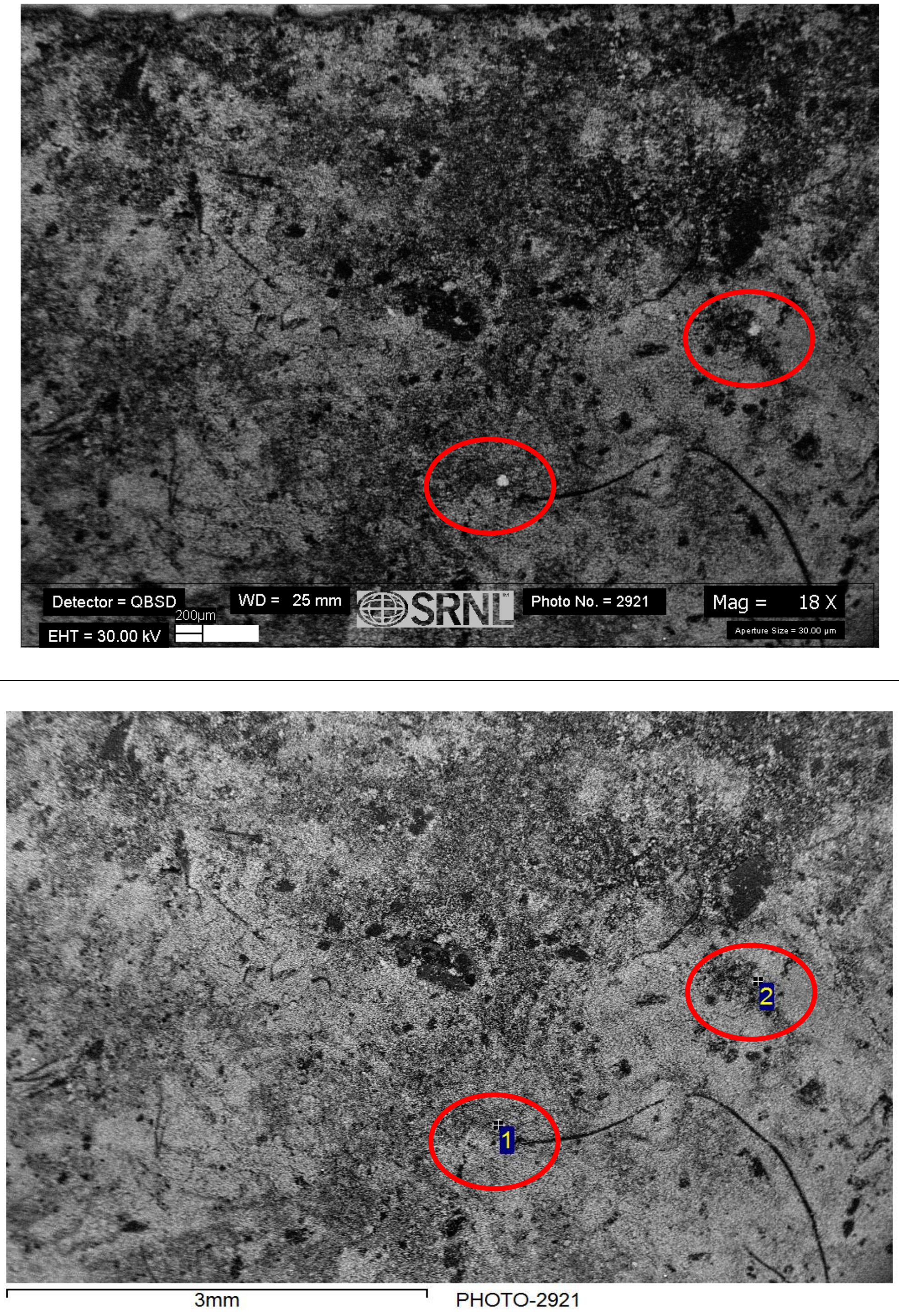


Figure C- 1. SEM image of first LWPT sample with 18 X magnification indicating various spots analyzed by EDS

