Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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TANK 49H SALT BATCH SUPERNATE QUALIFICATION FOR ARP/MCU

August 25, 2008

Waste Processing Technology Section
Savannah River National Laboratory
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500

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Prepared For
U.S. Department of Energy

Key Words:
MCU
ARP
Solvent Extraction
Supernate Analysis

Retention: Permanent

Key References:
Technical Task Request: HLW-DWPF-TTR-2007-0024
Interim Report: SRNL-CST-2008-00009, Rev. 0
Task Plan: WSRC-RP-2007-00583, Rev. 0
Analytical Study Plan: SRNL-CST-2007-00105

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List of Acronyms

AA – atomic absorption
AD – Analytical Development
Am/Cm – americium/curium counting method
ARP – Actinide Removal Process
CSS – Clarified Salt Solution (product filtrate from ARP)
CSSX – Caustic Side Solvent Extraction
CV-Hg – cold vapor atomic absorption for mercury
DWPF – Defense Waste Processing Facility
ESS – extraction, scrub, strip (a test method for MCU)
DBP – dibutyl phosphate (method and result)
DF – decontamination factor
DSS – Decontaminated Salt Solution (aqueous product from MCU extraction)
GC-MS – gas chromatograph - mass spectroscopy
IC – ion chromatography
ICP-MS – inductively-coupled plasma - mass spectroscopy
ICP-ES – inductively coupled plasma – emission spectroscopy
Liq. Scint. – liquid scintillation
M – molar
MCU – Modular CSSX Unit
MST – Monosodium Titanate
pH – negative base-10 logarithm of hydrogen ion concentration
PuTTA – Plutonium thenoyl trifluoroacetone scintillation
VOA – volatile organic analysis
SDF – sludge dilution factor
SVOA - semivolatile organic analysis
TIC – total inorganic carbon (primarily carbonate)
TOC – total organic carbon
TSR – Technical Safety Requirement
TTR – Technical Task Request
TTQAP – Task Technical and Quality Assurance Plan
WAC – Waste Acceptance Criteria
WDF – Waste Dilution Factor
WFA – Waste Form Affecting
WAPS – Waste Acceptance Product Specification

SUMMARY

This report covers the methods and data for Tank 49H Salt Batch 1 feed sample qualification. Two sample sets designated A and C were received at SRNL and processed. The following observations are made from the work.

- The solids in the Tank 49H material settle well. Trace solids in Sample Set A liquid settled in-cell overnight and were separated from the liquid by decanting. The decanted liquid contained less than 0.01 wt % insoluble solids. Sample Set C was collected from the tank after an additional 43 days of settling. Sample Set C liquid appeared very clear and solids-free upon receipt and inspection.
- The various samples indicate that dissolved waste salt in Tank 49H was well mixed for both samplings. Densities at different tank elevations in Sample Sets A and C from different tank elevations did not show a statistically significant dependence on elevation in the tank. Uranium in Tank 49H supernate contains between 5 and 6 percent enrichment in ^{235}U.
- Tests using monosodium titanate (MST) with the Tank 49H material gave acceptable decontamination factors (DF) for plutonium and strontium. The strontium sorption agreed rather well (i.e., within 10%) with pretest predictions from sorption models; the plutonium predictions are only slightly worse, likely due to the low concentrations involved.
- A demonstration of cesium extraction, scrubbing and stripping – prototypical of the Modular Caustic-Side Solvent Extraction Unit – yielded expected and acceptable distribution values. The extraction coefficients agreed well (i.e., within ~5%) with pretest predictions using models.

1.0 INTRODUCTION

This report covers the laboratory testing and analyses of Tank 49H Qualification Sample Sets A and C, performed in support of initial radioactive operations of Actinide Removal Process (ARP) and Modular Caustic-Side Solvent Extraction Unit (MCU). Major goals of this work include (1) checking that Tank 49H was well mixed after the last receipt of Tank 23H, (2) characterizing Tank 49H supernate after solids are settled so that its composition can be compared to waste acceptance and hazard criteria, (3) verifying actinide and strontium adsorption with a small scale test using monosodium titanate (MST) and filtration, (4) checking MCU solvent performance when applied to the liquid produced from MST contact, and (5) verifying that in-tank settling after a minimum of 30 days was at least as good or better at reducing solids content after a Tank 49H to Tank 50H transfer occurred than what was observed in less time in the lab. The first four items were covered by Sample Set A. The fifth item was covered by Sample Set C, which had several analyses after compositing as required in the nuclear criticality safety evaluation (NCSE).[1]

There was the possibility of Sample Set B to be taken from Tank 49H 30 days after mixing pump shutdown. Favorable settling results and initial analytical results from Sample Set A eliminated the need for Sample Set B.

This work was specified by Task Technical Request and by Task Technical and Quality Assurance Plan (TTQAP).[2,3] In addition, a sampling plan was written to guide analytical work.[4] Safety and environmental aspects of the work were documented in a Hazard Assessment Package.[5]

Details for the work are contained in controlled laboratory notebooks.[6]

2.0 EXPERIMENTAL

2.1 EXAMINATION OF SAMPLES

Sample Set A and Sample Set C both came in six parts (i.e., 200-mL, stainless-steel sample bottles). The six bottles in each case were filled at three elevations in Tank 49H. Operations personnel collected a pair of samples at each elevation. The density of each part was measured in duplicate, providing 12 values for each of Sample Sets A and C. Densities were determined by weighing calibrated 2-mL volumetric vessels before and after sample addition. Liquids were filtered with 0.45 micron syringe tip filters in all cases.

This work used two methods of sample settling. Sample A settling occurred in-cell after combining the six parts. Observation of a distinct dark solids layer following the 13.5 hour overnight settling of the sample indicated significant removal of solids. Samples from decanted material were prepared with no other separation method – trace suspended fines were thus included in analyses of Sample A Tank 49H supernate.

Sample Set C was from Tank 49H material that settled in-tank for an additional 43 days (with no mixing pump operation during this period) before collection of samples. The composite Sample C as received at SRNL was considered to be homogeneous. No settling or other separations techniques were applied

to Sample C or its parts. Any trace fines were included in chemical and radiochemical analyses of that supernate. Sample Set A was pulled from the tank on December 7, 2007. Sample Set C was pulled on January 18, 2008, providing 43 days of in-tank settling beyond collection of Sample A. A transfer of 411,000 gallons from Tank 49H to Tank 50H completed on January 15, 2008. This transfer apparently did not disturb the settling process, given the observed clarity of Sample Set C.

2.2 SAMPLE PREPARATIONS AND ANALYSES

2.2.1 Sample Processing Flowchart

Figure 1 shows the processing for Sample A. Some changes from the original plan occurred. First, Tank 49H was found to contain less than 5.6 M in sodium, so no dilution was necessary to reduce sodium content. However, a minimal amount of dilution water (actual domestic water from ARP) is added in practice. On 12/17/2008 ARP Engineering (Bob Voegtlen) advised that when no process dilution is required the facility would combine 200 gallons of domestic water, 3614 gallons of Tank 49H decanted supernate, and 10 gallons of MST slurry. In lab work, 5.5 mL dilution water was added to each 100 mL of input Tank 49H supernate. This is the minimum dilution water step shown in Figure 1.

Filtrate from MST testing was used for ESS testing. Original plans scheduled parallel testing with unfiltered Sample A for MST testing while filtered Sample A fed ESS testing. However, the schedule in practice allowed the more process-typical serial use of liquid sample with filtrate from MST testing as the input for ESS tests.

MST and sludge from MST filtrations were prepared for analyses by a relatively new method. A mixture of nitric acid and hydrogen peroxide is effective for digesting samples that contain sludge plus titanium compounds. This preparation allowed the most complete chemical and radiochemical characterization of MST/sludge solids.

Turbidity was used to estimate weight percent insoluble solids in this work because the solids level was so low. Tank samples settled well in practice, so it was clear that use of the filtration and gravimetric method would only provide “below detection” results.

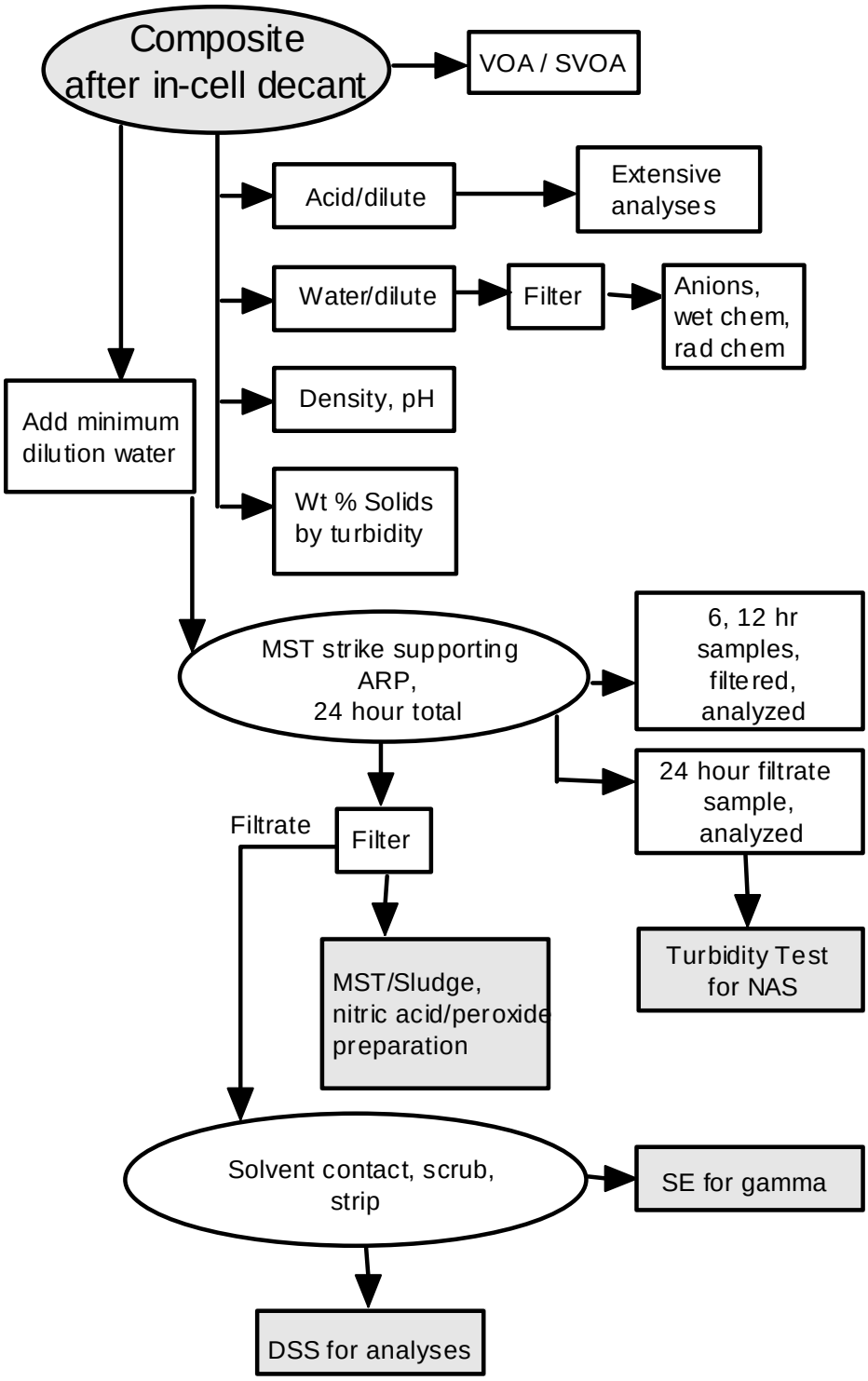


Figure 1. Flowchart for Sample A

2.2.2 Methods of Sample Preparation

Tank 49H supernate sample preparations included sodium peroxide ("alkali") fusions followed by acid uptake.[7] This preparation is considered best for measuring actinides, silicon, and titanium, though the solids-free nature of the materials in this work may have reduced the need for this protocol. Sodium peroxide preparations add unknown but significant levels of sodium (from reagent sodium peroxide) and zirconium (from crucibles). Sodium peroxide fusion preparations are referred to as "Prep 1" in the TTQAP and this report. Acid dilutions ("Prep 2"), in contrast, do not add metallic elements to samples.[8]

The MST sample was dissolved using a preparation consisting of nitric acid and hydrogen peroxide, allowing titanate dissolution without the addition of any metallic elements. The second MST sample was prepared in a radiological hood using the same dissolution method.

Simple water dilution or straight sample submission was appropriate for analytical needs for anions, organics, and some radionuclides like tritium, ^{14}C and ^{129}I. Both ^{14}C and ^{129}I are found in acid-sensitive or volatile components which would be altered or evolved by acid or high temperature preparations. Simple water dilution is referred to as "Prep 3" in this report. Straight sample submission is referred to as "Prep 0". Prep 0 thus covers special preparations that are parts of specific methods as well.

Table 1 below provides the names and procedure citations for the methods used in this work. A detailed description of the methods was provided by Bannochie and Bibler.[9]

Table 1. SRNL-AD Methods, Procedure Citations, and Resulting Components

SRNL-AD METHOD	RESULT
RAD ICPES[10] – Radioactive Inductively Coupled Plasma Emission Spectroscopy	Ag, Al, B, Be, Ba, Ca, Cd, Co, Cr, Cu, Fe, Gd, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Si, Sn, Ti, Zn, Zr
RAD ICPMS[11] - Radioactive Inductively Coupled Plasma Mass Spectroscopy	Isotopes from mass number 81 to 209 and 230 to 252, including ^{233}U and above, ^{237}Np, ^{230}Th, ^{232}Th
Gamma Scan[12]	^{137}Cs, ^{134}Cs
Total Gamma in cells	^{137}Cs
LSC Rad Screen[13]	Total alpha, total beta.
AAAs, AASe[14]	Arsenic and selenium
CV Hg[14] - Cold Vapor Atomic Absorption Hg	Total Mercury
AAK, AANa[15]	Potassium, sodium
Pu TTA, Pu238/241[16]	^{238}Pu, $^{239/240}$Pu, ^{241}Pu
^{90}Sr [17]	^{90}Sr
^{59}Ni /^{63}Ni [18]	^{59}Ni and ^{63}Ni
^{147}Pm /^{151}Sm[13]	^{147}Pm and ^{151}Sm
^{129}I[12]	^{129}I
^{14}C[13]	^{14}C
^{99}Tc[19]	^{99}Tc
Am/Cm[20]	Am and Cm isotopes
Cs removal, then gamma analysis[12]	^{60}Co, ^{106}Ru, ^{125}Sb, ^{125m}Te, ^{137m}Ba, ^{144}Ce, ^{154}Eu, ^{155}Eu, ^{241}Am, ^{226}Ra, emitters outside of ^{137}Cs and ^{134}Cs
Tritium[21]	^{3}H
IC ANIONS[22]	Anions F^-, Cl^-, NO_3^-, NO_2^-, SO_4^{2-}, PO_4^{3-}, $C_2O_4^{2-}$, HCO_2^-
IC CATIONS[22]	Ammonium ion
Total Base / free OH excluding carbonate[23]	Total base and free hydroxide
TIC/TOC[24]	Carbonate
VOA and SVOA[25,26]	VOA and SVOA organics
pH, density[27], wt % solids[28]*	pH, density, wt % solids

* wt % solids were so low in this work that estimates from turbidity were used.

