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Revision 0

Identification and Characterization of the Solids Found in Extraction Contactor SEP-401 in June 2012

F. F. Fondeur
S. D. Fink

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Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

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REVIEWS AND APPROVALS

AUTHORS:

F. F. Fondeur, E&CPT Date

TECHNICAL REVIEW:

T. B. Peters, E&CPT Date

APPROVAL:

S. D. Fink, Manager Date
Environmental & Chemical Process Technology Research Program

S. L. Marra, Manager Date
Environmental & Chemical Process Technology Research Programs

D. J. Martin, Manager Date
SRR Process Engineering

EXECUTIVE SUMMARY

The Modular Caustic-Side Solvent Extraction Unit (MCU) recently conducted an outage that included maintenance on the centrifugal contactors. Operations personnel observed solids or deposits in two contactors and attempted to collect samples for analyses by Savannah River National Laboratory (SRNL).

The residues found in Extraction Contactor SEP-401 are a mixture of amorphous silica, aluminosilicate, titanium, and debris from low alloy steel. The solids contain low concentrations of plutonium and strontium. These isotopes are associated with the titanium that came from the monosodium titanate (MST) added in the Actinide Removal Process (ARP) most likely as leached Ti from the MST that precipitated subsequently in MCU.

An attempt was also made to obtain samples from the contents of Wash Contactor SEP-702. However, sampling provide ineffective.

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LIST OF ABBREVIATIONS

ARG	Analytical Reference Glass
ARP	Actinide Removal Process
CSSX	Caustic Side Solvent Extraction
FTIR	Fourier-transformed Infrared Spectroscopy
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
MST	monosodium titanate (Mw =199.7 g)
PuTTA	plutonium thenoyltrifluoroacetone
RCRA	Resource Recovery Conservation Act
%RSD	percentage residual standard deviation
SEM-EDS	scanning electron microscopy with energy dispersive spectroscopy
SRNL	Savannah River National Laboratory
SSFT	Salt Solution Feed Tank
SVOA	Semivolatile Analysis
SWPF	Salt Waste Processing Facility
XRD	X-ray diffraction

1.0 Introduction

Since March 2008, the ARP-MCU facility has successfully processed nearly three million gallons of supernate. The process has experienced a few outages arising from plugged coalescers. Recently, the facility began an outage to perform maintenance activities aimed at extending the original life cycle for the facility in light of delays in the construction of the Salt Waste Processing Facility (SWPF).

The outage included cleaning and maintenance of the centrifugal contactors. Inspection of the extraction centrifuges revealed the presence of accumulated solids in the bottom channels of Extraction Contactor SEP-401. SEP-401, the first centrifuge the supernate enters the solvent extraction process, is where radioactive supernate from ARP meets solvent scrubbed with 0.05 M nitric acid. The aqueous carry over from the solvent mixes with the supernate possibly resulting in a lowering of its pH to the point of solid precipitation.

Any large variance in the concentration of the aluminum and silicon concentrations, elements known to readily precipitate throughout the tank farm, may indicate precipitation driven by a mild pH swing. A similar deposit discovery (sodium aluminosilicate) was also found on September 2010 at the Salt Solution Feed Tank.[1]

Another possible way for solids to get into SEP-401 is the transfer of a colloidal suspension by pumps 102A and 102B from the Salt Solution Feed Tank. The colloidal suspension may then aggregate at and then settle in SEP 401. Regardless of the mechanism, solid formation and settling is detrimental to flow in small clearances and to moving parts within the contactors.

In June of 2012, a sample of the solids found in SEP-401 was sent to the laboratory for characterization and identification.[2] A similar attempt was made to collect a sample of a minor deposit visually observed in Wash Contactor SEP-702.[3] Unfortunately, that sampling evolution failed to provide a measurable sample. This memo summarizes the analytical analysis and reports the identification of the solids.

2.0 Experimental Procedure

Using a glass pipette, personnel carefully removed the liquid entrained inside the sample bag MCU-12-262, containing the material collected from SEP-401. The amount of liquid removed (1.5 mL) limited the analysis performed. A portion of the liquid was analyzed by Fourier-transformed Infrared Spectroscopy (FTIR). The remaining liquid was analyzed by SemiVolatile Analysis (SVOA) to determine Isopar® L, Modifier[4] (or Cs-7SB), and other organic compounds.

A portion of the solids was digested by both aqua regia (25 vol% HNO_3 and 75 vol % HCl) and blended with sodium peroxide to dissolve sodium, silicon, and other elements. The digested liquid was then analyzed by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma - Mass Spectroscopy (ICP-MS), gamma counting (with and without ^{137}Cs), and analysis following extraction with plutonium thenoyltrifluoroacetone (PuTTA). The remaining solid portion was analyzed by scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), X-ray diffraction (XRD), and FTIR.

3.0 Results and Discussion

A picture of the sample received by SRNL is shown in Figure 1. As shown in Fig. 1, a 250 mL glass bottle contained approximately 23 grams of wetted solids. The solids appeared as aggregates of smaller particles compacted together. The granular nature of the solids indicates

the particles formed and aggregated in solution before settling to the bottom of the centrifuge. The largest agglomerate measured approximately five millimeters across. Also shown in Fig.1 is an entrained yellow liquid that was retrieved with a glass pipette. That liquid had a pH value of 1 (as expected since the extractors were cleaned with 3 M nitric acid).