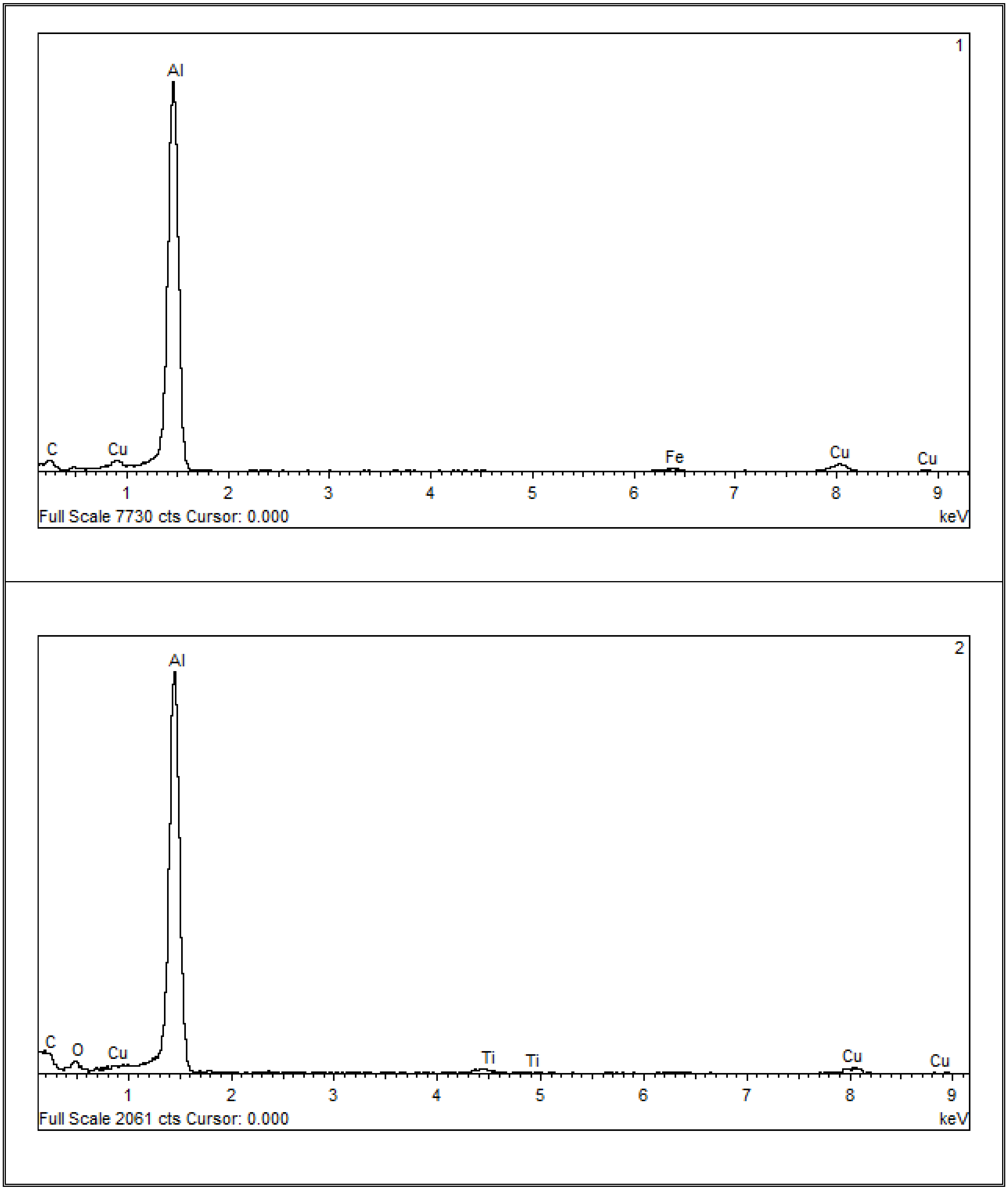


Figure C- 2. EDS spectrum from first LWPT sample at 18 X magnification showing primarily Al elemental peaks associated with spots 1 and 2

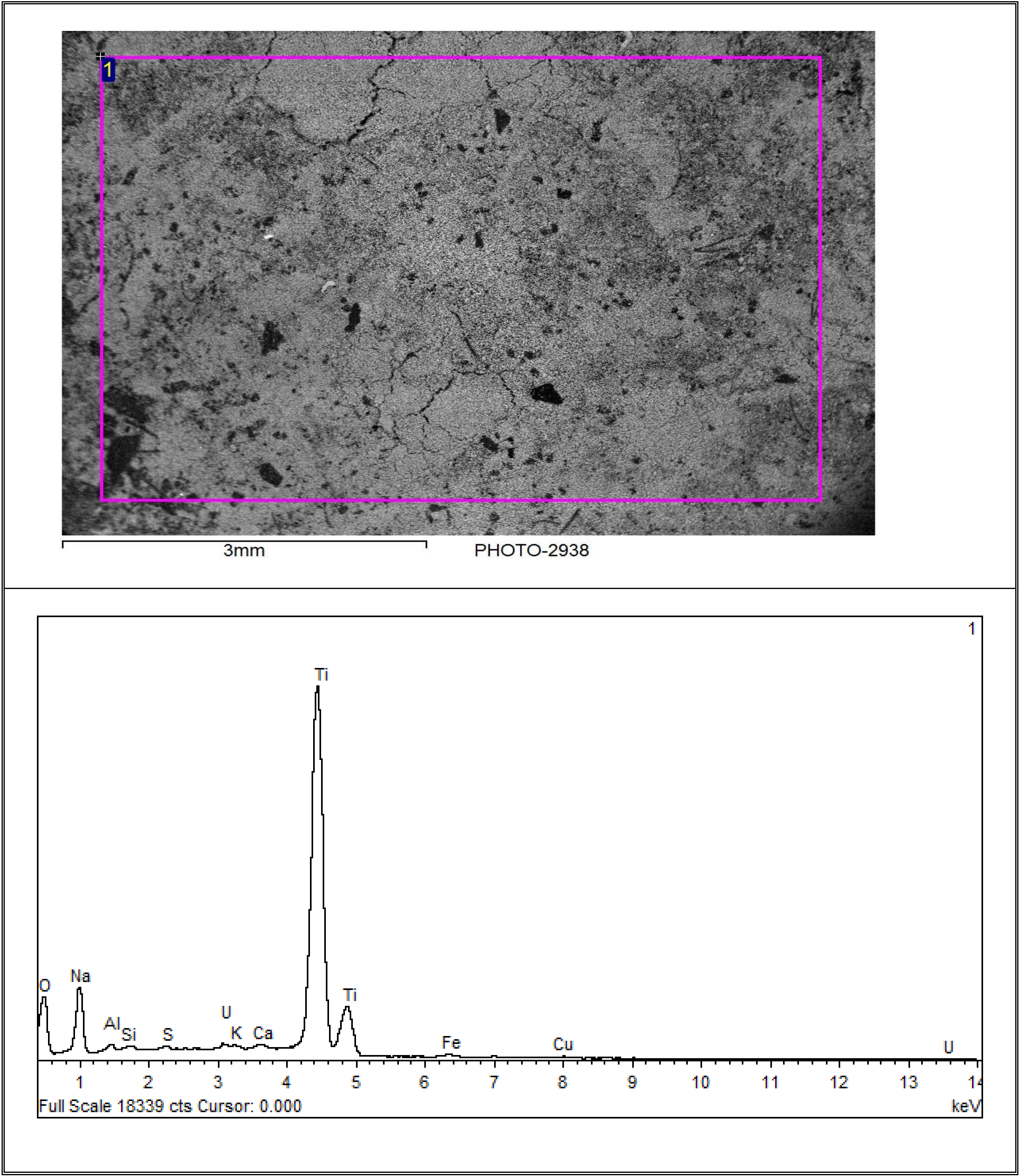


Figure C- 3. SEM image and EDS spectrum from first LWPT sample showing primarily Ti and Na elemental peaks associated with the average area within the box 1