2.2.3 Guidance for Suites of Analyses

Initial guidance on suites of analyses and their driving documents was provided by Jeff Ray in July of 2007. The notes are shown in Appendix A. This information was used to develop the TTQAP, but some simplifications were made as work evolved with customer agreement.

2.3 ACTINIDE REMOVAL PROCESS (ARP) DEMONSTRATION

For the MST tests, technicians dispensed 200 mL of the Tank 49H material into each of two 250-mL polyethylene bottles. We used one of the bottles to conduct the MST experiment, while one bottle served as a control. We added 0.4 g/L of MST solids (from an archived batch of material from Blue Grass Chemical Specialties, lot # MST-2753) to the experiment bottle at time = 0 hours. During the experiment, personnel collected samples from each of the three bottles at 0, 6, 12, and 24 hours. For the sample at 0 hours, sampling occurred immediately prior to MST addition. Throughout the course of the experiment, the bottles were agitated using a magnetic stir plate and stir bars. Temperature control (to 25 ºC) was provided by an actively controlled water bath.

Technicians filtered the samples using 0.45 μm syringe filters, diluted them with acid, removed them from the cells for analysis, and analyzed for plutonium (PuTTA), strontium (beta scintillation), and cesium (gamma scan).

Three weeks after the first MST strike, the researchers performed a MST strike on the control bottle, using the same procedure as the experimental MST strike. Historically, MST tests use an equilibration period after adjusting the ionic strength of the solution to allow post precipitation of actinides to occur and avoid crediting MST sorption for this contribution for the decontamination. In actual facility operations, sampling will not allow discerning the relative contributions from sorption and precipitation. The authors performed this second MST treatment to assess whether appreciable actinide precipitation occurred after adjustment of the ionic strength within the ARP protocols.

2.4 EXTRACTION, SCRUB, STRIP (ESS) EXPERIMENTAL

An ESS test is a series of aqueous-solvent contacts designed to approximately mimic the MCU process, and to test the ability of the solvent to complex and release cesium. Using a 125-mL Teflon ™ separatory funnel, the test starts by contacting 90 mL of aqueous phase (in this case, the Tank 49H material after contact with MST and filtration) with 30 mL of CSSX solvent. After mixing and contacting for ~24 hours, the phases are separated and sampled. The organic phase is transferred back into the funnel, and ~5 mL of scrub acid (0.05 M HNO_3) is added. After mixing and contacting for ~24 hours, the phases are separated and sampled. This general procedure is repeated one more time with scrub acid, followed by three cycles of using strip acid (0.001 M HNO_3).

SRNL measured cesium distribution coefficients (see Section 3.4) of the batch of solvent that most closely matched what is in the MCU facility ("S2-D1-YESBOB-T-WI"). In a previous document,[29] we measured the D values from this same batch of solvent. Those results are presented here for comparison.

3.0 RESULTS AND DISCUSSION

This report provides results in sections that correspond to required analyses described in the TTQAP. Data are generally grouped into mass spectroscopy, chemical (mg/L or μg/g), and radiochemical (pCi/mL) results. Table 1 and Table 2 of the TTQAP are referred to as "Suite A", an abbreviated initial set of analyses, and "Suite B", a more thorough suite of analyses, respectively. Table 3, Table 4 and of the TTQAP cover intermediate MST test samples, MST slurry/sludge filter retentate and filtrate (also known as Clarified Salt Solution, CSS), respectively. Table 6 and Table 7 of the TTQAP cover the ESS testing where MCU solvent contacts supernate, scrub acid, and strip acid solutions. Note that CSS in this work was used as feed to ESS testing, providing a more typical test series than the parallel testing mentioned in the TTQAP.

AD reported some radiochemical results with "upper limit" values rather than "below detection" results with detection limits. Results qualified as "upper limits" are results which were biased high due to one of two circumstances. In cases with low sample activity, such as ^{238}Pu results, small levels of background activity in blank samples (random errors) can elevate the sample result. In the second case, spectral interferences can be present in the analyses. For example, a high ^{137}Cs background in the sample can interfere with alpha or beta liquid scintillation analyses.

In this report "upper limit" data are handled as "below detection" values in the tables and "less than" signs are shown. If two of three results are reported as in the range of detection, then the tables report the average and standard deviation of the two values.

3.1 SAMPLE APPEARANCE, UNIFORMITY, AND DENSITY

Sample Set A (hereafter "Sample A") consisted of six 200-mL stainless steel sample bottles delivered to SRNL on December 7, 2007, and labeled HTF-49-07-145, -146, -147, -148, -149, and -151. Sample Set C (hereafter "Sample C") consisted of six 200-mL stainless steel sample bottles delivered to SRNL on January 18, 2008, and labeled HTF-49-08-17, -18, -20, -21, -22, and -23. Sample C was obtained after 43 days of quiescent time – except for one transfer to Tank 50H – in Tank 49H allowing solids to settle. Sample C underwent a small suite of analyses to provide input into the Nuclear Criticality Safety Assessment. Such input is required by the NCSE.[1] Table 2 below provides summary physical data from the samples.

Table 2. Description of Samples from Tank 49H

Sample Bottle	Sample Elevation from Tank Bottom	Weight, g	Volume, mL	Density, first measurement, g/mL	Density, second measurement, g/mL
Sample A*, December 7, 2007:					
HTF-49-07-145	38 inches	204.569	163.59	1.252	1.250
HTF-49-07-146	38 inches	206.773	165.35	1.259	1.264
HTF-49-07-147	134 inches	209.711	167.70	1.247	1.255
HTF-49-07-148	134 inches	184.458	147.51	1.253	1.243
HTF-49-07-149	230 inches	173.349	138.62	1.247	1.235
HTF-49-07-151	230 inches	194.58	155.60	1.246	1.258
Sample C**, January 18, 2008:					
HTF-49-08-17	154 inches	181.153	144.04	1.229	1.247
HTF-49-08-18	154 inches	226.169	179.84	1.282	1.249
HTF-49-08-20	96 inches	193.11	153.55	1.258	1.266
HTF-49-08-21	96 inches	131.016	104.18	1.258	1.263
HTF-49-08-22	38 inches	184.223	146.48	1.255	1.253
HTF-49-08-23	38 inches	183.775	146.13	1.264	1.270

* The temperature in the Shielded Cells was 17 ºC during density measurements.
** The temperature in the Shielded Cells was 13 ºC during density measurements.

3.1.1 Solution Densities

Liquid density is directly correlated with sodium concentration and of degree of uniformity of the liquid phase composition in the tank. The values in Table 2 provide the following averages of 12 measurements. Variations shown are all standard deviations of one sigma.

Sample A: 1.251 +/- 0.0078 g/L at 17 ^{o}C.
Sample C: 1.258 +/- 0.013 g/L at 13 ^{o}C.

The density data in Table 2 demonstrated near completeness of mixing for the solution throughout the tank. Figure 2 shows the relationship of solution density to tank elevation for Sample A. Figure 3 shows similar data for Sample C. In both cases density declines with height as expected, but the scatter in the data is significant. Standard linear regression in a Microsoft Excel spreadsheet was used to assess the sensitivity of density as a dependent variable to the independent variable (elevation). Sensitivity was assessed by examining the P-value (i.e., the two-tailed probability value) of the elevation variable within each data set. The P-value compares the regression coefficient on elevation divided by its standard error to the Student's t statistic. The P-value is the probability of obtaining a test statistic at least as extreme as the one that was actually measured. Since calculations were performed at the 95% confidence level, P-values of less than 0.05 indicate that elevation is a significant variable. P-values for elevation exceeded 0.05 in each case (0.07 for Sample A and 0.36 for Sample C). Elevation was thus not a statistically significant influence on density, indicating that the solution in Tank 49H was well mixed.

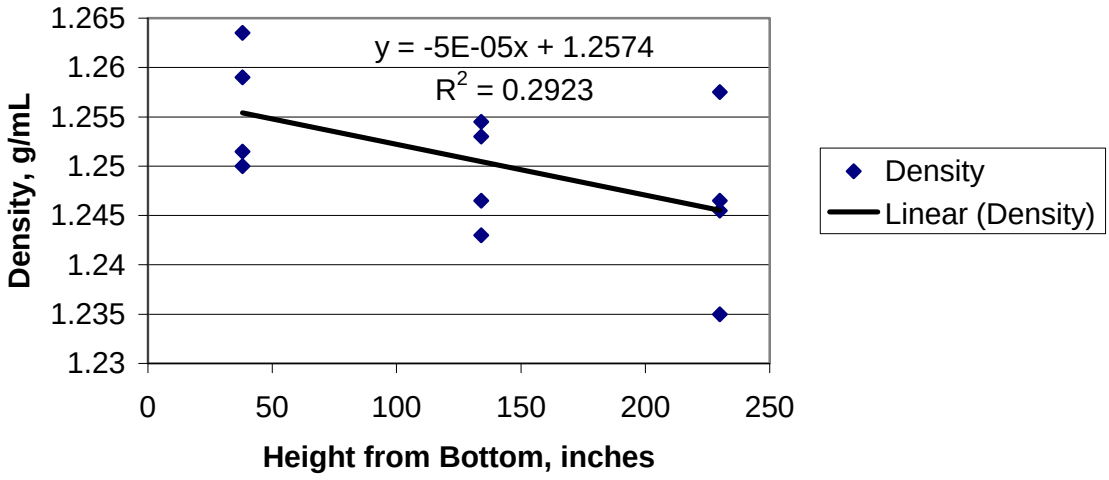


Figure 2. Sample A Solution Density versus Height in Tank 49H

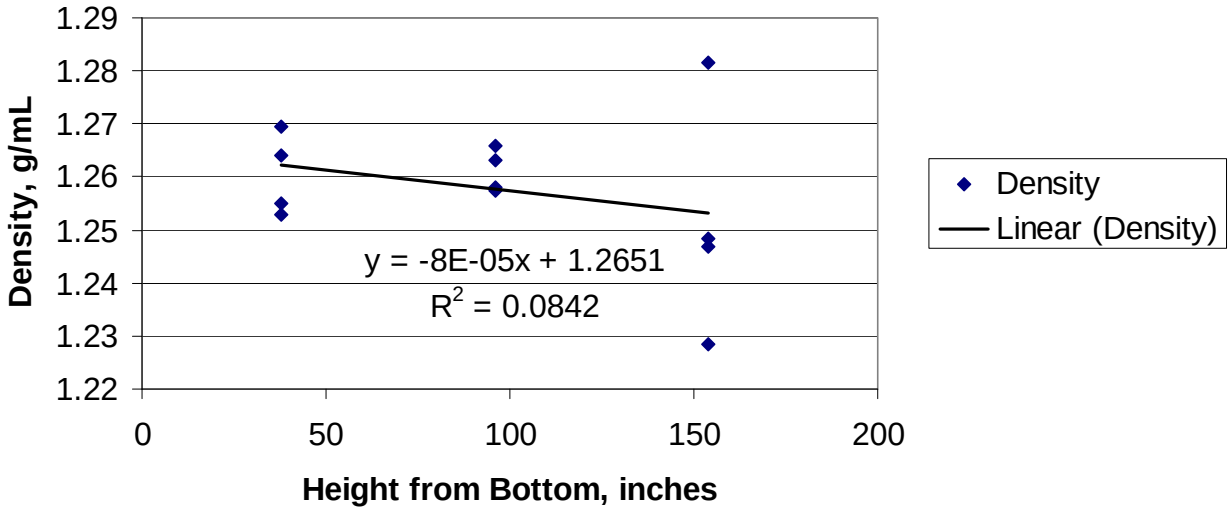


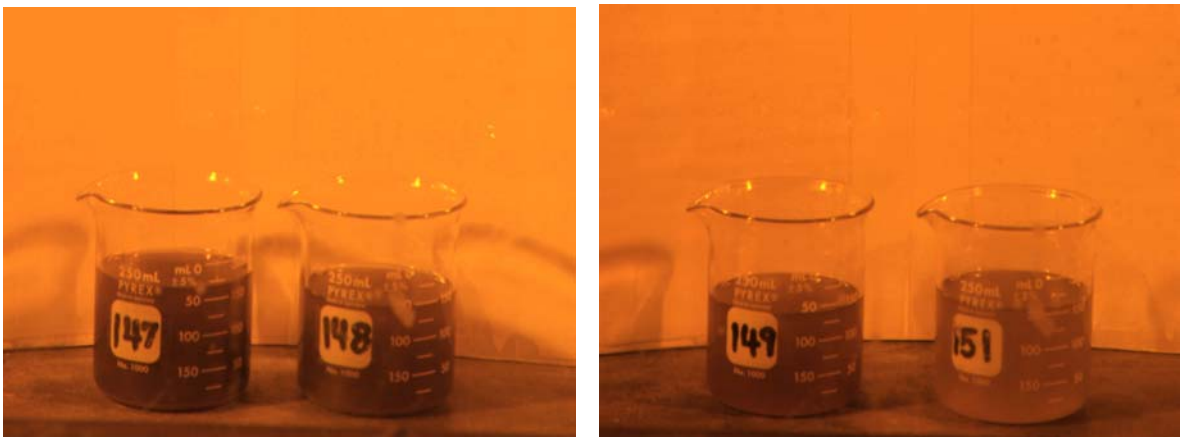
Figure 3. Sample C Solution Density versus Height in Tank 49H

3.1.2 Visual Examination of Sample A

The Tank 49H history leading up to the pulling of Sample A on Friday, December 7, 2007, was provided by Caroline Atseff as recorded in Appendix B. From this information, there was some mixing during the transfer, and the most complete mixing by pumps occurred post transfer from 12/6/07, Thursday, 0330 until 12/6/07, 1047. This duration is 7 hours and 17 minutes of mixing post transfer.