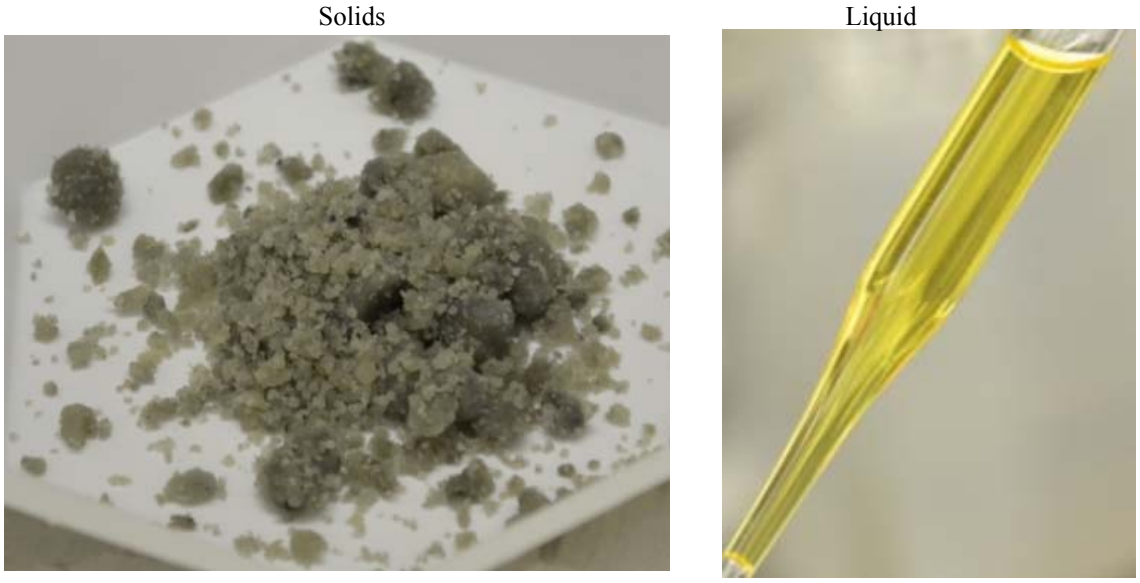

Figure 1. Physical appearance of sample MCU-12-262 obtained from SEP-401.

Approximately 23 g of wet solid was recovered. The liquid sample (approximately 1.5 mL) was found between the primary (that held the glass container) and secondary plastic bags. The liquid has a pH of 1.

3.1 Liquid Analyses

As shown in Table 1, the results from the SVOA analysis indicate that the liquid contained nitrated aromatic compounds. The nitro group in these compounds may have originated from the acid cleaning of the contactors coming in contact with residual organic compounds from the tank's supernate. No Isopar® L or Modifier (Cs-7SB) solvent component was found in this liquid. The diisooctyl adipate is a plasticizer used in the plastic bag for transporting the sample. Some of these compounds may have originated from the release agents used to make the plastic bag which usually contains diesel and petroleum products.

Table 1. Chemical composition of the liquid sample found in the sample container. The diisooctyl adipate likely comes from the plastic bag (since it is a common plasticizer).

Sample ID 300299978 **Status** COMPLETE **Condition** APF

LIMS Method	Description	Result (one sigma % unc)	Units
SVOA (B123)	5-Formylsalicylaldehyde	6.1 (20)	mg/L
SVOA (B123)	Biphenyl	2.2 (20)	mg/L
SVOA (B123)	4-Hydroxy-3-nitrobenzaldehyde	30 (20)	mg/L
SVOA (B123)	2-hydroxy-5-nitrobenzaldehyde	3.1 (20)	mg/L
SVOA (B123)	2 4-dinitrophenol	12 (20)	mg/L
SVOA (B123)	2 5-Dimethylbenzoxazole	23 (20)	mg/L
SVOA (B123)	Diisooctyl adipate	56 (20)	mg/L
SVOA (B123)	METHOD DETECTION LIMIT	1	mg/L
SVOA (B123)	EXPERIMENTAL NOTE	Samples were analyzed by GC/MS and dichloromethane was used for extractions.	TEXT

As shown in Fig. 2, the FTIR analysis reveals the liquid is mostly water containing a suspension of silica nuclei as noted by peak at 993 cm^{-1}. This finding confirms the occurrence of secondary nucleation (i.e., precipitation on suspended solids) in this liquid. A small signal was observed at 3100, 2920, and 2875 cm^{-1} typically associated with aromatic and aliphatic C-H stretch. No signal that can be attributed to the Modifier or the extractant (BOBCalixC6) was observed.