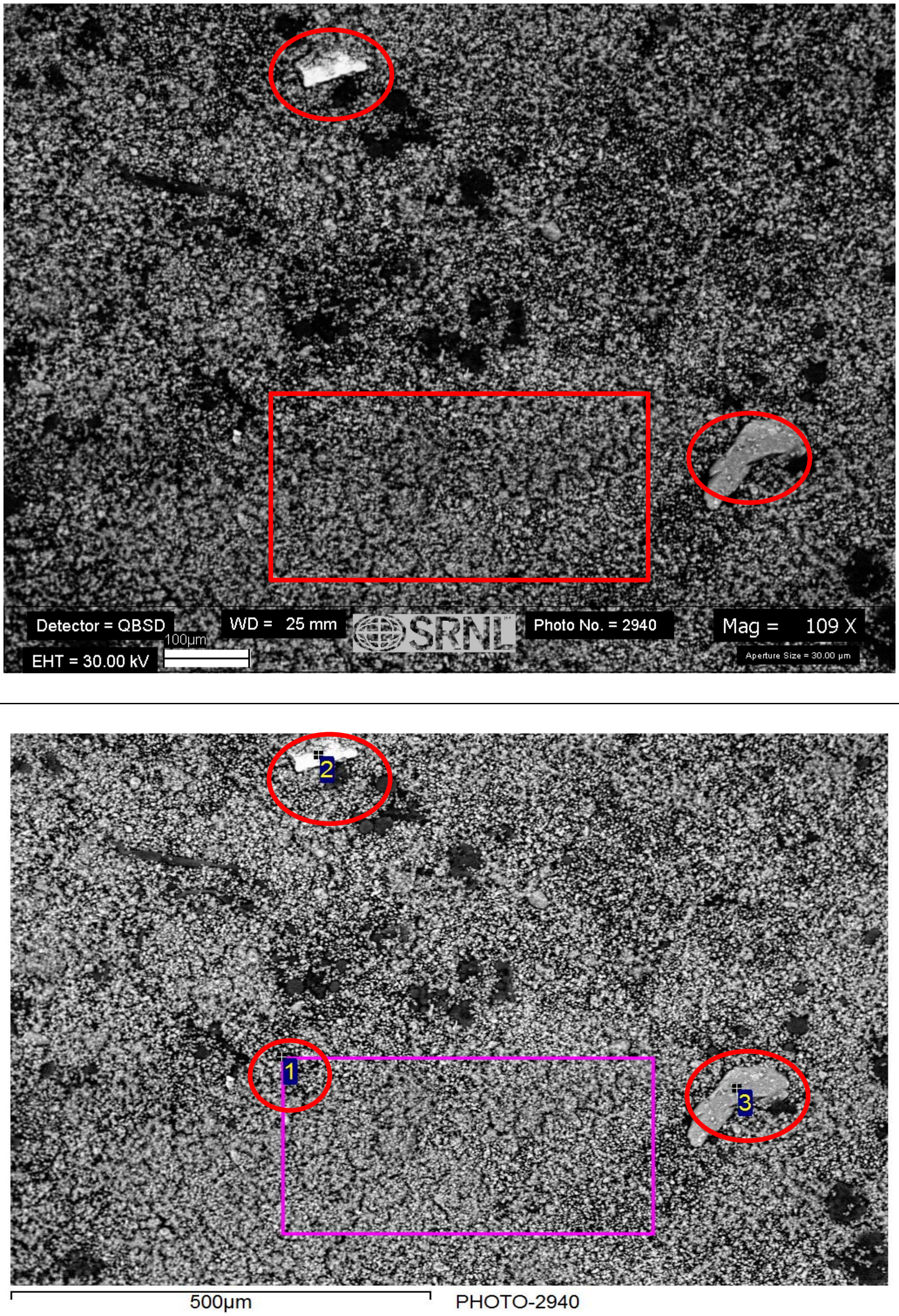


Figure C- 4. SEM image of first LWPT sample at 109 X magnification indicating various regions (box 1 and spots 2 and 3) analyzed by EDS