Densities of the six parts of Sample A were measured on December 8, 2007. Results were communicated to both SRNL management and the customer, and it was agreed that the similarity of densities (less than 1.3% difference) indicated good mixing in Tank 49H. The uniformity of densities justified compositing the six parts of the sample. Figure 4 below shows that contents of the bottles varied in color and dark fines were present in all the parts of the sample. The composite was allowed to settle overnight in a 2-liter Teflon® bottle.

Figure 4. Parts of Sample A (two photographs)



The next morning (i.e., approximately 13.5 hours later) the settled sample was examined and photographed without disturbing the state of the settled materials. Figure 5 and Figure 6 show that settling had been fairly effective. Figure 6 is especially revealing – there is a dense layer of solids at the bottom, and there appears to be a second phase of the settling process visible in the liquid. A thin layer of very clear liquid is visible at the top while the bulk of the liquid has visible turbidity. Figure 7, taken two days later, shows that the solids continued to settle after decanting was complete.

Figure 5. Sample A After Overnight Settling

Figure 6. Sample A after Overnight Settling

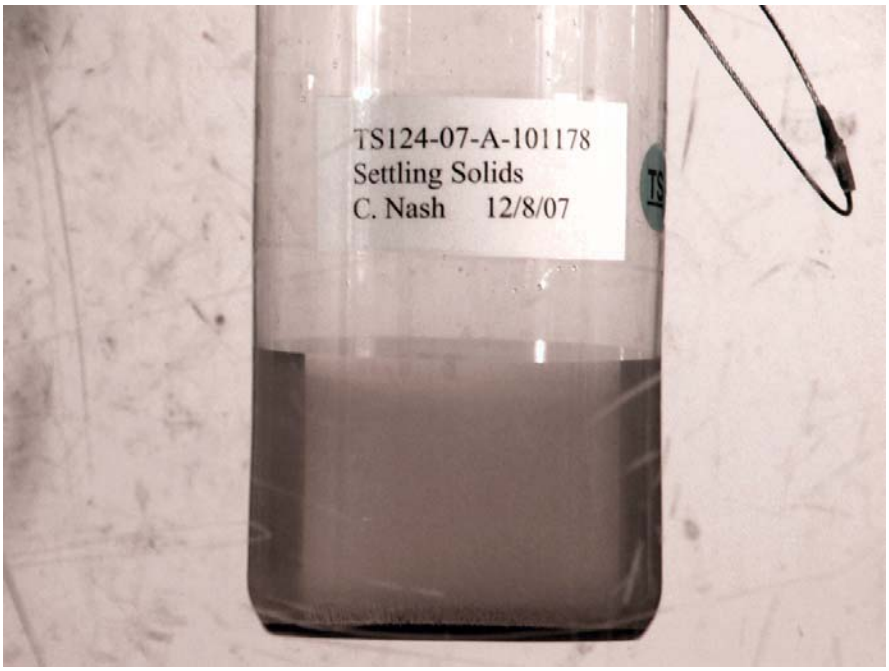
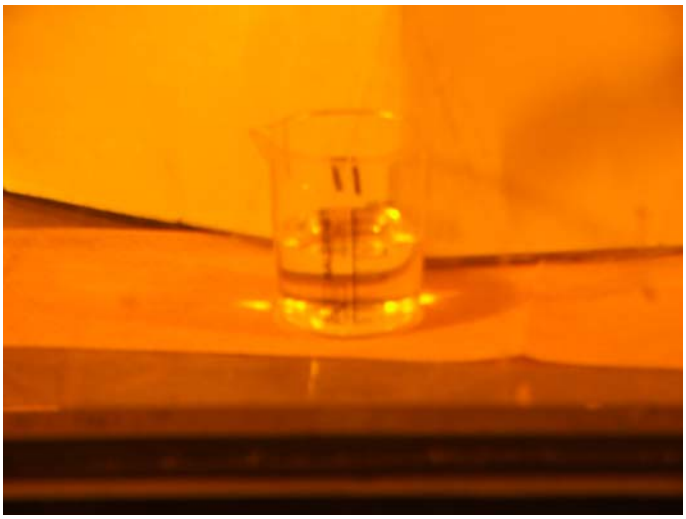


Figure 7. Solids Obtained after Decanting Sample A.

3.1.3 Visual Examination of Sample C

In-tank settling (43 days) of Tank 49H material produced sample parts with no visible fines, in either the beakers or in the emptied metal sample bottles. The liquid in each of the bottles was very clear. Figure 8 shows a typical sample.

Figure 8. Part of Sample C.



3.2 RESULTS OF THE ANALYSIS OF SAMPLE A

3.2.1 Weight Percent Solids

Suite A of the TTQAP specified weight percent solids measurement of the decanted Sample A liquid going forward into Suite B and further experiments. The in-cell settling and decantation clearly provided a liquid of very low total suspended solids (TSS) content, so turbidity was used to provide a much lower estimate of weight percent solids than possible with filtration and mass measurements. The decanted sample had turbidity measurements of 30.7, 31.6, and 30.7 nephelometric turbidity units (NTU), providing an average turbidity of 31.0 +/- 0.52 NTU. Comparison of this value with relationships of NTU to salt supernate weight percent TSS show that Sample A conservatively contained less than 0.01 wt % insoluble solids.[30,31]

3.2.2 Suite A and Suite B Analytical Results

Throughout this report sample Prep #1 is peroxide fusion, Prep #2 is aqua regia or acid dilution prep, and Prep #3 is water dilution, consistent with the TTQAP. Prep 0 is used here to indicate liquid submitted to AD without preparation. In many cases a special preparation specific to the method was performed within AD, ^{129}I measurement being one example. Raw samples were liquid except for the case where MST sludge/slurry was obtained by filtration. ICP-Mass spectroscopy (MS) results are converted to activity when identity of an isotope is straightforward.

Counting methods for specific isotopes are considered more reliable than the MS method when the same data are available by both methods. Examples of counting methods are Am/Cm, PuTTA, $^{238\text{-}241}$Pu (Pu TTA plus ^{241}Pu liquid scintillation providing ^{238}Pu, $^{239\text{ plus }240}$Pu, and ^{241}Pu) and ^{99}Tc. Less preferred values are shown in gray for cases where the same analyte is measured by more than one means. ^{99}Tc by counting is preferred over ^{99}Tc by MS.

In some cases MS results cannot be equated to one isotope directly. In the case of ^{137}Cs, for example, "Mass 137" is not equivalent to the gamma counting result because Mass 137 includes ^{137m}Ba and ^{137}Ba (stable). This report does not convert Mass 137 into an equivalent ^{137}Cs activity for that reason. Stable ^{135}Ba also contributes to Mass 135 in a similar way.

The ^{90}Y and ^{137m}Ba are two important isotopes that are obtained by simple calculation of other existing data. The ^{90}Y is in secular equilibrium with and is the only daughter of ^{90}Sr, so the activity of ^{90}Y is equal to that of ^{90}Sr. The ^{137m}Ba, in contrast, is the product of 94.5% of all ^{137}Cs decays, so the activity of ^{137m}Ba is 0.945 times the ^{137}Cs activity. This report gives ^{90}Sr and ^{137}Cs activities. The ^{90}Y and ^{137m}Ba values are easily calculated from the data with the conversion fractions given here.

Sodium by ICP-ES is considered to be more reliable than sodium by AA (AANa). AANa was used in initial testing to confirm the lower than expected sodium concentration found in Tank 49H.

Potassium by AA is considered to be the most reliable measure for potassium. It is favored over potassium by ICP-ES. A standard solution with 10 mg/L potassium was reported to contain 9.95 mg/L potassium by AA while ICP-ES reported 11.9 mg/L. Total lead by MS is considered to be a better measure of lead than ICP-ES, mainly because of the better detection limits.

Total alpha and beta by liquid scintillation was found to have interference from the high ^{137}Cs in the sample. Total alpha with ^{137}Cs included in the sample provided upper limit values for alpha because the beta activity was over 100 times that of the alpha activity. The alpha values are biased high and should not be used. Cesium-removed alpha values are considered reliable. Removal of ^{137}Cs was found to reduce the total beta.

Suite B results complement those of Suite A. Suite B together with Suite A provides a full set of Tank 49H supernate analyses for use in WAC and other evaluations.

Table 3. Suite A Chemical and MS Data, Acid Preparation

Method	First Result	Second Result	Third Result	Average	1 sigma, units
ICP-ES sodium	1.20E+05	1.16E+05	1.12E+05	1.16E+05	0.04E+05 mg/L
ICP-ES sodium				5.05	M
AA sodium	1.15E+05	1.11E+05	1.07E+05	1.108 E+05	0.04 E+05 mg/L
AA sodium				4.82	M
CV-AA Mercury	9.43E+00	9.22E+00	8.85E+00	9.17	0.29 mg/L
MS, (stable) ^{59}Co	4.75E-01	4.75E-01	4.55E-01	4.69 E-01	0.12 E-01 mg/L
MS, Total Rb	1.00E+00	9.58E-01	9.33E-01	9.64 E-01	0.36 E-01 mg/L
MS, Mass 99 (^{99}Tc)	4.89E+00	4.74E+00	4.80E+00	4.81	0.075 mg/L
MS, Mass 99 (^{99}Tc)				8.15 E+04	0.13 E+04 pCi/mL
MS, Mass 133 (^{133}Cs)	2.24E+00	2.25E+00	2.03E+00	2.17	0.13 mg/L
MS, Mass 135	2.90E-01	2.89E-01	2.69E-01	2.83 E-01	0.12 E-01 mg/L
MS, Mass 137	1.10E+00	1.07E+00	1.04E+00	1.07	0.03 mg/L
MS total lead	3.33E-01	4.24E-01	3.75E-01	3.77 E-01	0.46 E-01 mg/L
MS ^{233}U	<1.71E-02	<1.69E-02	<1.66E-02	<1.66 E-02	mg/L
MS ^{233}U				< 1.61E+02	pCi/mL
MS ^{234}U	3.88E-02	2.63E-02	3.63E-02	3.38 E-02	0.66 E-02 mg/L
MS ^{234}U				2.11 E+02	0.41 E+02 pCi/mL
MS ^{235}U	1.01E-01	8.63E-02	1.01E-01	9.63 E-02	0.87 E-02 mg/L
MS ^{235}U				2.08 E-01	0.188 E-01 pCi/mL
MS ^{236}U	4.00E-02	4.50E-02	3.38E-02	3.96 E-02	0.56 E-02 mg/L
MS ^{236}U				2.56	0.36 pCi/mL
MS ^{238}U	1.68E+00	1.69E+00	1.68E+00	1.68	0.098 mg/L
MS ^{238}U				5.65 E-01	0.33 E-01 pCi/mL
MS ^{237}Np	2.38E-02	2.38E-02	2.88E-02	2.54 E-02	0.29 E-02 mg/L
MS ^{237}Np				1.79 E+01	0.204 E+01 pCi/mL
MS ^{230}Th	<1.71E-02	<1.69E-02	<1.66E-02	<1.66 E-02	mg/L
MS ^{230}Th				<3.50E+02	pCi/mL
MS ^{232}Th	<1.29E-01	<1.26E-01	<1.25E-01	<1.25E-01	mg/L
MS ^{232}Th				<1.37E-02	pCi/mL
MS ^{239}Pu	<1.71E-02	<1.69E-02	<1.66E-02	<1.66E-02	mg/L
MS ^{239}Pu				<1.03E+03	pCi/mL
MS ^{240}Pu	<1.71E-02	<1.69E-02	<1.66E-02	<1.66E-02	mg/L
MS ^{240}Pu				<3.78E+03	pCi/mL
MS ^{241}Pu, ^{241}Am	<1.71E-02	<1.69E-02	<1.66E-02	<1.66E-02	mg/L
MS ^{242}Pu	<2.58E-02	<2.54E-02	<2.49E-02	<2.49E-02	mg/L
MS ^{242}Pu				<9.51E+01	pCi/mL
MS ^{243}Am, ^{243}Cm	<1.71E-02	<1.69E-02	<1.66E-02	<1.66E-02	mg/L
MS ^{244}Pu, ^{244}Cm	<1.71E-02	<1.69E-02	<1.66E-02	<1.66E-02	mg/L

Note: Gray shading indicates the less preferred data when exact same analytes are obtained by different methods. For example, ^{99}Tc by radiochemistry is preferred over MS data. Sodium by ICP-ES is recommended over sodium AA data.

Table 4. Suite A Radiochemical Data (pCi/mL)

Method	Prep	First Result	Second Result	Third Result	Average, (pCi/mL)	1 sigma, (pCi/mL)
Gamma ^{137}Cs	2	6.76E+07	6.59E+07	6.08E+07	6.48E+07	0.35 E+07
^{137m}Ba		Calculated from ^{137}Cs			6.12E+07	0.33 E+07
Gamma ^{134}Cs	2	<1.47E+05	<1.61E+05	<1.46E+05	<1.46E+05	
^{137}Cs by gamma in-cell	2	7.32E+07	7.38E+07	7.32E+07	7.34E+07	0.033 E+07
Total Alpha w/ Cs	2	6.82E+05	<4.39E+05	6.03E+05	<4.39E+05 (see text)	
Total beta w/ Cs	2	7.55E+07	8.56E+07	7.60E+07	7.90E+07	0.57 E+07
Total alpha, Cs-removed	2	4.95E+04	4.90E+04	4.39E+04	4.75E+04	0.31 E+04
Total beta, Cs-removed	2	9.74E+05	9.80E+05	9.74E+05	9.76E+05	0.033 E+05
^{238}Pu (PuTTA)*	1	3.97E+04	4.42E+04	4.11E+04	4.17E+04	0.23 E+04
^{238}Pu (238/241)*	1	3.74E+04	4.58E+04	4.24E+04	4.19E+04	0.42 E+04
$^{239-240}$Pu (PuTTA)*	1	5.50E+02	4.52E+02	2.73E+03**	1.24E+03**	1.29 E+03
$^{239-240}$Pu (238/241)*	1	6.87E+02	5.34E+02	5.52E+02	5.91E+02	0.84 E+02
^{241}Pu(Liq. Scint.)*	1	1.67E+04	7.83E+03	7.38E+03	1.06E+04	0.53 E+04
^{90}Sr (^{90}Y)	2	3.74E+05	4.25E+05	3.76E+05	3.92E+05	0.29 E+05
^{60}Co by gamma	0	9.05E+00	1.12E+01	<1.46E+01	1.01E+01	0.15 E+01
^{106}Ru by gamma	0	<1.19E+02	<1.26E+02	<1.12E+02	<1.12E+02	
^{125}Sb by gamma	0	<7.66E+01	<7.39E+01	<5.54E+01	<5.54E+01	
^{126}Sb by gamma	0	2.18E+02	2.26E+02	2.27E+02	2.24E+02	0.05 E+02
^{126}Sn by gamma	0	2.91E+02	3.19E+02	2.99E+02	3.03E+02	0.14 E+02
^{144}Ce by gamma	0	<2.05E+02	<2.09E+02	<2.09E+02	<2.05E+02	
^{154}Eu by gamma	0	1.22E+02	1.19E+02	1.12E+02	1.18 E+02	0.05E+02
^{155}Eu by gamma	0	<1.15E+02	<3.60E+02	<1.17E+02	<1.15E+02	
^{226}Ra by gamma	0	<5.68E+02	<3.44E+02	<5.59E+02	<3.44E+02	
^{241}Am by gamma	0	<2.34E+02	<2.37E+02	<2.34E+02	<2.34E+02	

* The means of measuring ^{238}Pu and $^{239-240}$Pu are the same for the PuTTA and "238/241" analytical requests. The "238/241" request also provides ^{241}Pu measurement by liquid scintillation. All data points are provided to allow the user to apply best judgment for specific applications.