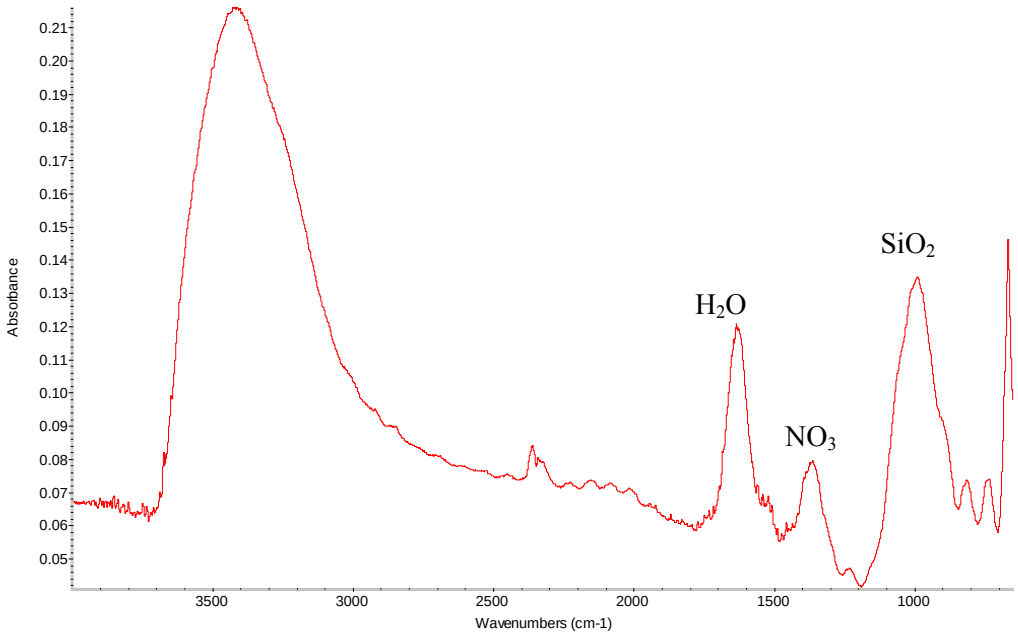

Figure 2. FTIR spectrum of the liquid found in sample from SEP-401

3.2 Digestion analysis of the solids form MCU-12-262

Table 2 shows the chemical composition, as determined by ICP-AES, of the solids digested by aqua regia and by nitric acid dissolution following sample fusion with sodium peroxide.

Additional elements detected in lesser amounts are shown in Appendix A. The digestion method's efficiency and spectroscopic analysis accuracy was evaluated by digesting Analytical Reference Glass (ARG) whose composition is well documented.

As can be seen from Table 2, the solids are composed of mostly silica (as indicated by the large mass ratio of silicon to aluminum) and aluminosilicate. There are also elements from steel alloy debris such as iron, manganese, and chromium. Also found is titanium. Titanium enriched deposits has also been seen on spent coalescers and filters at MCU.[5] The absence of titanium alloys in the separators and other structural units used at MCU, rules them out as a titanium source. Similarly, the zirconium observed in Table 2 is probably analysis contamination. The detection of strontium is probably due to fines from ARP or sorption on precipitated hydrous titanium dioxide of soluble titanium from ARP.

Table 2. Summary of the elements detected above detection limits in the ICP-AES

Component	Digested with Alkaline Fusion and nitric acid (%RSD) in $\mu g/g_{wet\ sample}$	Digested with Aqua Regia (%RSD) in $\mu g/g_{wet\ sample}$	Possible Source
Al	5450 (10.2)	5180 (10)	Salt Solution
Ca	1270 (10)	97.5 (10)	Salt Solution
Cr	292 (12)	249 (10)	Steel Alloy
Fe	1340 (10)	1300 (10)	Steel Alloy
Mg	33.5 (10)	44.2 (10)	Salt Solution/ Steel Alloy
Mn	70.3 (10.4)	28.3 (10)	Steel Alloy
Na	Not Measured	5890 (18.4)	Salt Solution
Ni	162 (10.8)	< 792	Steel Alloy
Si	86,100 (10)	Not Measured	Salt Solution
Sn	< 2.94	180 (11.1)	Not Determined
Sr	< 12	1.14 (10)	Titania from ARP
Ti	2460 (10)	2640 (10)	Titania from ARP/Titanium Alloy
Zn	15.4 (10.5)	< 220	Analysis Contamination
Zr	8.0 (10.7)	Not Measured	Analysis Contamination

Aqua Regia is 75 vol % HCl and 25 vol % HNO_3

A further consideration of the aluminum and silicon concentration shown in Table 2 indicates that MCU-12-262 has excess silicon. Table 3 compares the aluminum to silicon ratio of MCU-12-262 to that of the macrobatches or from the tank components that makes up the macrobatches. None of the macrobatches had an aluminum to silicon ratio similar to MCU-12-262 giving an indication that a silica supersaturated solution entered ARP/MCU that led to the precipitation of both aluminosilicate ($Na_8Al_6Si_6O_{24}(NO_3)_2$ where Al/Si is nearly one) and silica. There is no evidence to indicate that a stream rich with frit entered ARP/MCU or that it is fine residuals from an earlier simulant test where aluminum and aluminosilicate rich solids where found in the SSFT.[1] It is likely that the addition of sodium hydroxide in the tanks increased the solubility of silicon and, in turn, any silica rich mounds readily dissolved into the macrobatches. Also selective silicon leaching from the solids found in the SSFT on September 2010 may be source of silicon but this mechanism can't be proven at this time.

Table 3. Comparison of the aluminum to silicon ratio between the macrobatches and MCU-12-262 (liquid concentration given in mg/L)

Reference	Al	Si	Al/Si
Batch 2 STI-2008-00446 (Tank 22H)	16.13	112.7	0.14
Batch 2 STI-2008-00446 (Tank 41H)	17752	<137	> 130
Batch 4 STI-2011-00061 (Tank 21H)	2890	58.7	49
Batch 5 STI-2012-00076 (Tank 21H)	7125	46.8	152
Tank 49H STI-2008-00117 (Tank 49H)	9280	< 174	> 53
Solids from SSFT on 9-2010 (μg/g)	136,000	36,270	3.75
MCU-12-262 (μg/g)	5450	86,100	0.06

The digested solids were analyzed in an Atomic Adsorption Spectrometer. This analysis provides the concentration of some of the seven RCRA metals. Cold vapor deposition was performed for mercury detection. RCRA analysis of the solids is shown in Table 4. As can be seen in Table 4, the sodium concentration is within 9% of the reported value from ICP-AES in Table 2. Since the ICP-AES sodium measurement of the ARG glass agreed with its published value, the AA analysis is slightly under reporting the sodium concentration.