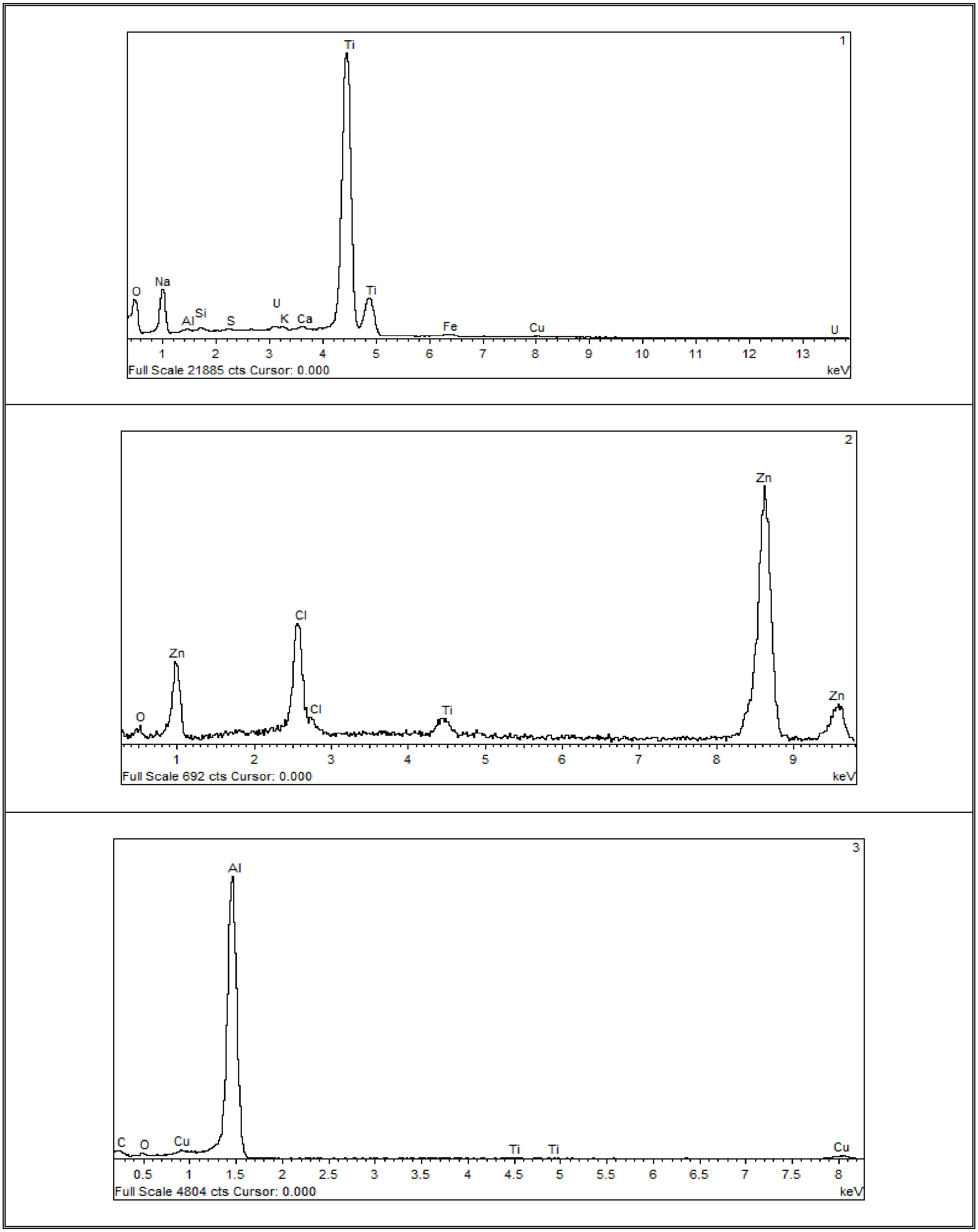


Figure C- 5. EDS spectrum from first LWPT sample at 109 X magnification showing elemental peaks associated with box 1 (primarily Na and Ti), spot 2 (Zn and Cl) and spot 3 for Al

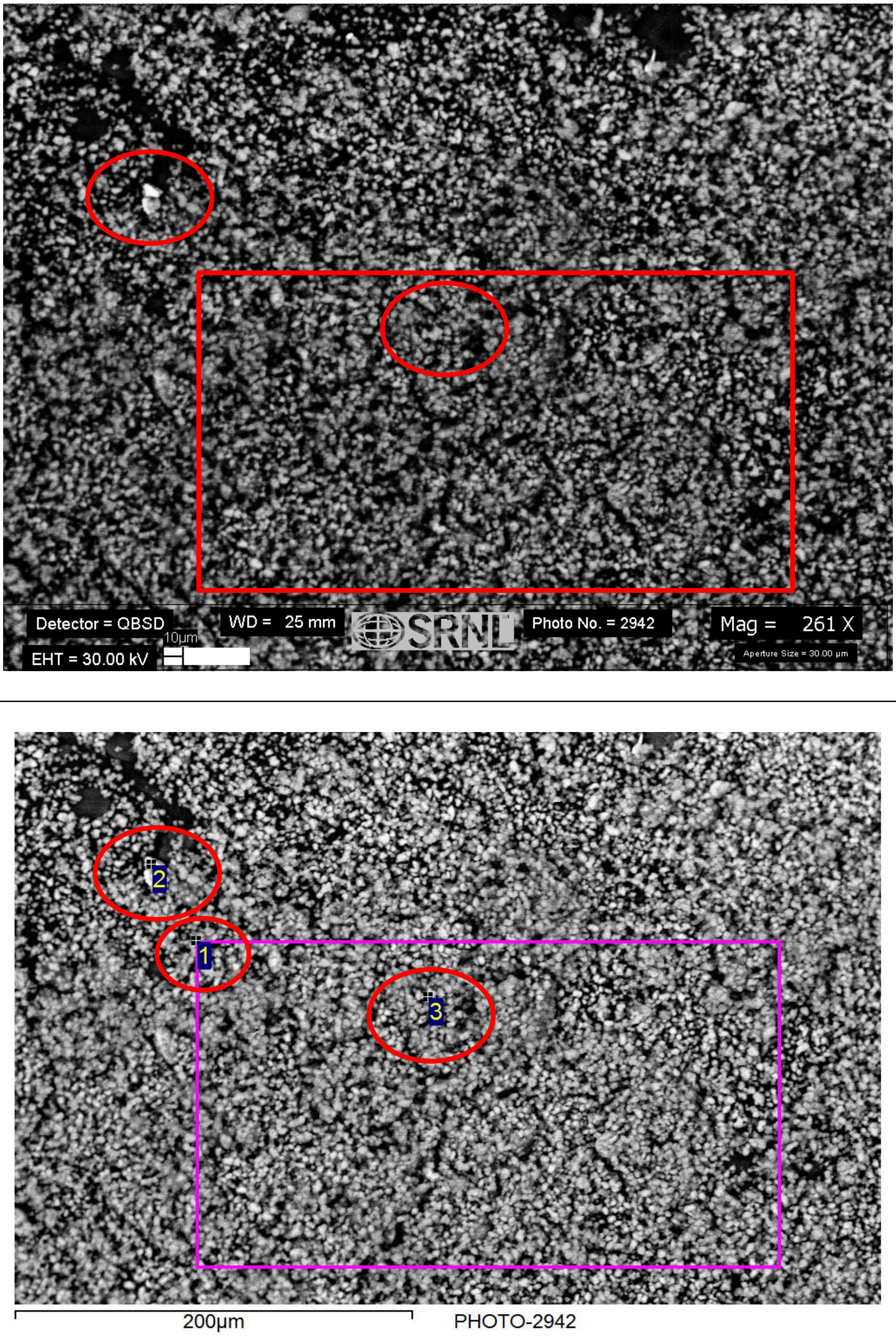


Figure C- 6. SEM image of first LWPT sample at 261 X magnification indicating various regions (box 1 and spots 2 and 3) analyzed by EDS