** One apparent outlier is noted here. This line is grayed for this reason. The triplicate analysis just below this appears to be more reliable.

Table 4 note: Total alpha and beta data with cesium removal are preferred over data from measurements without such removal.

Table 5. Sample A Suite B Chemical Data – Anions, Organic, and AA Results

Method	Prep	First Result	Second Result	Third Result	Average	1 sigma, units
AA Arsenic	2	<1	<1	<1	<1	mg/L
AA Selenium	2	<2	<2	<2	<2	mg/L
AA Potassium	2	234	239	243	238	4.4 mg/L
Fluoride	3	<585	<540	<526	<526*	mg/L
Fluoride*					<150*	mg/L
Formate	3	<585	<540	<526	<526	mg/L
Chloride	3	<585	<540	<526	<526*	mg/L
Chloride*					<150*	mg/L
Nitrite	3	12200	12500	12520	12400 (0.271 M)	133 mg/L
Bromide	3	<585	<540	<526	<526	mg/L
Nitrate	3	172000	174000	174000	173000 (2.79 M)	946 mg/L
Phosphate	3	<585	<540	<526	<526**	mg/L
Phosphate*	3				1100**	mg/L
Sulfate	3	12200	12200	12100	12200 (0.127 M)	53 mg/L
Oxalate	3	<585	<540	<526	<526	mg/L
Carbonate (TIC)	3	1.85E+04	1.88E+04	1.86E+04	18600 (0.31 M)	151 mg/L
Total Base	3	1.59	1.55	1.51	1.55	0.039 M
Free OH[t]	3	0.5314	0.6650	0.6427	0.613	0.072 M
Other Base	3	0.5981	0.5646	0.5449	0.569	0.027 M
VOA	0	<0.25	<0.25	<0.25	<0.25	mg/L
SVOA	0	See note[tt]	<4	<4	<4	mg/L

* Repeated analysis of this sample for only halides and phosphate provided "below detection" results of <150 mg/L for fluoride and chloride.

**An additional measurement provided a value of 1100 mg/L phosphate. An error in the original reporting caused the initial "below detection" result. We recommend using the ICP-ES results (Table 7 acid prep) for phosphorous to calculate an inferred maximum phosphate concentration.

[t] This range of Free OH is equivalent to a pH range of 13.6 to 13.8.

[tt] In this sample dioctylphthalates were detected at 120 mg/L. These chemicals are plasticizers in polyethylene bottle material. This result is not considered representative of organic in Sample A liquid.

The workscope included two preparation methods for ICP-ES. ICP-ES was performed on both alkali and acid prepared samples to obtain the most comprehensive view of feed composition. The alkali preparation is best for titanium, zinc, and silicon. Table 6 below provides the results for the alkali fusion/acid uptake preparation. Extra potassium may have come in with the alkali reagent. Table 7 results are from the same input material, acid diluted. The acid preparation is better for most metals and provides the only ICP-ES results for sodium and zirconium. No metals are expected to be added in that preparation method. Sulfur from the acid preparation is probably better than that of the alkali preparation since it agrees more closely with sulfate by IC anions. Phosphorus from the acid preparation is recommended for a conservative calculation of phosphate in the solution.

Table 6. **Suite B Chemical Data – ICP-ES Results, Alkali Prep (mg/L)**

Element	First Analysis	Second Analysis	Third Analysis	Average mg/L	1-sigma, mg/L
Ag	<64.2	<67.8	<65.7	<64.2	
Al	8720	9820	9300	9280	550
B	<16.9	<17.8	<17.3	<16.9	
Ba	<2.99	<3.16	<3.06	<2.99	
Be	<0.74	<0.79	<0.76	<0.74	
Ca	<148.8	<245.1	<192.6	<148.8	
Cd	<5.50	<5.81	<5.64	<5.50	
Ce	<215.1	<226.3	<220.1	<215.1	
Cr	93	108	99	99.7	8
Cu	<11.0	<11.7	<11.3	<11.0	
Fe	20	57	38	38	19
Gd	<23.3	<24.6	<23.8	<23.3	
K	559	632	593	594	36
La	<28.5	<30.0	<29.1	<28.5	
Li	<19.8	<20.9	<20.1	<19.8	
Mg	37.1	56.0	38.9	44.0	10
Mn	<6.43	<6.79	<6.58	<6.43	
Mo	<21.3	<22.5	<21.8	<21.3	
Na [°]	--	--	--	--	
Ni	<15.4	<16.3	<15.8	<15.4	
P	365	254	360	326	63
Pb	<55.3	<58.4	<56.5	<55.3	
S	5630	5080	5600	5440	310
Sb	<106	<112	<108	<106	
Si	<174	<184	<178	<174	
Sn	<315	<333	<323	<315	
Sr	<28.0	<29.6	<28.8	<28.0	
Ti	<12.6	<13.3	<12.9	<12.6	
U	<1280	<1350	<1310	<1280	
V	<12.2	<12.9	<11.7	<11.7	
Zn	<68.8	<72.7	<70.4	<68.8	
Zr [°]	--	--	--	--	

Gray shading indicates where acid preparation data (next table) are preferred over data from alkali preparations.

[°] The preparation method for these analyses invalidates the sodium and zirconium results.

Table 7. Suite B Chemical Data – ICP-ES Results, Acid Prep (mg/L)

Element	First Analysis	Second Analysis	Third Analysis	Average mg/L	1-sigma, mg/L
Ag	<32.9	<32.4	<31.9	<31.9	
Al	9120	9180	9180	9160	40
B	<8.62	<8.49	<8.35	<8.35	
Ba	<1.54	<1.51	<1.49	<1.49	
Be	<0.38	<0.38	<0.37	<0.37	
Ca	<27.4	<27.0	<26.7	<26.7	
Cd	<2.83	<2.78	<2.74	<2.74	
Ce	<109.9	<108.3	<106.5	<106.5	
Cr	89	90	89	89	1
Cu	<5.65	<5.58	<5.48	<5.48	
Fe	10	6	10	9	2
Gd	<11.9	<11.7	<11.6	<11.6	
K	325	283	343	317	31
La	<14.6	<14.4	<14.1	<14.1	
Li	<10.1	<9.95	<9.79	<9.79	
Mg	<1.19	<1.17	<1.15	<1.15	
Mn	<3.30	<3.25	<3.19	<3.19	
Mo	<10.89	<10.73	<10.55	<10.55	
Na	119000	123000	120000	121000	2250
Ni	<7.87	<7.75	<7.63	<7.63	
P	418	439	441	433	13
Pb	<28.3	<27.9	<27.4	<27.3	
S	4690	4950	4700	4780	148
Sb	<54.3	<53.4	<52.5	<52.5	
Si	<89.2	<87.8	<86.4	<86.4	
Sn	<161	<159	<156	<156	
Sr	<14.4	<14.1	<13.9	<13.9	
Ti	<6.45	<6.35	<6.25	<6.25	
U	<655	<645	<635	<635	
V	<6.25	<6.15	<6.05	<6.05	
Zn	<35.3	<34.8	<34.1	<34.1	
Zr	<3.90	<3.84	<3.78	<3.78	

Gray shading indicates where alkali preparation data (previous table) are preferred over data from acid preparations. See Table 3 MS data on Pb and U for best detection of these two elements.

Table 8. Suite B Radiochemical Data (pCi/mL)

Method	Prep	First Result	Second Result	Third Result	Average, pCi/mL	1 sigma, pCi/mL
^{79}Se	0	<46.4	<49.1	<54.1	<46.4	
^{129}I	0	1.75E+01	1.69E+01	1.54E+01	1.66 E+01	0.11 E+01
^{14}C	0	6.89E+02	7.34E+02	4.01E+02	6.08 E+02	1.8 E+02
^{59}Ni	0	<191	<191	<104	<104	
^{63}Ni	0	112	223	134	156	59
^{99}Tc	0	7.03E+04	7.16E+04	7.30E+04	7.16 E+04	0.14 E+04
^{147}Pm	1	<9.74E+02	<9.35E+02	<9.07E+02	<9.07E+02	
^{151}Sm	1	<5.86E+02	<6.25E+02	<5.53E+02	<5.53E+02	
^{3}H	3	<3.78E+03	<3.49E+03	<3.40E+03	<3.40E+03	
^{241}Am by Am/Cm	1	<2.98E+03	<2.93E+03	<2.09E+03	<2.09E+03	
^{243}Am by Am/Cm	1	<5.35E+02	<7.30E+02	<7.02E+02	<5.35E+02	
^{242m}Am by Am/Cm	1	<6.75E+02	<9.18E+02	<5.68E+02	<5.68E+02	
^{243}Cm by Am/Cm	1	<1.55E+03	<1.98E+03	<2.66E+03	<1.55E+03	
^{245}Cm by Am/Cm	1	<1.26E+03	<1.61E+03	<2.18E+03	<1.26E+03	
^{247}Cm by Am/Cm	1	<2.29E+03	<2.75E+03	<3.60E+03	<2.29E+03	
^{249}Cf by Am/Cm	1	<2.45E+03	<3.13E+03	<3.80E+03	<2.45E+03	
^{251}Cf by Am/Cm	1	<1.50E+03	<1.91E+03	<2.70E+03	<1.50E+03	
^{242}Cm by Am/Cm	1	<6.05E+01	<7.98E+01	<8.48E+01	<6.05E+01	
^{244}Cm by Am/Cm	1	<5.59E+02	<7.59E+02	<4.70E+02	<4.70E+02	

The workscope called for plutonium analysis of filtered Sample A liquid. Table 9 below shows the results. Plutonium concentrations in Table 9 are considered to be of soluble plutonium in the supernate. The samples had seen filtration with filters of 0.45 micron nominal pore size.

Table 9. Filtered Sample A, Plutonium Results

Method	Prep	First Result	Second Result	Third Result	Average, pCi/mL	1 sigma, pCi/mL
$^{239-240}$Pu (238/241)	1	1.12E+02	1.90E+02	1.74E+02	1.59 E+02	0.42 E+02
^{238}Pu (238/241)	1	3.41E+04	3.09E+04	3.07E+04	3.19 E+04	0.19 E+04
^{241}Pu(Liq. Scint)	1	<2.50E+03	<2.80E+03	<2.55E+03	<2.50 E+03	

Sample C concentrations of sodium, strontium (cold), plutonium isotopes, and analytes provided by MS are provided in Table 10 and

Table 11 below. The standard deviations on $^{239-240}Pu$ and ^{241}Pu were high because they were near method detection limits, though still in the range of quantification in these cases.

Table 10. **Non-Filtered Sample C Sodium, Strontium and Plutonium Results**

Method	Prep	First Result	Second Result	Third Result	Average	1 sigma, units
Sodium by ICP-ES	2	1.20E+05	1.25E+05	1.19E+05	1.21 E+05	0.031 E+05 mg/L
Sodium by ICP-ES	2				(5.26 M)	M
Strontium (cold) by ICP-ES	2	<0.277	NA	NA	<0.277	mg/L
^{238}Pu (238/241)	2	3.48E+04	3.46E+04	3.11E+04	3.35 E+04	0.21 E+04 pCi/mL
$^{239-240}Pu$ (238/241)	2	1.13E+02	4.18E+02	3.80E+02	3.04 E+02	1.7 E+02 pCi/mL
^{241}Pu (Liq. Scint)	2	2.45E+03	8.04E+03	2.79E+03	4.43 E+03	3.1 E+03 pCi/mL

Table 11. Non-Filtered Sample C MS Data (Acid Prep, mg/L, pCi/mL)

Method	First Result	Second Result	Third Result	Average	1 sigma, units
MS, (stable) ^{59}Co	<4.01E-02	<4.01E-02	<4.01E-02	<4.01E-02	mg/L
MS, Total Rb	1.08	1.05	1.03	1.05	0.023 mg/L
MS, Mass 99 (^{99}Tc)	4.57	4.62	4.84	4.68	0.14 mg/L
MS, Mass 99 (^{99}Tc)				7.93 E+04	0.237 E+03 pCi/mL
MS, Mass 133 (^{133}Cs)	2.26	2.29	2.32	2.29	0.032 mg/L
MS, Mass 135	0.31	0.325	0.318	0.318	0.0075 mg/L
MS, Mass 137	0.974	0.983	1.02	0.991	0.022 mg/L
MS total lead	0.249	0.240	0.258	0.249	0.0088 mg/L
MS ^{233}U	<2.51E-02	<2.51E-02	<2.51E-02	<2.51E-02	mg/L
MS ^{233}U				<2.43E+02	pCi/mL
MS ^{234}U	5.03E-02	4.40E-02	3.77E-02	0.044	0.00628 mg/L
MS ^{234}U				2.75 E+02	0.392 E+02 pCi/mL
MS ^{235}U	1.07E-01	1.04E-01	1.06E-01	0.106	0.00126 mg/L
MS ^{235}U				2.29 E-01	0.0272 E-01 pCi/mL
MS ^{236}U	4.02E-02	4.52E-02	4.15E-02	0.0423	0.0026 mg/L
MS ^{236}U				2.74	0.168 pCi/mL
MS ^{238}U	1.58E+00	1.55E+00	1.57E+00	1.57	0.0192 mg/L
MS ^{238}U				5.28 E-01	0.0646 E-01 pCi/mL
MS ^{237}Np	2.76E-02	2.89E-02	3.52E-02	3.06 E-02	0.404 E-02 mg/L
MS ^{237}Np				2.16 E+01	0.285 E+01 pCi/mL
MS ^{230}Th	<1.50E-02	<1.50E-02	<1.50E-02	<1.50 E-02	mg/L
MS ^{230}Th				<3.16 E+02	pCi/mL
MS ^{232}Th	<3.51E-02	<3.51E-02	<3.51E-02	<3.51 E-02	mg/L
MS ^{232}Th				<3.85 E-03	pCi/mL
MS ^{239}Pu	<2.00E-02	<2.00E-02	<2.00E-02	<2.00 E-02	mg/L
MS ^{239}Pu				<1.24 E+03	pCi/mL
MS ^{240}Pu	<2.51E-02	<2.51E-02	<2.51E-02	<2.51E-02	mg/L
MS ^{240}Pu				<5.72E+03	pCi/mL
MS ^{241}Pu, ^{241}Am	<1.00E-02	<1.00E-02	<1.00E-02	<1.00 E-02	mg/L
MS ^{242}Pu	<2.00E-02	<2.00E-02	<2.00E-02	<2.00 E-02	mg/L
MS ^{242}Pu				<7.64E+01	pCi/mL
MS ^{243}Am, ^{243}Cm	<2.51E-02	<2.51E-02	<2.51E-02	<2.51 E-02	mg/L
MS ^{244}Pu, ^{244}Cm	<2.51E-02	<2.51E-02	<2.51E-02	<2.51 E-02	mg/L