Table 4. A summary of the RCRA and sodium elemental content of MCU-12-262 digested by aqua regia

Component	$\mu g/g_{\text{wet sample}}$ (1 σ) (digested by Aqua Regia)
Na	0.035 (20)
Hg	93.1 (20)
As, Pb, Ba, and Ag	< 5, <141, <7.8, and <85.6

Table 5 lists the detected radionuclides found in the digested solids. As can be seen from Table 5, the solids contain a significant concentration of ^{137}Cs indicating the aluminosilicate portion of this solid trapped or sorbed cesium from the radioactive salt solution.[6,1] Also shown in Table 5, the solid contain plutonium 238 and 239/240. This is most likely associated with the titanium portion of the solid (as shown in Appendix B). The measured gross alpha indicates the only alpha source is plutonium either brought in by fines from ARP or sorption on precipitated hydrous titanium dioxide. The value is nearly the sum of the plutonium isotopes. Although the exact total mass of solids found in SEP-401 is not known, the concentration of fissionable plutonium 239 is markedly below the 300 $\mu g/g_{MST}$ of plutonium on MST typically seen at 512-S in ARP. Therefore, there is no criticality concern with these solids. It can also be stated that sorption on precipitated hydrous titanium dioxide (from precipitation of soluble titanium) is the most likely mechanism that these isotopes are observed in this residue. The strontium-90 concentration (0.3 $\mu g/g_{wet}$) is also associated with the titanium in the solid residue and is below the value observed in Table 2 (1.14 $\mu g/g_{wet}$) for total strontium. Approximately, 8.6 ug/gwet of uranium 238 is contained in the residue.

As shown in Table 5, no detectable levels of other fissionable transuranic isotopes were detected. The detection limits of these transuranic are shown in Appendix B.

Table 5. Radiochemical data of MCU-12-262

^{137}Cs	2.27 E7 (5%) dpm/g_{wet}
^{238}Pu	3.02 E5 (5.37%) dpm/g_{wet}
$^{239/240}Pu$	1.88 E4 (10.5%) dpm/g_{wet}
Gross Alpha	2.99 E5 (12%) dpm/g_{wet}
^{90}Sr	8.67 E7 (17.8%) dpm/g_{wet}
241 Am	2.76 E3 (31 %) dpm/g_{wet}
242m Am	< 134 dpm/g_{wet}
243 Am	< 939 dpm/g_{wet}
242 Cm	< 111 dpm/g_{wet}
243 Cm	< 3.23 E3 dpm/g_{wet}
244 Cm	< 111 dpm/g_{wet}
245 Cm	< 2.65 E dpm/g_{wet}
247 Cm	< 3.35 E3 dpm/g_{wet}
232-244$^{\#}$ except 238*	< 0.391 μg/$g_{wet\ sample}$
Mass 238 (^{238}U)	8.6 (6.54%) μg/$g_{wet\ sample}$

*Detected by Mass Spectrometry

Less than 0.391 μg/g of solids of each mass from 232 to 244 were detected by Mass Spectrometry. Therefore, the solid contains less than reportable masses of Am and Cm.

Table 6 shows the calculated concentration of ^{239}Pu and ^{240}Pu using weapons grade and heat source grade composition.

Table 6. Isotopic plutonium concentration of MCU-12-262

Pu isotope	Measured Pu (ug Pu/$g_{sample\ wet}$)	% Pu in Residue	Weapons Grade wt%	Heat Source wt%
^{238}Pu	0.008	8.0	0.02	83.5
^{239}Pu	0.0825	82.0	93	14
^{240}Pu	<0.01	<10	6	2

As can be seen from Table 6, ^{239}Pu is the largest concentration of plutonium in the residue. It comprises approximately 82 % of the plutonium in MCU-12-262. To obtain this concentration with the typical isotopic plutonium distribution output from FB-line (no longer in service) and HB-line, approximately 12.3 to one ratio of weapons grade to heat source grade is required. Table 7 shows the ratio of ^{238}Pu to $^{239/240}Pu$ for this sample, the macrobatches, and Tank 49H. An inspection of Table 7 reveals that the plutonium containing residues of MCU-12-262 may have precipitated when processing macrobatch 4 and 5.

Table 7. Comparison of the plutonium ^{238}Pu to $^{239/240}Pu$ ratio between macrobatches and 12-262

Sample	^{238}Pu	$^{239}Pu + ^{240}Pu$	238/(239 + 240)
Batch 5 STI-2012-00207 (Tank 49H)	40.4 E5	4.55 E4	9
Batch 5 STI-2012-00076	1.46E4	1.49 E3	10
Batch 4 STI-2011-00061	1.26E4	<1.87 E3/<4.56 E3	> 2
Tank 49H STI-2008-00117	4.17 E4	1.24 E3	34
MCU-12-262	30.2 E4	18.8 E3	16

3.2 Solid Sample Characterization

An electronic and vibrational analysis of the solids found in SEP-401 is shown in Figure 3. Figure 3 shows an XRD, EDS, and infrared spectrum of the solid. As can be seen from Fig. 3, the solid is an amorphous mixture of silica and aluminosilicate solids with trapped nitrates and carbonates (i.e., amorphous crancrinite or sodalite).