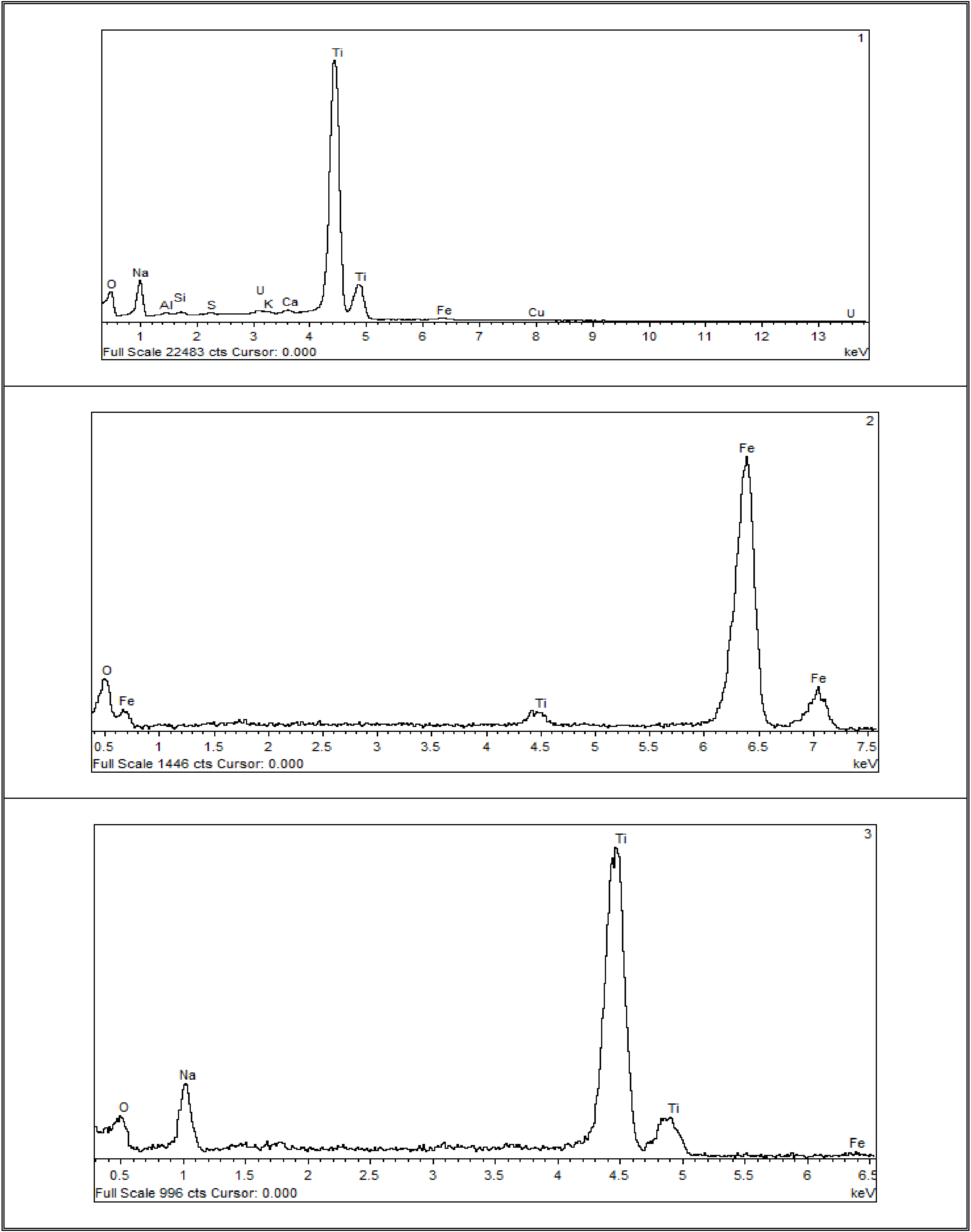


Figure C- 7. EDS spectrum from first LWPT sample at 261 X magnification showing elemental peaks associated with box 1 (primarily Ti and Na), spot 2 containing Fe and spot 3 with Ti and Na