3.2.3 ARP Domestic Water

The work following this section required feed diluted with ARP domestic water. While sodium concentration in Tank 49H supernate was lower than that required to dilute the material, ARP provides some dilution regardless due to tank heels and MST addition water. The plant provided a sample of domestic water from an eyewash station connected to the ARP water header. The sample was discolored and contained solids. After discussion with ARP/MCU Process Engineering, researchers used decanted liquid from the top of the sample bottle. The water was not filtered. The dilution added the equivalent of 200 gallons dilution water to 3614 gallons supernate (i.e., 5.5 mL dilution water added to each 100 mL of input supernate). A single set of analyses of that water is given in Table 12 below. Potassium by AA is the better value to use versus the other (ICP-ES) value. The previous section discusses the comparison test of the methods with a standard potassium solution.

Table 12. ARP Domestic Water Chemical Data

Method:	ICP-ES		Method:	ICP-ES		Method:	IC-Anions
Component	Conc., mg/L		Component	Conc., mg/L		Component	Conc., mg/L
Ag	<0.019		Mn	0.029		Fluoride	<5
Al	<0.280		Mo	<0.043		Formate	<5
B	<0.033		Na	31.5		Chloride	6
Ba	0.005		Ni	<0.014		Nitrite	<5
Ca	0.865		P	<0.419		Bromide	<5
Cd	<0.008		Pb	<0.049		Nitrate	<5
Ce	<0.142		S	0.613		Phosphate	<12
Cr	<0.009		Si	1.32		Sulfate	<5
Cu	<0.022		Sn	<0.032		**Miscellaneous**	**Methods**
Fe	0.823		Sr	<0.204		pH	7.59
K	0.456		Ti	<0.017		TIC	18.1 mg/L
La	<0.017		V	<0.011		TOC	28.7 mg/L
Li	<0.014		Zn	0.519		Total C	46.8 mg/L
Mg	<0.189		Zr	<0.008		K by AA	0.287 mg/L

3.3 ARP RESULTS

3.3.1 MST Strike for the ARP

The Tank 49H material was tested to determine if it would process correctly in the ARP.

First MST Strike Plutonium Results

For the first MST strike, researchers analyzed the filtered samples for ^{238}Pu. Table 13 shows the plutonium results while Figure 9 shows the graphical results for ^{238}Pu. The ^{238}Pu data is more useful than the $^{239/40}$Pu as the former is not limited by detection limit values.

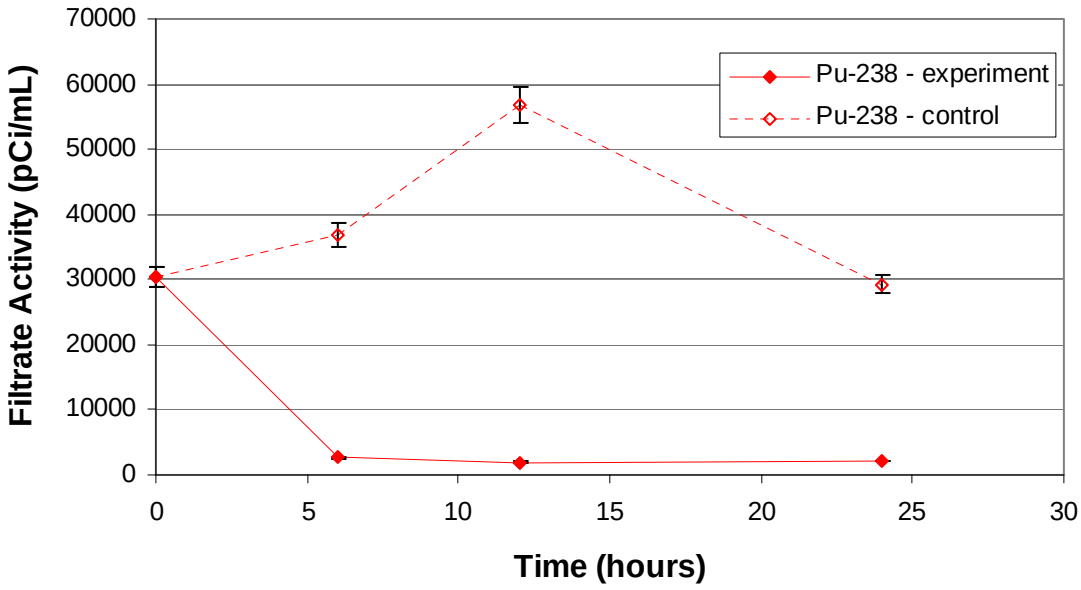
Table 13. ^{238}Pu Concentrations in the First MST Strike Filtrates

Time (hours)	Experiment ^{238}Pu, pCi/mL	Control ^{238}Pu, pCi/mL
0*	3.05(±0.148)E+04	3.05(±0.148)E+04
6	2.72(±0.147)E+03	3.69(±0.184)E+04
12	1.93(±0.106)E+03	5.68(±0.281)E+04
24	2.18(±0.172)E+03	2.93(±0.145)E+04

*The time = 0 data are the same data point.

The uncertainty is the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

Figure 9. ^{238}Pu in Solution over Time for the First MST Strike



Both the 6 and, especially, the 12 hour control samples showed notable deviation from the expected behavior. This increase in filtrate activity also appears in the cesium data (and is probably present in the strontium data even though the values are not statistically significant), indicating that this deviation is most likely not due to an analytical error. The errors most likely lie either in the handling of the sample bottle, or from fine Pu solids; it does not appear to be due to loss of sample diluent acid (before the sample addition), or from contamination from the high activity cells.

Table 14 lists the decontamination factors (DF) after the first MST strike.

Table 14. ^{238}Pu Decontamination Factors over Time

Time (hours)	Experiment	Control
	DF	DF
6	11.2	0.84
12	15.9	0.54
24	14.0	1.04

The plutonium shows excellent DF behavior at all times and are in good agreement with pretest predictions of near 11 (see Appendix C). The pretest work was based on models that had been benchmarked to a wide variety of data.[32] The slight decline in plutonium DF from 12 to 24 hours is most likely due to experimental and analytical variances, and not desorption of plutonium from the MST.

Second MST Strike Plutonium Results

For the second MST strike (on the control), researchers analyzed the filtered samples for ^{238}Pu. Table 15 shows the plutonium results while Figure 10 shows the graphical results for ^{238}Pu. Again, ^{238}Pu data was used to avoid detection limit values.

Table 15. Plutonium-238 Concentrations in the Second MST Strike Filtrates

Time (hours)	Second Strike
	^{238}Pu, pCi/mL
0	2.96(±0.158)E+04
6	2.10(±0.139)E+03
12	1.77(±0.117)E+03
24	1.40(±0.0841)E+03

The uncertainty is the analytical uncertainty associated with the measurement and does not include errors from experimental and sampling protocols. Note that the initial plutonium concentration is not significantly different from the initial concentration measured in the prior test (i.e., Table 14) approximately three weeks earlier. This finding suggests negligible precipitation of actinides occurred upon addition of dilution water.

Figure 10. ^{238}Pu in Solution over Time for the Second MST Strike

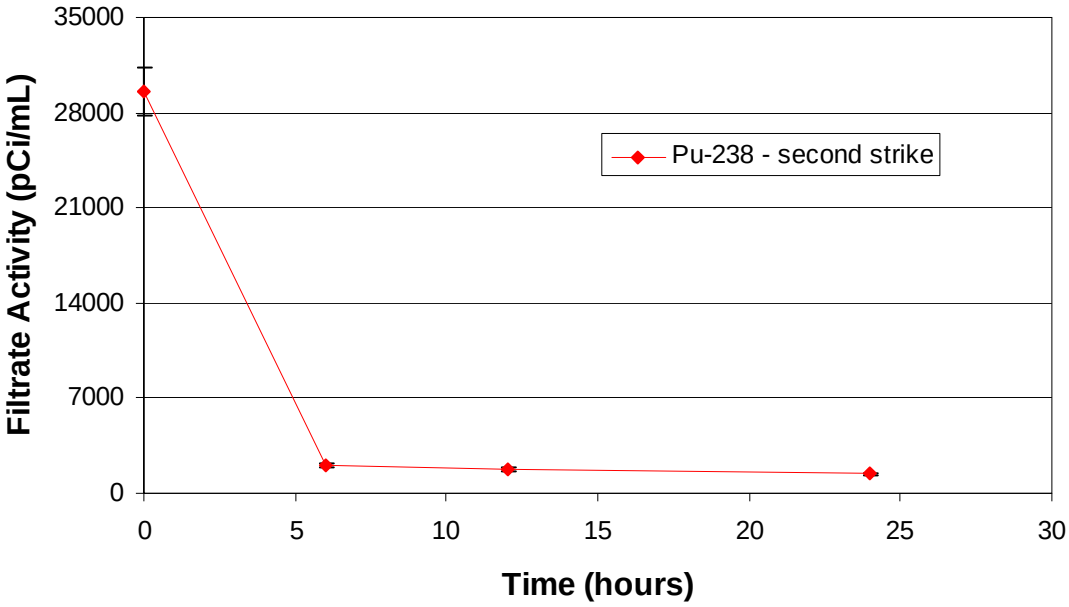


Table 16 lists the DF values after the second MST strike. The values are greater than those of the first experiment but likely within analytical uncertainty. Lower values may also indicate settling of a minor amount of particulate fines containing Pu between experiments. As with the first MST strike, the plutonium shows excellent DF behavior at all times.

Table 16. ^{238}Pu Decontamination Factors over Time

Time (hours)	DF
6	14.1
12	16.7
24	21.1

First MST Strike Strontium Results

For the first MST strike, researchers analyzed the filtered samples for ^{90}Sr. Table 17 shows the strontium results while Figure 11 shows the graphical results for ^{90}Sr.

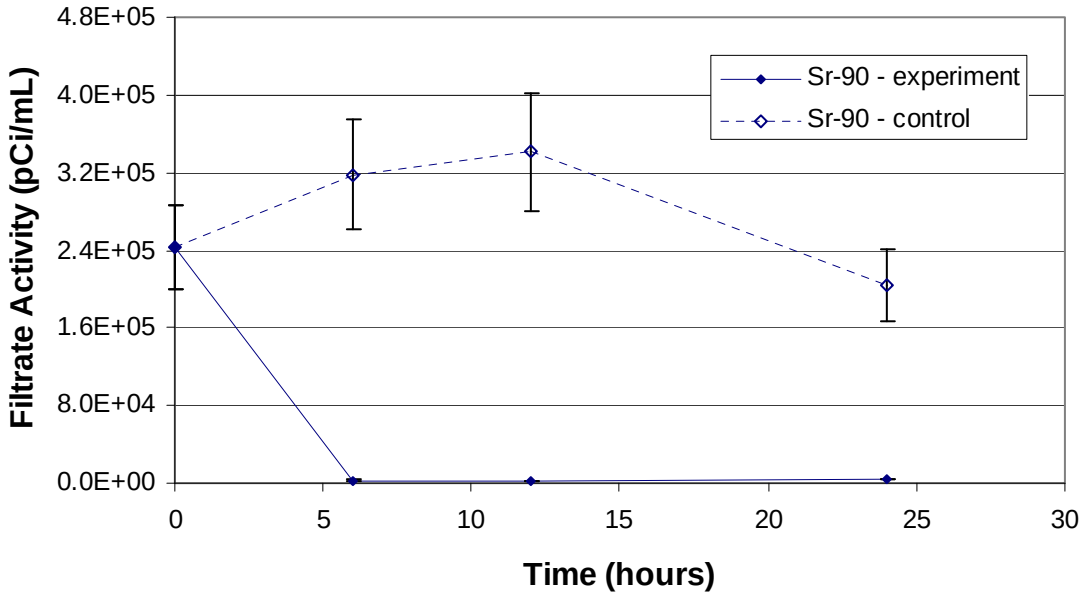
Table 17. ^{90}Sr Concentrations In the First MST Strike Filtrates

Time (hours)	Experiment	Control
	^{90}Sr (pCi/mL)	^{90}Sr, pCi/mL
0*	2.43(±0.411)E+05	2.43(±0.411)E+05
6	2.81(±0.512)E+03	3.18(±0.614)E+05
12	1.72(±0.343)E+03	3.41(±0.553)E+05
24	4.10(±0.749)E+03	2.03(±0.338)E+05

*The time = 0 data are the same data point.

The uncertainty is the analytical uncertainty associated with the measurement.

Figure 11. ^{90}Sr in Solution over Time for the First MST Strike



As with the plutonium data, the 6 and 12 hour control points are showing unexpected behavior. The other observation that merits comment is the slight increase in filtrate activity at the final data point. This seems to be a real effect, and is behavior SRNL has observed before.[33] Even with the slight increase, the strontium DF values are still very good.

Table 18 lists the DF values after the first MST strike.