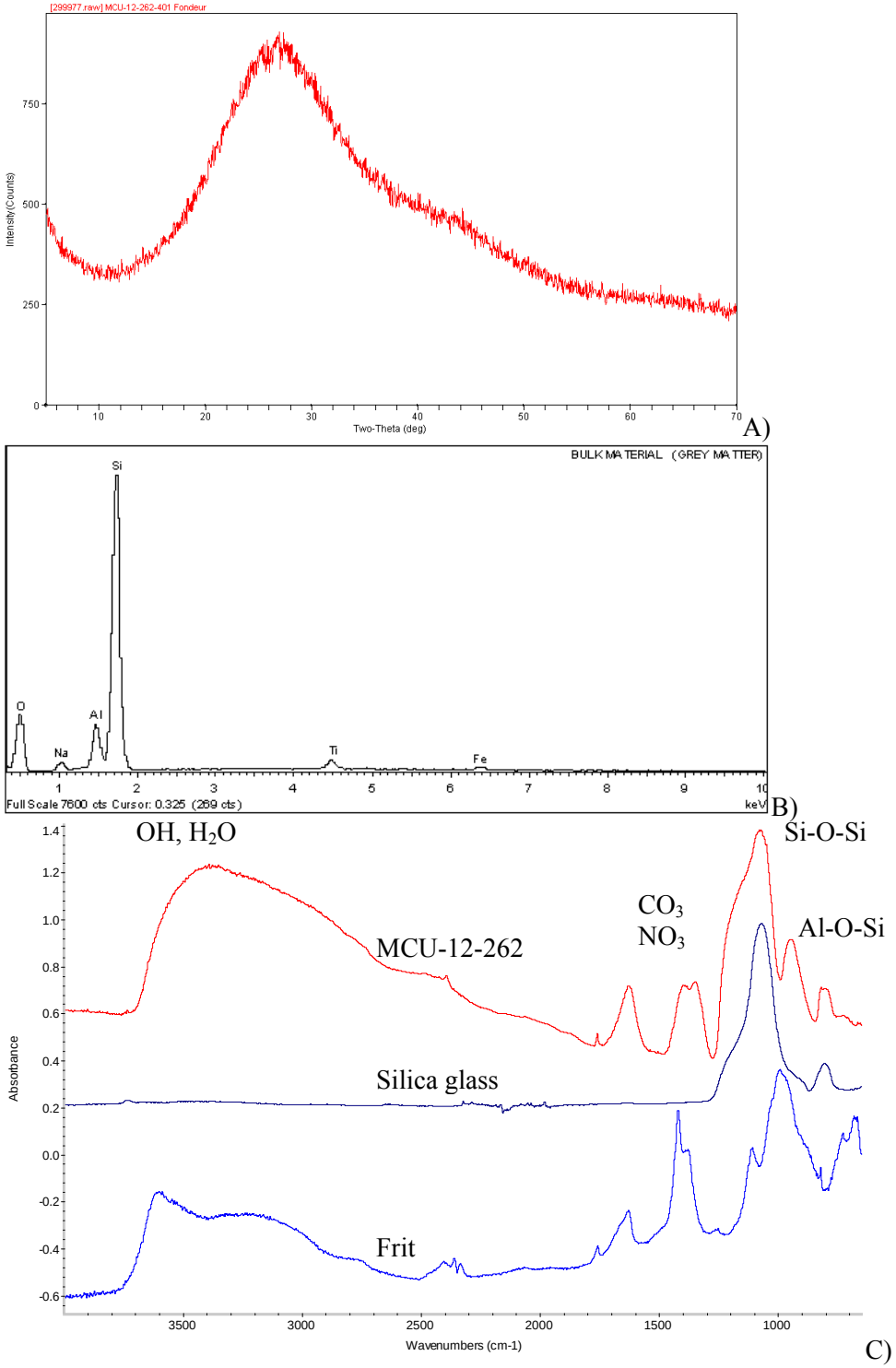

Figure 3. XRD (A), EDS (B), and FTIR (C) of sample MCU-12-262. The solid is a mixture of amorphous silica and amorphous aluminosilicate.

The solid being amorphous confirms the speed with which the solids formed in that no time was available for local ordering (i.e., broad peaks in the XRD spectrum) and even less for large scale ordering (i.e., broad peaks in the FTIR spectrum) to occur. As indicated by the FTIR plot in Fig. 3, the observation of trapped nitrates and carbonates is another indication that these solids formed quickly trapping anions as the solids settled to the bottom of SEP-401.

Gamma spectroscopy revealed that the solids contained 2.27 E7 dpm/g. This suggests the aluminosilicates in these solids sorbed some cesium. PuTTA revealed these solids containing 3.02 E5 dpm/g ^{238}Pu and 1.88 E4 dpm/g $^{239/240}$ Pu. This matches the gross alpha measurement of 2.99 E5 suggesting the only alpha source is this sample is plutonium (238). The small concentration of plutonium is confirmed further by the backscattering imaging and EDS spectra of the particles that make up these solids. Figure C1 in Appendix C shows that only very small amount of Pu-containing particles were found and that whenever a particle contained plutonium, it also contained titanium. This conclusion suggests that MST fines or soluble Ti from ARP continues to deposit at MCU. Also shown in Fig. C1 are particles from stainless steel debris and debris from a titanium alloy of zirconium and tin. This debris has been found in the Decontaminated Salt Solution and Strip Effluent coalescers in the past.[7]

Atomic Absorption spectroscopy showed a negligible concentration of sodium (0.035 ug/g) but more significantly detectable concentration of mercury in these solids (93.1 ug/g). Mercury is used in the canyon to facilitate aluminum cladding dissolution and it is found throughout the tank farm system. The lack of sodium (low concentration) in these solids differentiates them from the sodium aluminosilicate typically found in the scale that forms on the surfaces of the 2H and 3H evaporators.