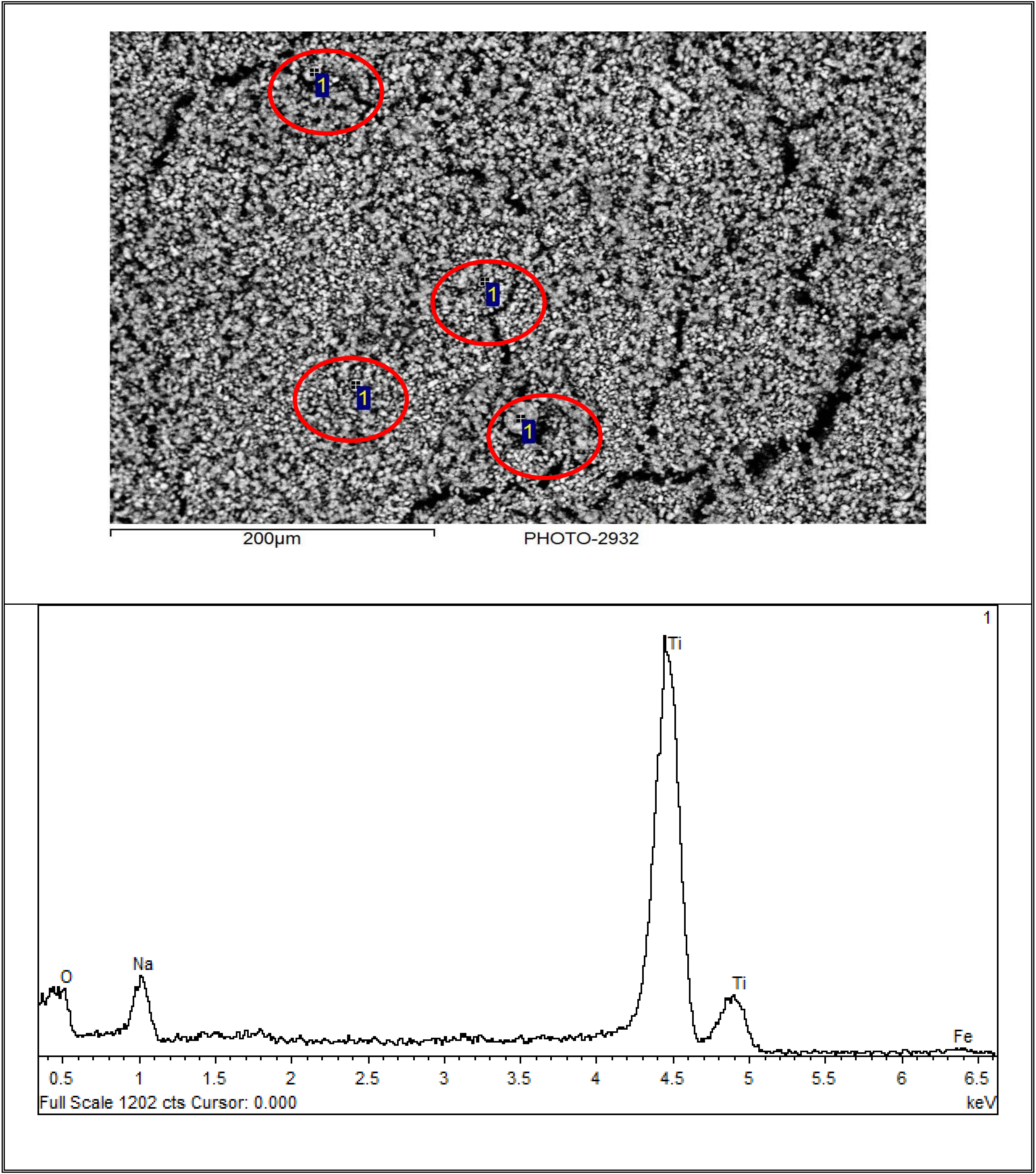


Figure C- 8. SEM image of second LWPT sample at 226 X magnification indicating various spots analyzed by EDS that are shown to be Ti and Na

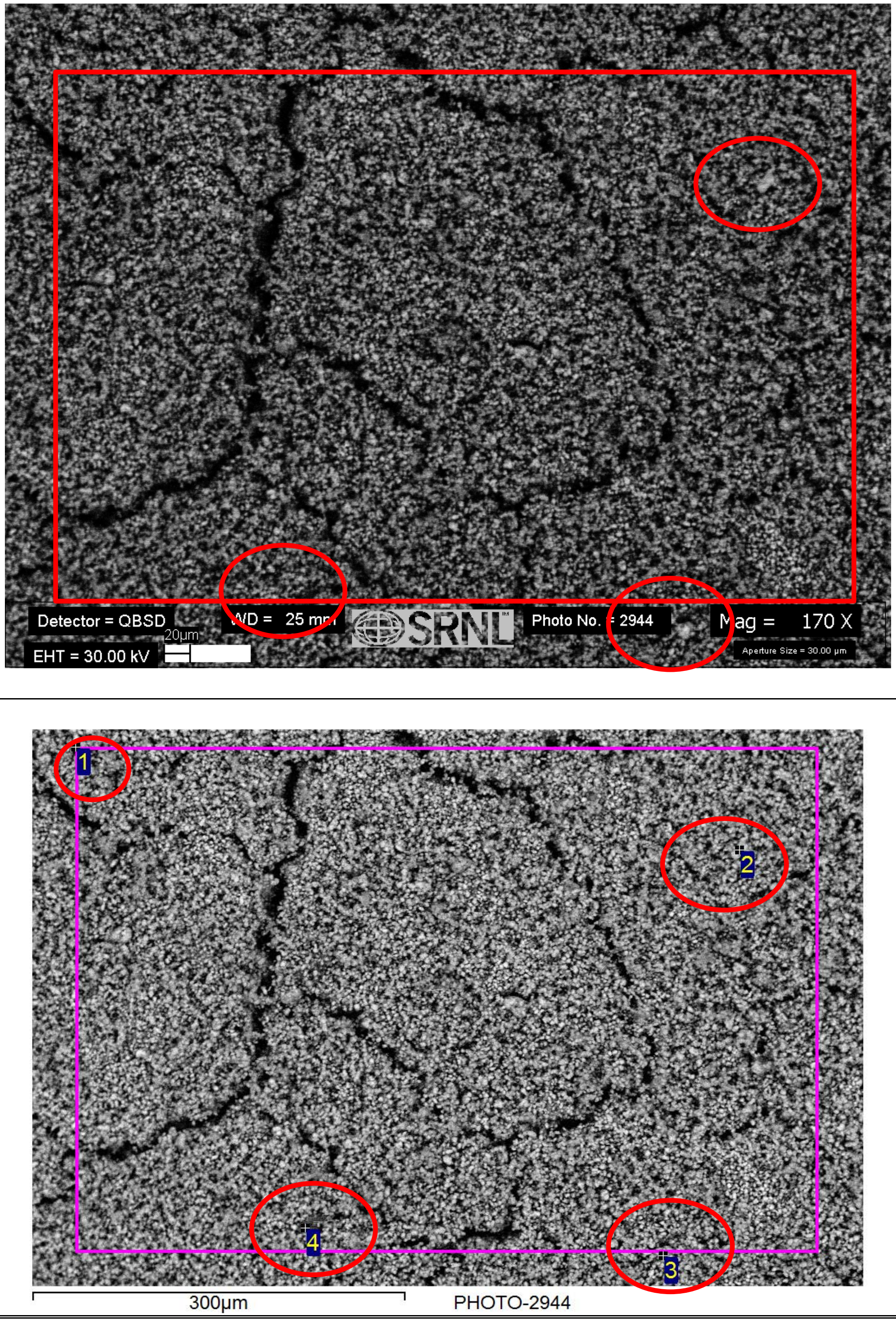


Figure C- 9. SEM image of second LWPT sample at 170 X magnification indicating various regions (box 1 and spots 2, 3 and 4) analyzed by EDS