Table 18. ^{90}Sr Decontamination Factors over Time

Time (hours)	Experiment	Control
	DF	DF
6	86.5	0.765
12	141	0.713
24	59.4	1.20

The strontium shows reasonable DF behavior at all times, even when one considers the slight increase in filtrate activity at the 24 hour data point. These values are in good agreement with the pretest predictions (i.e., DF near 63) using models (see Appendix C).

Second MST Strike Strontium Results

For the second MST strike (on the control), researchers analyzed the filtered samples for ^{90}Sr. Table 19 shows the strontium results while Figure 12 shows the graphical results for ^{90}Sr.

Table 19. ^{90}Sr Concentrations in the Second MST Strike Filtrates

Time (hours)	Second Strike ^{90}Sr, pCi/mL
0	1.42(±0.264)E+05
6	≤2.28E+03
12	≤1.47E+03
24	≤2.61E+03

The uncertainty is the analytical uncertainty associated with the measurement.

Data listed as "≤" in the table means the value was an upper limit value. For the same reason, data points in Figure 12 that have an asterisk next to them are the upper limit data points.

Figure 12. ^{90}Sr in Solution over Time for the Second MST Strike

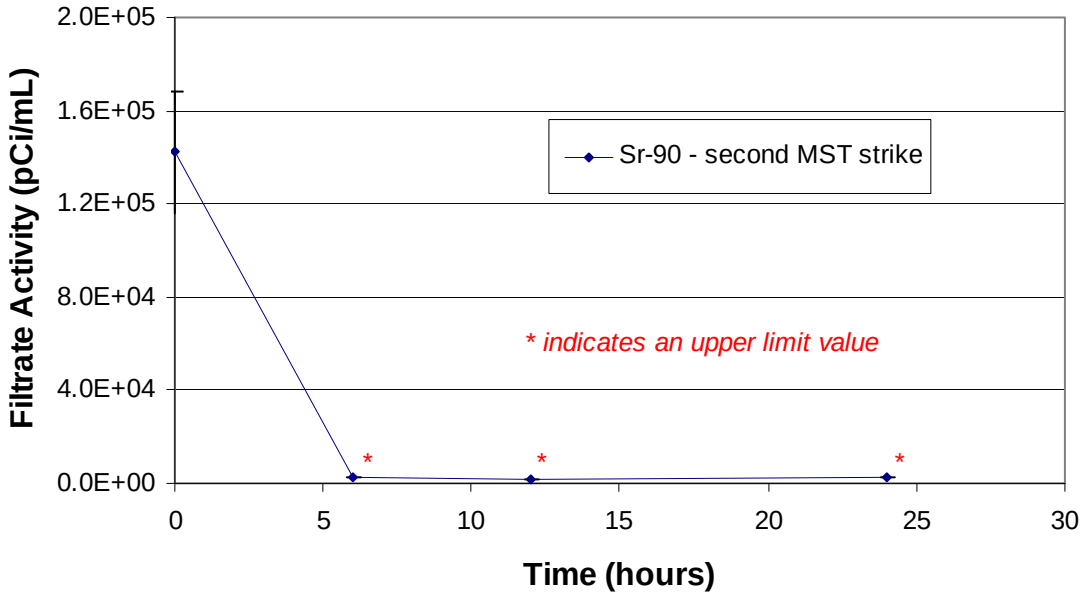


Table 20 lists the DF values after the second MST strike.

Table 20. ^{90}Sr Decontamination Factors over Time

Time (hours)	DF
6	≥62.3
12	≥96.9
24	≥54.4

As with the first MST strike, the strontium shows acceptable DF behavior at all times, comparable to the results from the first MST strike. The slight increase in filtrate activity at the final data point seems to be a real effect, and something SRNL has observed before.[33] Even with the slight increase, the strontium DF values are still very good.

Note that the initial strontium concentration is significantly different (~58%) from the control concentrations measured in the prior test (i.e., Table 17) approximately three weeks earlier. This finding suggests that the control bottle was not agitated enough to prevent settling of insoluble strontium solids (which would then not be captured for analysis). We do know that insoluble solids were present due to the difference in unfiltered samples (Table 4, 3.92E+05 pCi/mL) and the average filtered data point from the first MST strike (Table 17, 2.55E+05 pCi/mL).

First MST Strike Cesium Results

Researchers analyzed the filtered solutions for ^{137}Cs. Table 21 shows the numerical values while Figure 13 shows the graphical results. While MST has nominally has no effect on cesium, the filtrate cesium serves as a tracker for contamination as we do not expect the cesium to change.

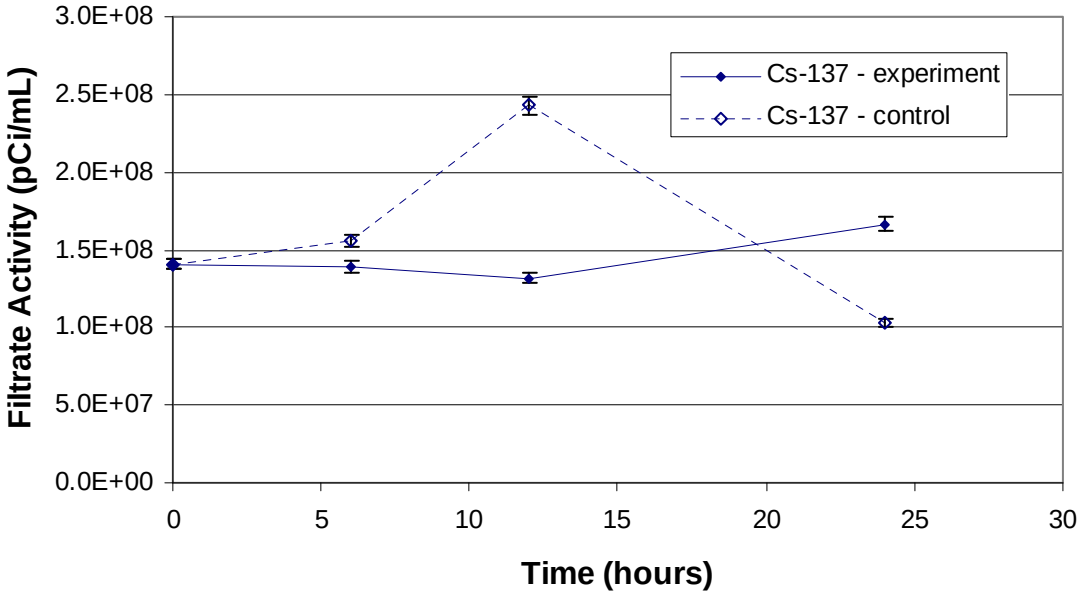
Table 21. ^{137}Cs Concentrations in the First MST Strike Filtrate

Time (hours)	Experiment	Control
	^{137}Cs pCi/mL	^{137}Cs, pCi/mL
0*	6.33(±0.152)E+07	6.33(±0.152)E+07
6	6.26(±0.150)E+07	7.02(±0.162)E+07
12	5.93(±0.147)E+07	1.09(±0.0210)E+08
24	7.49(±0.166)E+07	4.61(±0.126)E+07

* The time = 0 data are the same data point.

The uncertainty is the analytical uncertainty associated with the measurement.

Figure 13. ^{137}Cs in Solution over Time



As with the plutonium data, some of the control points show unexpected behavior, although in this case, it is the 12 and 24 hour points. Table 22 lists the DF values after the second MST strike.

Table 22. ^{137}Cs Decontamination Factors over Time

Time (hours)	Experiment	Control
	DF	DF
6	1.01	0.902
12	1.07	0.579
24	0.845	1.37

Second MST Strike Cesium Results

For the second MST strike (on the control), researchers analyzed the filtered solutions for ^{137}Cs. Table 23 shows the numerical values while Figure 14 shows the graphical results.

Table 23. ^{137}Cs Concentrations in the First MST Strike Filtrate

Time (hours)	Second MST Strike
	^{137}Cs, pCi/mL
0*	6.98(±0.0970)E+07
6	6.17(±0.0882)E+07
12	6.48(±0.092)E+07
24	6.17(±0.0888)E+07

* The time = 0 data are the same data point.

The uncertainty is the analytical uncertainty associated with the measurement.

Figure 14. ^{137}Cs in Solution over Time

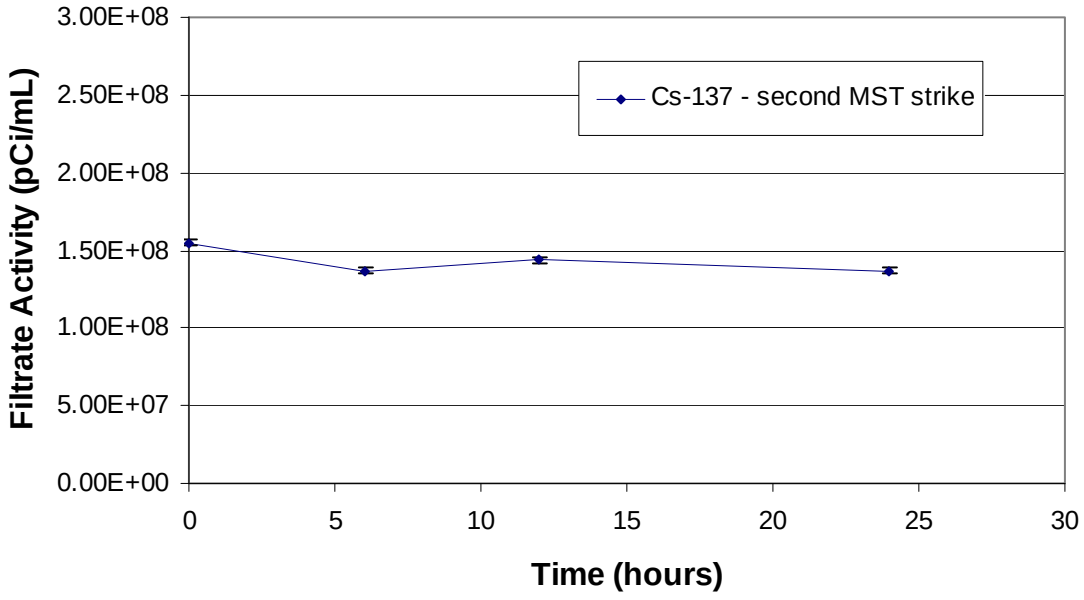


Table 24 lists the DF values after the second MST strike.

Table 24. ^{137}Cs Decontamination Factors over Time

Time (hours)	DF
6	1.13
12	1.08
24	1.13

3.3.2 Dissolved MST Samples after Filtration

Two MST sludge/slurries were processed in this work as discussed in the previous section. The first material was from an experiment having a separate control bottle. The first MST/sludge recovered from the filter was dissolved in-cell using a solution of nitric acid and hydrogen peroxide. The second MST/sludge sample was dissolved out-of-cell using an acid/hydrogen peroxide mixture in an attempt to provide a more concentrated sample preparation.

Tables 25 to 30 on the following pages provide the analyses of these two MST/sludge samples. The differences in sample handling led to the differing units of reporting the results. The first MST/sludge is reported in mg/L or pCi/mL of liquid (volume basis) from the in-cell dissolution. The second MST/sludge sample is reported on the basis of grams of slurry that was provided to the out-of-cell method (mass basis). The direct mass of solids for the samples is unknown in both cases. The samples were very small and could only in practice be prepared once. The basis for these analyses is the known titanium from the added MST. Individual results for each MST sample were determined by comparing the ratio of the individual element to titanium.

Table 25. First MST Sludge/Slurry – ICP-ES and AA Results, Acid/peroxide Prep (mg/L)

Element	First Analysis	Second Analysis	Third Analysis	Average mg/L	1-sigma, mg/L
Ag	<0.96	<0.96	<0.96	<0.96	
Al	260	261	261	261	1
B	<0.25	<0.25	<0.25	<0.25	
Ba	0.124	0.106	0.110	0.113	0.009
Be	<0.01	<0.01	<0.01	<0.01	
Ca	<0.80	<0.80	<0.80	<0.80	
Cd	<0.08	<0.08	<0.08	<0.08	
Ce	<2.29	<2.29	<2.29	<2.29	
Cr	1.36	1.36	1.35	1.36	0.01
Cu	<0.17	<0.17	<0.17	<0.17	
Fe	1.09	1.19	1.14	1.14	0.05
Gd	<0.35	<0.35	<0.35	<0.35	
K	<4.79	<4.79	<4.79	<4.79	
La	<0.43	<0.43	<0.43	<0.43	
Li	<0.29	<0.29	<0.29	<0.29	
Mg	0.067	0.064	0.066	0.065	0.002
Mn	0.125	0.129	0.123	0.13	
Mo	<0.32	<0.32	<0.32	<0.32	
Na	1130	1100	1110	1110	15
Ni	<0.23	<0.23	<0.23	<0.23	
P	2.85	3.22	3.30	3.12	0.24
Pb	<0.83	<0.83	<0.83	<0.83	
S	35.2	33.5	30.6	33	2.3
Sb	<1.58	<1.58	<1.58	<1.58	
Si	<2.51	<2.51	<2.51	<2.51	
Sn	<4.70	<4.70	<4.70	<4.70	
Sr	<0.42	<0.42	<0.42	<0.42	
Ti	74.9	75.0	74.8	74.9	0.12
U	<19.10	<19.10	<19.10	<19.10	
V	<0.18	<0.18	<0.18	<0.18	
Zn	<1.03	<1.03	<1.03	<1.03	
Zr	<0.11	<0.11	<0.11	<0.11	
Mercury	<0.11	<0.11	<0.11	<0.11	
AA Arsenic	<0.0275	<0.0275	<0.0275	<0.0275	
AA Selenium	<0.055	<0.055	<0.055	<0.055	

Table 26. First MST Sample, MS Data (mg/L or pCi/mL)