4.0 Conclusions

The residues found in contactor SEP-401 are a mixture of amorphous silica, aluminosilicate, titanium, and debris from stainless steel. The solids contain low concentrations of ^{238}Pu, fissionable plutonium ^{239}Pu,^{240}Pu, and strontium. These isotopes are associated with titania that came from ARP either as fines or as soluble titanium and then precipitated as hydrous titanium dioxide.

5.0 References

[1] T. B. Peters and S. D. Fink, "Sample Results from Solids Isolated from the SSFT", SRNL-STI-2011-00022, March 2011.
[2] C. E. Duffey and K. L. Lang, "Extraction Contactor 410 Solids Analysis," X-TAR-H-00009, 9 May 2012.
[3] C. E. Duffey and K. L. Lang, "MCU Contactor Solids Analysis," X-TAR-H-00010, 24 May 2012.
[4] Modifier, or Cs-7SB, is 1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, added to the solvent to enhance the solubility of the calixarene and prevent the formation of a third phase.
[5] T. B. Peters,F. F. Fondeur, S. D. Fink "Results of Analyses of Macrobatch 3 Decontaminated Salt Solution (DSS) Coalescer and Pre-filters" SRNL-STI-2011-00513, June 2012
[6] W. R. Wilmarth, J. T. Mills, V. H. Dukes, and R. C. Sullivan, "Fate of Uranium during Sodium Aluminosilicate Formation under Waste Tank Conditions," WSRC-TR-2005-00412, June 2005.
[7] T. B. Peters, F. F. Fondeur, "Results of Analyses of Macrobatch 3 Decontaminated Salt Solution (DSS) Coalescer and Pre-filter," SRNL-STI-2011-00513, June 2012.

Appendix A. ICP-AES of MCU-12-262 digested by alkaline fusion and Aqua Regia.

USER_SAMPLEID:	ADS GENERATED BLANK		ARG-1		MCU_12_262_401_H2O2	
SAMPLE_ID:	300299975		300300242		300299976	
UNITS:	mg/L		ug/g		ug/g	
Element						
Ag	< 2.14	(N/A %RSD)	< 842	(N/A %RSD)	< 855	(N/A %RSD)
Al	< 2.36	(N/A %RSD)	24700	(10 %RSD)	5450	(10.2 %RSD)
B	< 1.14	(N/A %RSD)	25200	(10 %RSD)	< 455	(N/A %RSD)
Ba	< 0.194	(N/A %RSD)	773	(10 %RSD)	< 77.5	(N/A %RSD)
Be	< 0.048	(N/A %RSD)	27.2	(10.7 %RSD)	< 19.2	(N/A %RSD)
Ca	4.77	(10 %RSD)	11800	(10 %RSD)	1270	(10 %RSD)
Cd	< 0.242	(N/A %RSD)	< 95.2	(N/A %RSD)	< 96.6	(N/A %RSD)
Ce	< 2.47	(N/A %RSD)	< 974	(N/A %RSD)	< 988	(N/A %RSD)
Co	< 0.366	(N/A %RSD)	< 144	(N/A %RSD)	< 146	(N/A %RSD)
Cr	< 0.432	(N/A %RSD)	636	(11.6 %RSD)	292	(12 %RSD)
Cu	< 0.13	(N/A %RSD)	< 51.2	(N/A %RSD)	< 51.9	(N/A %RSD)
Fe	0.537	(10.8 %RSD)	98100	(10 %RSD)	1340	(10 %RSD)
Gd	< 0.902	(N/A %RSD)	< 355	(N/A %RSD)	< 360	(N/A %RSD)
K	< 6.8	(N/A %RSD)	23700	(10.4 %RSD)	< 2710	(N/A %RSD)
La	< 0.382	(N/A %RSD)	< 150	(N/A %RSD)	< 153	(N/A %RSD)
Li	< 0.344	(N/A %RSD)	14700	(10 %RSD)	< 137	(N/A %RSD)
Mg	0.045	(10 %RSD)	5160	(10 %RSD)	33.5	(10 %RSD)
Mn	< 0.068	(N/A %RSD)	13600	(10 %RSD)	70.3	(10.4 %RSD)
Mo	< 2.23	(N/A %RSD)	< 878	(N/A %RSD)	< 891	(N/A %RSD)
Ni	< 1.98	(N/A %RSD)	7820	(10.1 %RSD)	< 792	(N/A %RSD)
P	< 4.57	(N/A %RSD)	< 1800	(N/A %RSD)	< 1830	(N/A %RSD)
Pb	< 3.53	(N/A %RSD)	< 1390	(N/A %RSD)	< 1410	(N/A %RSD)
S	< 120	(N/A	< 47200	(N/A	< 47900	(N/A

		%RSD)		%RSD)		%RSD)
Sb	< 7.24	(N/A %RSD)	< 2850	(N/A %RSD)	< 2890	(N/A %RSD)
Si	1.55	(19 %RSD)	194000	(10.4 %RSD)	86100	(10 %RSD)
Sn	< 2.94	(N/A %RSD)	< 1160	(N/A %RSD)	< 1170	(N/A %RSD)
Sr	0.042	(10 %RSD)	48	(10 %RSD)	< 12	(N/A %RSD)
Th	< 1.46	(N/A %RSD)	< 575	(N/A %RSD)	< 583	(N/A %RSD)
Ti	< 0.08	(N/A %RSD)	6740	(10 %RSD)	2460	(10 %RSD)
U	< 11.7	(N/A %RSD)	< 4600	(N/A %RSD)	< 4670	(N/A %RSD)
V	< 0.124	(N/A %RSD)	< 48.8	(N/A %RSD)	< 49.5	(N/A %RSD)
Zn	< 0.552	(N/A %RSD)	< 217	(N/A %RSD)	< 220	(N/A %RSD)

Instrument:	**Leeman Prodigy ICP-ES**
Reviewer:	Boyd Wiedenman
Comments:	2x (ARG 20x Al Ca Fe K Li Mn Na U) S @ 20% Unc

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertanty is the RMS of the method uncertanty and the sample uncertainty.