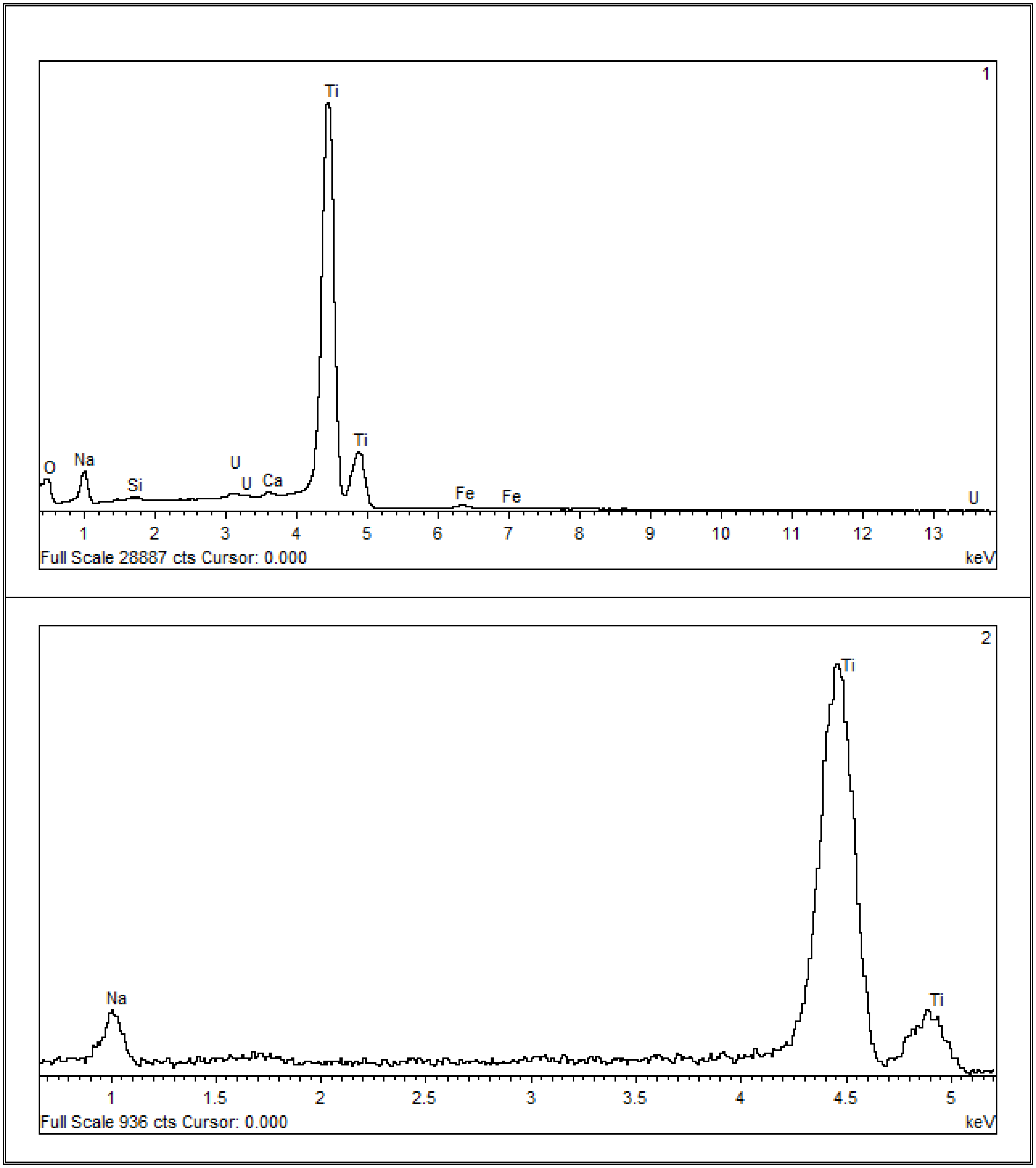


Figure C- 10. EDS spectrum from second LWPT sample at 170 X magnification showing elemental peaks associated with box 1 and spot 2 that contain primarily Ti and Na

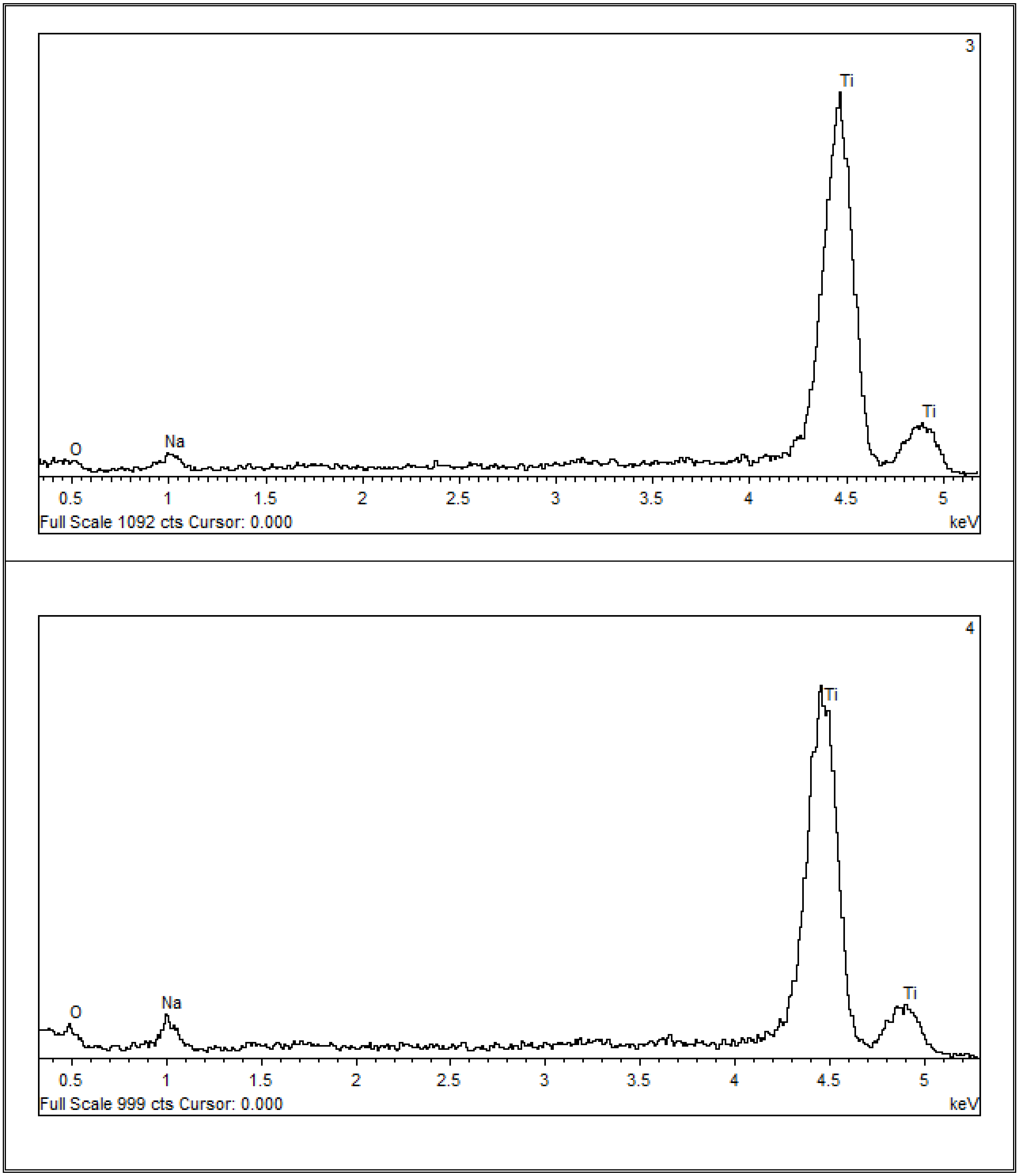


Figure C- 11. EDS spectrum from second LWPT sample at 170 X magnification showing elemental peaks associated with spots 3 and 4 that contain primarily Ti and Na