Method	First	Second Result	Third Result	Average	1 sigma, units
MS, (stable) ^{59}Co	1.55E-02	1.57E-02	1.63E-02	1.58 E-02	0.042 E-02 mg/L
MS, Total Rb	8.99E-03	7.54E-03	8.9E-03	8.48 E-03	0.809 E-03 mg/L
MS, Mass 99	1.44E-01	1.40E-01	1.45E-01	1.43 E-01	0.0265 E-01 mg/L
MS, Mass 99				2.42 E+03	4.49 E+01 pCi/mL
MS, Mass 133	2.37E-02	2.15E-02	2.17E-02	2.23 E-02	1.22 E-03 mg/L
MS, Mass 135	3.45E-03	3.19E-03	2.97E-03	3.20 E-03	0.24 E-03 mg/L
MS, Mass 137	9.25E-02	9.22E-02	9.40E-02	9.29 E-02	0.0964 E-02 mg/L
MS total lead (Pb)	6.32E-02	6.52E-02	6.12E-02	6.32 E-02	0.2 E-02 mg/L
MS ^{233}U	1.02E-03	1.64E-03	1.24E-03	1.30 E-03	0.314 E-03 mg/L
MS ^{233}U				1.26 E+01	3.04 pCi/mL
MS ^{234}U	7.99E-03	8.20E-03	8.50E-03	8.23 E-03	0.256 E-03 mg/L
MS ^{234}U				5.14 E+01	1.60 pCi/mL
MS ^{235}U	2.17E-02	2.14E-02	2.34E-02	2.22 E-02	0.108 E-02 mg/L
MS ^{235}U				4.80E-02	.233E-02 pCi/mL
MS ^{236}U	8.82E-03	8.33E-03	9.74E-03	8.96 E-03	0.715 E-03 mg/L
MS ^{236}U				5.80 E-01	0.463 E-02 pCi/mL
MS ^{238}U	3.79E-01	3.77E-01	3.77E-01	3.78 E-01	0.012 E-01 mg/L
MS ^{238}U				1.27 E-01	4.03 E-04 pCi/mL
MS ^{237}Np	1.14E-02	9.16E-03	1.13E-02	1.06 E-02	0.127 E-02 mg/L
MS ^{237}Np				7.47	0.895 pCi/mL
MS ^{230}Th	<2.50E-03	<2.50E-03	<2.50E-03	<2.50E-03	mg/L
MS ^{230}Th				<5.27 E+01	pCi/mL
MS ^{232}Th	<4.00E-03	<4.00E-03	<4.00E-03	<4.00E-03	mg/L
MS ^{232}Th				<4.39 E-04	pCi/mL
MS ^{239}Pu	<1.50E-03	2.24E-03	<1.50E-03	2.24E-03	single value mg/L
MS ^{239}Pu				1.39 E+02	pCi/mL
MS ^{240}Pu	<1.50E-03	<1.50E-03	<1.50E-03	<1.50E-03	mg/L
MS ^{240}Pu				<3.42 E+02	pCi/mL
MS ^{241}Pu, ^{241}Am	<1.00E-03	<1.00E-03	<1.00E-03	<1.00E-03	mg/L
MS ^{242}Pu	<1.50E-03	<1.50E-03	<1.50E-03	<1.50E-03	mg/L
MS ^{242}Pu				<5.73	pCi/mL
MS ^{243}Am, ^{243}Cm	<2.00E-03	<2.00E-03	<2.00E-03	<2.00E-03	mg/L
MS ^{244}Pu, ^{244}Cm	<1.50E-03	<1.50E-03	<1.50E-03	<1.50E-03	mg/L

Analysis of ^{14}C and ^{129}I for the MST/sludge samples was found to be not possible. The small amounts of samples dictated one preparation per sample, and preparations using acid were required to dissolve the other analytes. Acid is known to drive off some carbon and iodine so that recoveries of ^{14}C and ^{129}I would be significantly less than 100% and would not be known.

Table 27. First MST Sample, Radiochemical Data (pCi/mL)

Isotope	First Analysis	Second Analysis	Third Analysis	Average, pCi/mL	1-sigma, pCi/mL
Gamma, ^{137}Cs	6.40E+05	6.67E+05	6.62E+05	6.56E+05	1.4E+04
^{137m}Ba	Calculated from ^{137}Cs			6.20E+05	1.3E+04
Gamma, ^{134}Cs	<2.05E+03	<4.15E+03	<4.50E+03	<2.05E+03	
Gamma, ^{94}Nb	<1.17E+01	<1.21E+01	<1.17E+01	<1.18E+01	
Gamma, ^{106}Ru	<7.88E+01	<7.93E+01	<8.20E+01	<7.88E+01	
Gamma, ^{125}Sb	<4.14E+01	<4.18E+01	<3.90E+01	<3.90E+01	
Gamma, ^{126}Sb	<1.34E+01	<1.42E+01	<1.34E+01	<1.34E+01	
Gamma, ^{126}Sn	<6.49E+01	<6.40E+01	<6.44E+01	<6.40E+01	
Gamma, ^{144}Ce	<1.36E+02	<1.38E+02	<1.40E+02	<1.36E+02	
Gamma, ^{154}Eu	4.00E+01	4.59E+01	4.32E+01	4.30E+01	3.0E+00
Gamma, ^{155}Eu	<7.21E+01	<7.12E+01	<7.16E+01	<7.12E+01	
Gamma, ^{226}Ra	<3.20E+02	<4.03E+02	<2.84E+02	<2.84E+02	
Gamma, ^{241}Am*	1.60E+02	<1.55E-02	2.30E+02	1.95E+02	4.9E+01
Gamma, ^{60}Co	<1.31E+01	<1.33E+01	<1.22E+01	<1.22E+01	
Am/Cm ^{241}Am	2.35E+02	3.13E+02	<2.32E+02	2.60E+02*	4.6E+01
Am/Cm ^{243}Am	<5.54E+01	<1.84E+01	<1.47E+02	<1.84E+01	
Am/Cm ^{242m}Am	<4.95E+01	<1.95E+02	9.91E+01	<4.95E+01*	
Am/Cm ^{243}Cm	<1.69E+02	<5.27E+01	<5.50E+02	<5.27E+01	
Am/Cm ^{245}Cm	<1.39E+02	<4.34E+01	<4.50E+02	<4.34E+01	
Am/Cm ^{247}Cm	<2.59E+02	<7.03E+01	<7.39E+02	<7.03E+01	
Am/Cm ^{249}Cf	<2.80E+02	<8.20E+01	<8.11E+02	<8.20E+01	
Am/Cm ^{251}Cf	<1.67E+02	<5.00E+01	<5.09E+02	<5.00E+01	
Am/Cm ^{242}Cm	<3.97E+00	<5.45E-01	5.59E+00	<5.45E-01*	
Am/Cm ^{244}Cm	4.10E+01	1.61E+02	8.20E+01	9.46E+01	6.1E+01
^{238}Pu	1.65E+04	1.72E+04	1.68E+04	1.68E+04	3.4E+02
$^{239\text{-}240}$Pu*	<1.29E+02	<1.49E+02	<7.07E+01	<1.16E+02	
^{241}Pu	2.04E+03	2.21E+03	2.10E+03	2.12E+03	8.9E+01
Total Alpha	<9.41E+03	<9.41E+03	<1.11E+04	<9.41E+03	
Total Beta	1.12E+06	1.09E+06	1.09E+06	1.10E+06	1.7E+04
Tritium	<2.58E+02	<2.58E+02	<2.58E+02	<2.58E+02	
^{99}Tc	2.50E+03	2.48E+03	2.49E+03	2.49E+03	9.0E+00
^{147}Pm	<1.11E+03	<1.61E+02	<1.92E+02	<4.87E+02	
^{151}Sm	<7.75E+02	<1.12E+02	<7.84E+01	<3.22E+02	
^{90}Sr (^{90}Y)	1.50E+05	1.59E+05	1.60E+05	1.56E+05	5.4E+03

*Analyses have data at or near detection limit.

Data for the second MST sample are in units of micrograms per gram (μg/g) of MST/sludge slurry. Titanium mass is again the key for relating content to MST level.

Table 28. Second MST Sludge/Slurry – ICP-ES and AA Results (μg/g)

Element	First Analysis	Second Analysis	Third Analysis	Average (μg/g)	1-sigma, (μg/g)
Ag	<7.13	<7.16	<7.05	<7.05	
Al	893	873	896	887.3	12.5
B	<18.30	<18.30	<18.10	<18.10	
Ba	2.92	2.23	3.32	2.823	0.551
Be	<0.15	<0.15	<0.15	<0.15	
Ca	<19.60	<19.70	<19.40	<19.40	
Cd	<2.01	<2.02	<1.99	<1.99	
Ce	<80.90	<81.30	<80.00	<80.00	
Cr	22.4	18.8	25.0	22.067	3.113
Cu	<4.04	<4.06	<4.00	<4.00	
Fe	33.8	26.9	37.0	32.57	5.16
Gd	<8.51	<8.55	<8.42	<8.42	
K	<117.00	<118.00	<116.00	<116.00	
La	<10.40	<10.50	<10.30	<10.30	
Li	<7.21	<7.25	<7.14	<7.14	
Mg	1.63	1.37	2.00	1.6667	0.3166
Mn	3.74	2.92	4.19	3.617	0.644
Mo	<7.78	<7.82	<7.70	<7.70	
Na	1.40E+04	1.38E+04	1.42E+04	1.40E+04	2.00E+02
Ni	<12.50	<12.60	<12.40	<12.40	
P	40.4	43.5	40.3	41.4	1.82
Pb	<20.20	<20.30	<20.00	<20.00	
S	444.0	360.0	287.0	363.7	78.6
Sb	<38.70	<38.90	<38.30	<38.30	
Si	<71.70	<72.00	<70.90	<70.90	
Sn	<115.00	<116.00	<114.00	<114.00	
Sr	<10.30	<10.30	<10.20	<10.20	
Ti	2660	2030	2990	2560	488
U	<468	<470	<463	<463.	
V	<4.46	<4.48	<4.42	<4.42	
Zn	<3.45	<3.47	<3.63	<3.45	
Zr	<4.98	<5.00	<4.93	<4.93	
Mercury	<2.694	<2.708	<2.667	<2.690	
AA Arsenic	<0.674	<0.677	<0.667	<0.672	
AA Selenium	<1.347	<1.354	<1.333	<1.345	

Table 29. Second MST MS Data Table (μg/g or pCi/g)

Method	First Result	Second Result	Third Result	Average	1 sigma, units
MS, (stable) ^{59}Co	2.20E-01	2.22E-01	2.18E-01	2.20 E-01	0.017 E-01 μg/g
MS, Total Rb	1.93E-01	2.13E-01	<1.45E-01	2.03 E-01	0.14 E-01 μg/g
MS, Mass 99 (^{99}Tc)	1.93E+00	1.80E+00	2.10E+00	1.94 E+00	0.15 E+00 μg/g
MS, Mass 99 (^{99}Tc)				3.29 E+04	0.254 E+04 pCi/g
MS, Mass 133 (^{133}Cs)	3.17E-01	3.75E-01	2.98E-01	3.30 E-01	0.401 E-01 μg/g
MS, Mass 135	<5.76E-01	<5.79E-01	<5.70E-01	<5.70E-01	μg/g
MS, Mass 137	2.76E+00	2.01E+00	3.04E+00	2.60 E+00	0.53 E+00 μg/g
MS total lead	2.57E+00	1.99E+00	2.95E+00	2.50 E+00	0.49 E+00 μg/g
MS ^{233}U	4.60E-02	4.70E-02	6.90E-02	5.40 E-02	1.30E-02 μg/g
MS ^{233}U				5.23 E+02	1.26 E+02 pCi/g
MS ^{234}U	3.24E-01	2.44E-01	3.68E-01	3.12 E-01	0.63E-01 μg/g
MS ^{234}U				1.95 E+03	3.94 E+02 pCi/g
MS ^{235}U	8.44E-01	6.38E-01	9.64E-01	8.15 E-01	1.65E-01 μg/g
MS ^{235}U				1.76	0.357 pCi/g
MS ^{236}U	3.04E-01	2.46E-01	3.61E-01	3.04 E-01	0.58E-01 μg/g
MS ^{236}U				1.97 E+01	0.375 E+01 pCi/g
MS ^{238}U	1.40E+01	1.11E+01	1.58E+01	1.36 E+01	0.237E+01 μg/g
MS ^{238}U				4.57	0.797 pCi/g
MS ^{237}Np	2.95E-01	2.38E-01	3.46E-01	2.93 E-01	0.54E-01 μg/g
MS ^{237}Np				2.07 E+02	0.381 E+02 pCi/g
MS ^{230}Th	<6.12E-02	<6.15E-02	<6.06E-02	<6.06E-02	μg/g
MS ^{230}Th				<1.28 E+03	pCi/g
MS ^{232}Th	<9.8E-02	<9.85E-02	<9.70E-02	<9.70E-02	μg/g
MS ^{232}Th				<1.06 E-02	pCi/g
MS ^{239}Pu	6.30E-02	4.80E-02	8.30E-02	6.47 E-02	1.76E-02 μg/g
MS ^{239}Pu				4.02 E+03	1.09 E+03 pCi/g
MS ^{240}Pu	<3.67E-02	<3.69E-02	<3.64E-02	<3.64E-02	μg/g
MS ^{240}Pu				<8.30 E+03	pCi/g
MS ^{241}Pu, ^{241}Am	<2.45E-02	<2.46E-02	<2.42E-02	<2.42E-02	μg/g
MS ^{242}Pu	<3.67E-02	<3.69E-02	<3.64E-02	<3.64E-02	μg/g
MS ^{242}Pu				<1.39 E+02	pCi/g
MS ^{243}Am, ^{243}Cm	<4.90E-02	<4.92E-02	<4.85E-02	<4.85E-02	μg/g
MS ^{244}Pu, ^{244}Cm	<3.67E-02	<3.69E-02	<3.64E-02	<3.64E-02	μg/g

Table 30. Second MST Sample, Radiochemical Data (pCi/g)