	USER_SAMPLEID:	ADS GENERATED BLANK	ARG-1	MCU_12_262_401_aqr
	SAMPLE_ID:	300299973	300300241	300299974
	UNITS:	ug/g	ug/g	ug/g
Element				
Ag		< 85.6 (N/A %RSD)	< 85.6 (N/A %RSD)	< 83.7 (N/A %RSD)
Al		< 47.9 (N/A %RSD)	25000 (10 %RSD)	5180 (10 %RSD)
Ba		< 7.76 (N/A %RSD)	769 (10 %RSD)	< 7.59 (N/A %RSD)
Be		< 0.64 (N/A %RSD)	21.8 (17.4 %RSD)	< 0.626 (N/A %RSD)
Ca		19.5 (10.3 %RSD)	11100 (10 %RSD)	97.5 (10 %RSD)
Cd		< 9.68 (N/A %RSD)	< 9.68 (N/A %RSD)	< 9.47 (N/A %RSD)
Ce		< 99 (N/A %RSD)	< 99 (N/A %RSD)	< 96.8 (N/A %RSD)

Co	< 14.6	(N/A %RSD)	68.3	(10.8 %RSD)	< 14.3	(N/A %RSD)
Cr	< 8.64	(N/A %RSD)	733	(10 %RSD)	249	(10 %RSD)
Cu	< 14.9	(N/A %RSD)	47	(12.1 %RSD)	< 14.6	(N/A %RSD)
Fe	48.9	(10.7 %RSD)	103000	(10 %RSD)	1300	(10 %RSD)
Gd	< 36.1	(N/A %RSD)	< 36.1	(N/A %RSD)	< 35.3	(N/A %RSD)
K	< 611	(N/A %RSD)	22800	(10.1 %RSD)	< 598	(N/A %RSD)
La	< 15.3	(N/A %RSD)	< 15.3	(N/A %RSD)	< 14.9	(N/A %RSD)
Li	< 13.8	(N/A %RSD)	15000	(10 %RSD)	< 13.5	(N/A %RSD)
Mg	6.88	(11.1 %RSD)	5300	(10 %RSD)	44.2	(10 %RSD)
Mn	< 4	(N/A %RSD)	15000	(10 %RSD)	28.3	(10.2 %RSD)
Mo	< 89.2	(N/A %RSD)	< 89.2	(N/A %RSD)	< 87.2	(N/A %RSD)
Na	336	(18.4 %RSD)	85600	(10 %RSD)	5890	(10.1 %RSD)
Ni	< 79.3	(N/A %RSD)	7980	(10 %RSD)	162	(10.8 %RSD)
P	< 183	(N/A %RSD)	1180	(10 %RSD)	< 179	(N/A %RSD)
Pb	< 141	(N/A %RSD)	< 141	(N/A %RSD)	< 138	(N/A %RSD)
S	< 4800	(N/A %RSD)	< 4800	(N/A %RSD)	< 4690	(N/A %RSD)
Sb	< 290	(N/A %RSD)	< 290	(N/A %RSD)	< 283	(N/A %RSD)
Sn	< 118	(N/A %RSD)	< 118	(N/A %RSD)	180	(11.1 %RSD)
Sr	< 1.2	(N/A %RSD)	33.7	(11.1 %RSD)	1.14	(10 %RSD)
Th	< 67.8	(N/A %RSD)	< 67.8	(N/A %RSD)	< 66.4	(N/A %RSD)
Ti	< 3.2	(N/A %RSD)	6980	(10 %RSD)	2640	(10 %RSD)
U	< 468	(N/A %RSD)	< 4680	(N/A %RSD)	< 457	(N/A %RSD)
V	< 4.16	(N/A %RSD)	115	(10 %RSD)	< 4.07	(N/A %RSD)
Zn	< 10.5	(N/A %RSD)	217	(10 %RSD)	15.4	(10.5 %RSD)
Zr	6.4	(37.1 %RSD)	1020	(10 %RSD)	7.98	(10.7 %RSD)

Appendix B. Radiochemical analysis of the digested solids from MCU-12-262.

Submission ID	200066051	**Task ID**	401179864
Sample ID	300299974	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_AQR	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	27-JUN-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
AANA (B143)	Na	0.0350 (20)	ug/g	1

Submission ID	200066051	**Task ID**	401179866
Sample ID	300299974	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_AQR	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	28-JUN-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
CVAA HG (B143)	Hg	93.1 (20)	ug/g	1

Submission ID	200066052	**Task ID**	401179882
Sample ID	300299976	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_H2O2	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	09-JUL-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
GAMMA SPEC (B145)	GAMMA SPEC COMMENTS	NA	TEXT	1
GAMMA SPEC (B145)	Cs-137	2.27E+07 (5.00%)	dpm/g	1

Submission ID	200066052	**Task ID**	401179885
Sample ID	300299976	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_H2O2	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	09-JUL-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
PU TTA (B145)	PU-238	3.02E+05 (5.37%)	dpm/g	1
PU TTA (B145)	PU-239/240	1.88E+04 (10.5%)	dpm/g	1

Submission ID	200066052	**Task ID**	401179879
Sample ID	300299976	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_H2O2	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	16-JUL-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
ALPHA COUNTING (B145)	GROSS ALPHA COUNTS	2.99E+05 (12%)	dpm/g	1