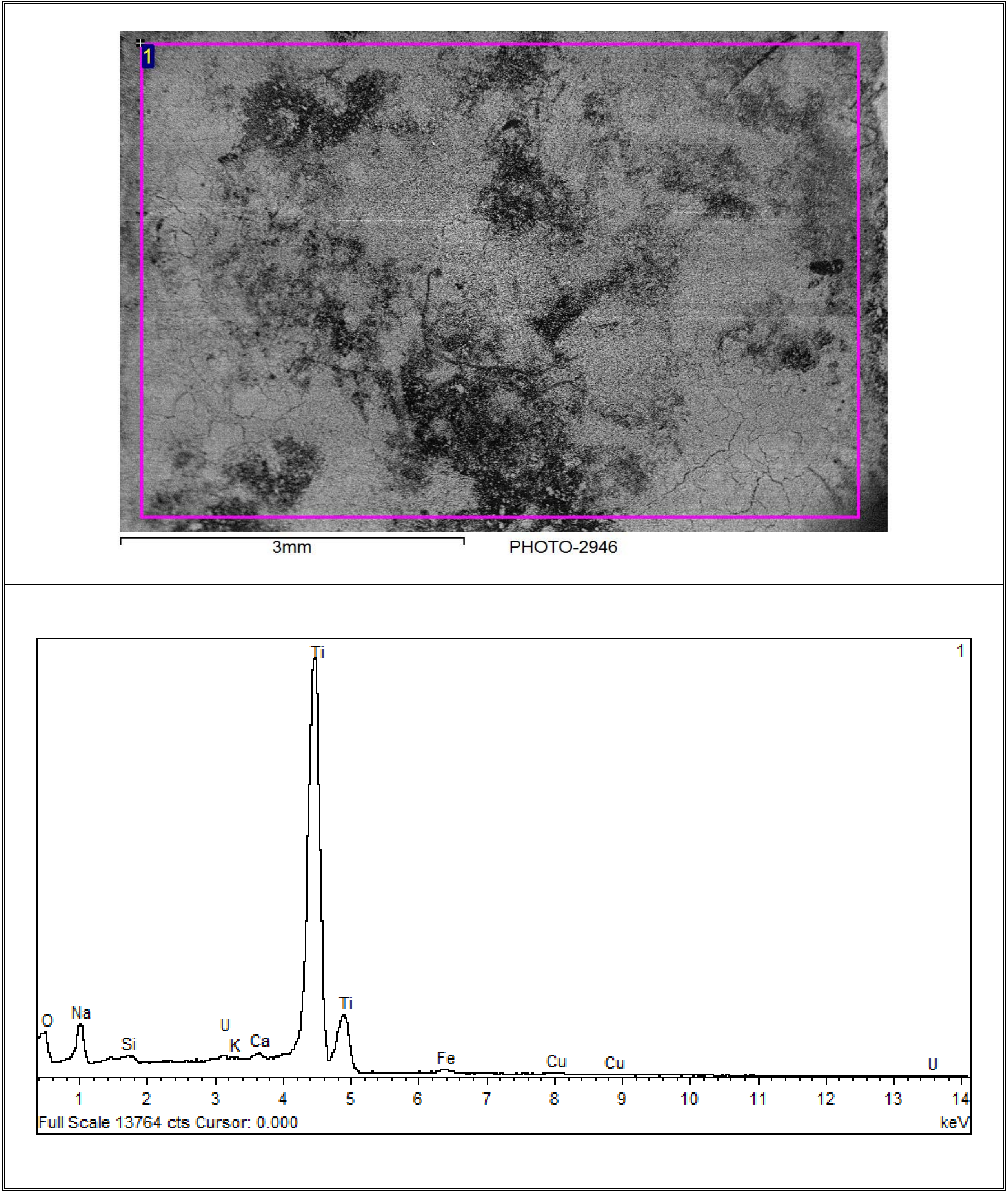


Figure C- 12. SEM image of second LWPT sample at 17 X magnification indicating box analyzed by EDS that shows primarily Ti and Na

Distribution:

S. L. Marra, 773-A
T. B. Brown, 773-A
D. H. McGuire, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
C. J. Martino, 999-W
T. B. Peters, 773-42A
M. R. Poirier, 773-42A
S. D. Fink, 773-A
C. A. Nash, 773-42A
Records Administration (EDWS)

J. M. Bricker, 704-30S
J. S. Contardi, 704-56H
H. H. Elder, 704-27S
T. L. Fellinger, 766-H
E. J. Freed, 704-S
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. W. Holtzscheiter, 766-H
J. F. Iaukea, 704-27S
D. K. Peeler, 999-W
J. W. Ray, 704-27S
P. J. Ryan, 704-S
A. Samadi-Dezfouli, 704-27S
H. B. Shah, 766-H
D. C. Sherburne, 704-S
M. E. Stone, 999-W

E. A. Brass, 241-12H
C. K. Chiu, 704-27S
E. J. Freed, 704-S
A. G. Garrison, 241-12H
B. A. Gifford, 704-56H
D. J. Martin, 241-152H
R. T. McNew, 241-152H
M. A. Rios-Armstrong, 766-H
A. R. Shafer, 704-27S
T. E. Smith,241-52H

P. R. Jackson, DOE-SR, 703-46A
J. A. Crenshaw, 703-46A