Submission ID	200066052	**Task ID**	401179889
Sample ID	300299976	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401_H2O2	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	19-JUL-12

LIMS Method	Description	Result (one sigma %unc)	Units	Rv
SR90 BETA LIQ SCINT (B145)	SR-90	2.17E+05 (17.8%)	dpm/mL	1

Submission ID	200066052	**Task ID**	401182459
Sample ID	300299976	**Task Status**	COMPLETE
User Sample ID	MCU_12_262_401 _H2O2	**Task Condition**	APPROVED
Submitter	FERNANDO.FONDEUR@SRS.GOV//	**Date Done**	23-JUL-12

LIMS Method	Description	Result (one sigma % unc)	Units
GAMMA SPEC CS REMOVED (B145)	Pu-239	<4.87E+06 (MDA)	dpm/g
GAMMA SPEC CS REMOVED (B145)	Am-241	<3.25E+03 (MDA)	dpm/g
GAMMA SPEC CS REMOVED (B145)	GAMMA SPEC COMMENTS	NA	TEXT

Appendix C. Elemental identification of spare particles found in MCU-12-262

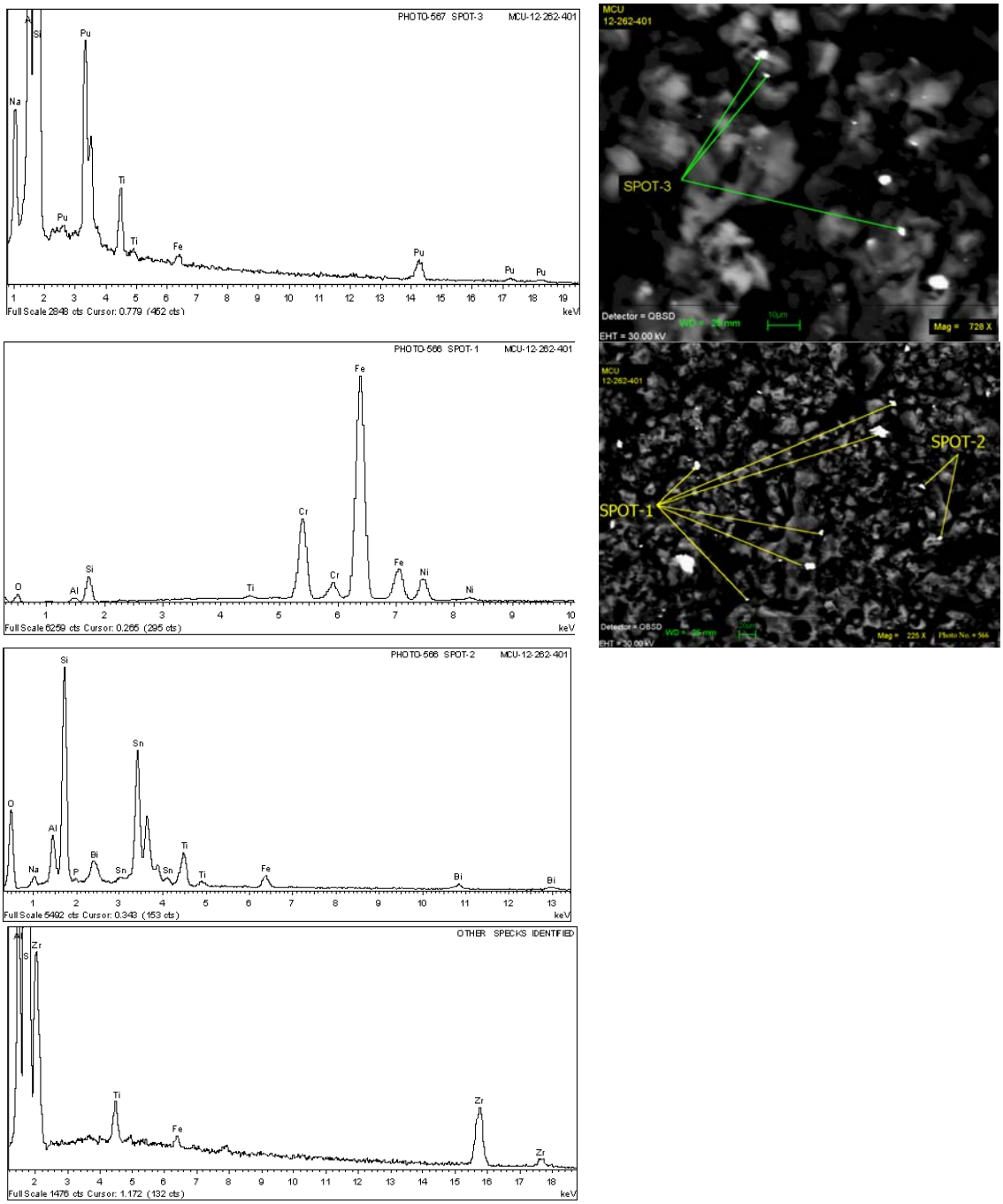

Figure B1. An EDS spectrum of spare particles found in MCU-12-262.

Large majority of the particles were from stainless steel debris. The remaining particles were Pu containing titania and debris from a possible titanium alloy containing Zr and Sn.

Distribution:

S. D. Fink, 773-A
K. M. Fox, 999-W
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)
C. Wilson, 773-A

E. J. Freed, 704-56H
D. J. Martin, 241-152H
M. T. Keefer, 704-56H
S. E. Campbell, 241-152H
B. A. Gifford, 704-56H
Q. L. Nguyen, 241-152H

P. R. Jackson, 703-46A