Isotope	First Analysis	Second Analysis	Third Analysis	Average, pCi/g	1 sigma, pCi/g
Gamma, ^{137}Cs	8.83E+06	8.74E+06	8.65E+06	8.74E+06	9.0E+04
^{137m}Ba	Calculated from ^{137}Cs			8.26E+06	8.5E+04
Gamma, ^{134}Cs	<1.07E+05	<1.14E+05	<1.01E+05	<1.01E+05	
Gamma, ^{94}Nb	<2.89E+02	<3.02E+02	<3.10E+02	<3.00E+02	
Gamma, ^{106}Ru	<2.06E+03	<2.02E+03	<2.45E+03	<2.02E+03	
Gamma, ^{125}Sb	<1.05E+03	<1.00E+03	<1.04E+03	<1.00E+03	
Gamma, ^{126}Sb	<3.64E+02	<3.51E+02	<3.72E+02	<3.51E+02	
Gamma, ^{126}Sn	<1.76E+03	<1.57E+03	<1.88E+03	<1.57E+03	
Gamma, ^{144}Ce	<3.69E+03	<3.47E+03	<3.98E+03	<3.47E+03	
Gamma, ^{154}Eu	1.46E+03	1.38E+03	2.08E+03	1.64E+03	3.8E+02
Gamma, ^{155}Eu	<1.96E+03	<1.75E+03	<2.09E+03	<1.75E+03	
Gamma, ^{226}Ra	<1.01E+04	<8.11E+03	<1.07E+04	<8.11E+03	
Gamma, ^{241}Am	<4.20E+03	<3.78E+03	<4.50E+03	<3.78E+03	
Gamma, ^{60}Co	<3.28E+02	<3.45E+02	<3.15E+02	<3.45E+02	
Am/Cm ^{241}Am	1.73E+03	1.42E+03	1.89E+03	1.68E+03	2.4E+02
Am/Cm ^{243}Am	<1.50E+02	<1.40E+02	<1.77E+02	<1.55E+02	
Am/Cm ^{242m}Am	<4.95E+00	<5.50E+00	<6.62E+00	<5.69E+00	
Am/Cm ^{243}Cm	<5.00E+02	<4.50E+02	<5.45E+02	<4.98E+02	
Am/Cm ^{245}Cm	<4.12E+02	<3.70E+02	<4.48E+02	<4.10E+02	
Am/Cm ^{247}Cm	<8.15E+02	<6.98E+02	<9.10E+02	<8.08E+02	
Am/Cm ^{249}Cf	<9.10E+02	<7.66E+02	<9.77E+02	<8.84E+02	
Am/Cm ^{251}Cf	<4.68E+02	<4.25E+02	<5.32E+02	<4.75E+02	
Am/Cm ^{242}Cm	<4.10E+00	<4.55E+00	<5.50E+00	<4.72E+00	
Am/Cm ^{244}Cm	9.91E+02	7.34E+02	1.25E+03	9.92E+02	
^{238}Pu	5.32E+05	4.12E+05	5.95E+05	5.13E+05	9.3E+04
$^{239\text{-}240}$Pu	5.32E+03	4.04E+03	5.63E+03	5.00E+03	8.4E+02
^{241}Pu	6.44E+04	5.18E+04	7.52E+04	6.38E+04	1.2E+04
Total Alpha	<2.93E+05	<2.43E+05	<5.63E+05	<2.43E+05	
Total Beta	1.90E+07	2.09E+07	2.07E+07	2.02E+07	1.0E+06
Tritium	<6.12E+02	<6.15E+02	<6.06E+02	<6.06E+02	
^{99}Tc	2.88E+04	2.37E+04	2.92E+04	2.72E+04	3.1E+03
^{147}Pm	<4.02E+03	<3.21E+03	<3.68E+03	<3.63E+03	
^{151}Sm	<4.21E+03	<4.17E+03	<5.05E+03	<4.48E+03	
^{90}Sr (^{90}Y)	5.05E+06	3.17E+06	5.90E+06	4.71E+06	1.4E+06

3.3.3 Activity Balance in the ARP Test

With the digested MST data in hand, we can compare the results to the Suite-A data (Table 4) to confirm that the plutonium and strontium activity going into the test (from the Tank 49H solution) is the same as what we analyze in the output (combined MST and CSS streams).

From the MST solids digestion, the amount of plutonium and strontium sorbed by the MST can be calculated. In Table 31, the first MST digestion results are listed.

Table 31. Plutonium and Strontium Sorbed on MST from First Digestion Results

	Ti	MST	^{238}Pu	^{90}Sr	units
Digested Material	74.9	156	9.82E-04	1.14E-03	mg/L
normalized to MST	0.479	1	6.28E-06	7.29E-06	g/g
g Pu or Sr			5.14E-07	5.95E-07	g
μg Pu or Sr			5.14E-01	5.95E-01	μg

Due to the fact that MST collection is not perfect, the plutonium and strontium results are normalized to the titanium results (giving gram of Pu/Sr per gram of MST). The normalized values are then multiplied by the amount of MST solids introduced (0.0818 g for test #1) to give the grams and micrograms of plutonium and strontium sorbed onto the MST.

For the digestion of the second MST strike, the results are displayed in Table 32.

Table 32. Plutonium and Strontium Sorbed on MST from Second Digestion Results

	Ti	MST	^{238}Pu	^{90}Sr	units
Digested Material	2560	5344	3.00E-02	3.44E-02	μg/g
normalized to MST	0.479	1	5.61E-06	6.43E-06	g/g
g Pu or Sr			5.01E-07	5.74E-07	g
μg Pu or Sr			5.01E-01	5.74E-01	μg

From the CSS results (Table 38), we can calculate how many μg of Pu and Sr are in the CSS stream (Table 33).

Table 33. Plutonium and Strontium (mg) Left in the CSS Stream

CSS Component	pCi/mL	μg
^{238}Pu	1.56E+03	1.82E-02
^{90}Sr	2.74E+03	4.03E-03

The respective sums of ^{238}Pu and ^{90}Sr on the MST and the amounts in the CSS stream are given in Table 34.

Table 34. Respective Sums of ^{238}Pu and ^{90}Sr in the MST and CSS Streams

CSS Component	μg
^{238}Pu	5.19E-01
^{90}Sr	5.78E-01

The respective sums can then be compared to the amount of ^{238}Pu and ^{90}Sr entering the system through the Tank 49H solution, giving the activity balance (Table 35).

Table 35. Activity Balance for the First Test

CSS Component	Amount on MST (μg)	Amount in CSS Stream (μg)	Amount from Tank 49H (μg)	Balance (%)
^{238}Pu	0.514	0.0182	0.489	109
^{90}Sr	0.595	0.00403	0.576	104

The typical analytical uncertainty for the ^{238}Pu measurement is ~5%, and ~18% for ^{90}Sr. The activity balance is within the sum of the respective two analytical uncertainties (you are comparing two of the same measurement). Therefore, we can declare a complete activity balance for both ^{238}Pu and ^{90}Sr.

For the second test, we can perform the same type of calculation, using the data form the second MST strike and the same CSS analysis, although we need to adjust the amount in the CSS to account for the material removed in the first test (Table 36).

Table 36. Activity Balance for the Second Test

CSS Component	Amount on MST (μg)	Amount in CSS Stream (μg)	Amount from Tank 49H (μg)	Balance (%)
^{238}Pu	0.501	0.0160	0.489	106
^{90}Sr	0.574	0.00347	0.576	100

As with the first test, we can declare a complete activity balance for both ^{238}Pu and ^{90}Sr.

Detailed Analyses of CSS

The TTQAP specified extensive analysis of CSS. Table 37 through Table 41 below provides the requested data for the CSS from the first MST contact. We did not perform a set of analyses on the second test material.

Table 37. CSS ICP-ES Chemical Data, Acid Preparation (mg/L)

Element	First Analysis	Second Analysis	Third Analysis	Average mg/L	1 sigma, mg/L
Ag	<5.81	<5.81	<5.81	<5.81	
Al	8400	8420	8250	8360	95
B	<5.02	<5.02	<5.02	<5.02	
Ba	<0.892	<0.892	<0.892	<0.892	
Be	<0.13	<0.13	<0.13	<0.13	
Ca	<16.0	<16.0	<16.0	<16.0	
Cd	<1.64	<1.64	<1.64	<1.64	
Ce	<66.0	<66.0	<66.0	<66.0	
Cr	75.59	75.09	75.22	75.3	0.26
Cu	<3.30	<3.30	<3.30	<3.30	
Fe	<3.11	<3.11	<3.11	<3.11	
Gd	<6.93	<6.93	<6.93	<6.93	
K	222	227	216	222	5.6
La	<8.48	<8.48	<8.48	<8.48	
Li	<5.89	<5.89	<5.89	<5.89	
Mg	<0.687	<0.687	<0.687	<0.687	
Mn	<0.442	<0.442	<0.442	<0.442	
Mo	<10.2	<10.2	<10.2	<10.2	
Na	108100	108200	105600	107300	1500
Ni	<4.59	<4.59	<4.59	<4.59	
P	412	414	410	412	2
Pb	<16.5	<16.5	<16.5	<16.5	
S	4300	4370	4420	4370	63
Sb	<31.6	<31.6	<31.6	<31.6	
Si	<51.9	<51.9	<51.9	<51.9	
Sn	<94.0	<94.0	<94.0	<94.0	
Sr	<1.11	<1.11	<1.11	<1.11	
Ti	2.34	2.36	2.44	2.38	0.052
U	<381.1	<381.1	<381.1	<381.1	
V	<3.64	<3.64	<3.64	<3.64	
Zn	<20.5	<20.5	<20.5	<20.5	
Zr	<2.27	<2.27	<2.27	<2.27	

Table 38. CSS Radiochemical Data (pCi/mL)

Isotope	Prep	First Result	Second Result	Third Result	Average, pCi/mL	1 sigma, pCi/mL
Gamma, ^{137}Cs	2	6.36E+07	6.13E+07	6.19E+07	6.23E+07	1.2E+06
^{137m}Ba		Calculated from ^{137}Cs			5.89E+07	1.1E+06
Gamma, ^{134}Cs	2	<8.78E+04	<9.01E+04	<8.22E+04	<8.67E+04	
Gamma, ^{94}Nb	2	<2.78E+02	<2.67E+02	<2.72E+02	<2.73E+02	
Gamma, ^{106}Ru	2	<1.96E+03	<1.88E+03	<1.99E+03	<1.94E+03	
Gamma, ^{125}Sb	2	<8.10E+02	<8.61E+02	<8.10E+02	<8.27E+02	
Gamma, ^{126}Sb	2	<3.15E+02	<3.11E+02	<3.13E+02	<3.13E+02	
Gamma, ^{126}Sn	2	<4.51E+02	<4.61E+02	<6.30E+02	<5.14E+02	
Gamma, ^{144}Ce	2	<1.40E+03	<1.35E+03	<1.36E+03	<1.37E+03	
Gamma, ^{154}Eu	2	<3.75E+02	<3.67E+02	<3.64E+02	<3.69E+02	
Gamma, ^{155}Eu	2	<5.09E+02	<5.19E+02	<7.26E+02	<5.85E+02	
Gamma, ^{226}Ra	2	<4.19E+03	<5.36E+03	nr	<4.78E+03	
Gamma, ^{241}Am	2	<1.40E+03	<1.27E+03	nr	<1.33E+02	
Gamma, ^{60}Co	2	<2.96E+02	<3.03E+02	<3.30E+02	<3.10E+02	
^{232}U	2	7.13E+00	7.62E+00	7.19E+00	7.31E+00	2.70E-01
Am/Cm ^{241}Am	2	3.41E+02	3.44E+02	2.28E+02	3.04E+02	6.6E+01
Am/Cm ^{243}Am	2	<1.23E+02	<1.12E+02	<8.33E+01	<1.06E+02	
Am/Cm ^{242m}Am	2	<2.32E+02	<1.94E+02	<2.13E+02	<2.13E+02	
Am/Cm ^{243}Cm	2	<3.49E+02	<4.20E+02	<2.55E+02	<3.41E+02	
Am/Cm ^{245}Cm	2	<2.86E+02	<3.44E+02	<2.09E+02	<2.80E+02	
Am/Cm ^{247}Cm	2	<5.58E+02	<5.25E+02	<3.23E+02	<4.68E+02	
Am/Cm ^{249}Cf	2	<5.22E+02	<5.74E+02	<3.78E+02	<4.91E+02	
Am/Cm ^{251}Cf	2	<3.66E+02	<3.91E+02	<2.49E+02	<3.35E+02	
Am/Cm ^{242}Cm	2	<1.20E+01	<1.01E+01	<1.10E+01	<1.10E+01	
Am/Cm ^{244}Cm	2	5.22E+02	5.23E+02	3.68E+02	4.71E+02	8.9E+01
^{238}Pu (238/241)	2	1.54E+03	1.72E+03	1.42E+03	1.56E+03	1.5E+02
$^{239-240}$Pu (238/241)	2	<1.23E+02	<3.72E+02	1.23E+02	<2.06E+02	
^{241}Pu(238/241)	2	1.16E+03	1.27E+03	8.44E+02	1.09E+03	2.2E+02
Total alpha	2	1.60E+03	1.01E+03	<1.02E+03	1.31E+03	4.1E+02
Beta	2	7.43E+07	7.54E+07	7.43E+07	7.47E+07	6.5E+05
Cs-removed beta	2	9.51E+04	9.57E+04	9.34E+04	9.47E+04	1.23E+03
^{129}I	0	1.54E+01	1.57E+01	1.93E+01	1.68E+01	2.2E+00
^{14}C	0	8.69E+02	8.83E+02	7.93E+02	8.48E+02	4.9E+01
^{59}Ni	2	<3.69E+03	<5.29E+03	<2.48E+03	<3.82E+03	
^{61}Ni	2	<1.25E+03	<1.81E+03	<1.59E+03	<1.55E+03	
^{90}Sr (^{90}Y)	2	2.66E+03	2.62E+03	2.95E+03	2.74E+03	1.8E+02

nr = not reported

The pH of CSS is best estimated by free OH (pH >13 when free OH > 0.1 M). A free hydroxide level of 0.5 M corresponds to a pH of 13.7.

Table 39. CSS Chemical Data

Element	First Analysis	Second Analysis	Third Analysis	Average mg/L	1 sigma, mg/L
Fluoride	<504	<529	<515	<516	
Formate	<504	<529	<515	<516	
Chloride	<504	<529	<515	<516	
Nitrite	12200	12470	12460	12380	152
Bromide	<504	<529	<515	<516	
Nitrate	165410	167000	155500	162600	6240
Phosphate	<1009	<1057	<1030	<1032	
Sulfate	12100	12370	12360	12280	150
Oxalate	<1009	<1057	<1030	<1032	
Carbonate (TIC)	1.77E+04	1.68E+04	1.75E+04	1.73E+04	450
TOC	333	127	113	191	123
Total Base	1.43 M	1.42 M	1.42 M	1.42 M	0.008 M
Free OH	0.563 M	0.515 M	0.500 M	0.526 M	0.033 M
Other Base	0.343 M	0.715 M	0.510 M	0.522 M	0.186 M
Mercury	<2.20	<2.20	<2.20	<2.20	
AA Arsenic	<0.550	<0.550	<0.550	<0.550	
AA Selenium	<1.10	<1.10	<110	<1.10	
AA Potassium	220	226	231	226	6
VOA	<0.25	<0.25	<0.25	<0.25	
SVOA	<4	<4	<4	<4	
Cations TMA	<5040	<5300	<5150	<5160	
DBP	76	87	80	81	6

The MS data in Table 40 are results from five rather than three analyses. The raw data is provided in Table 41.

Table 40. CSS MS Data (mg/L or pCi/mL)

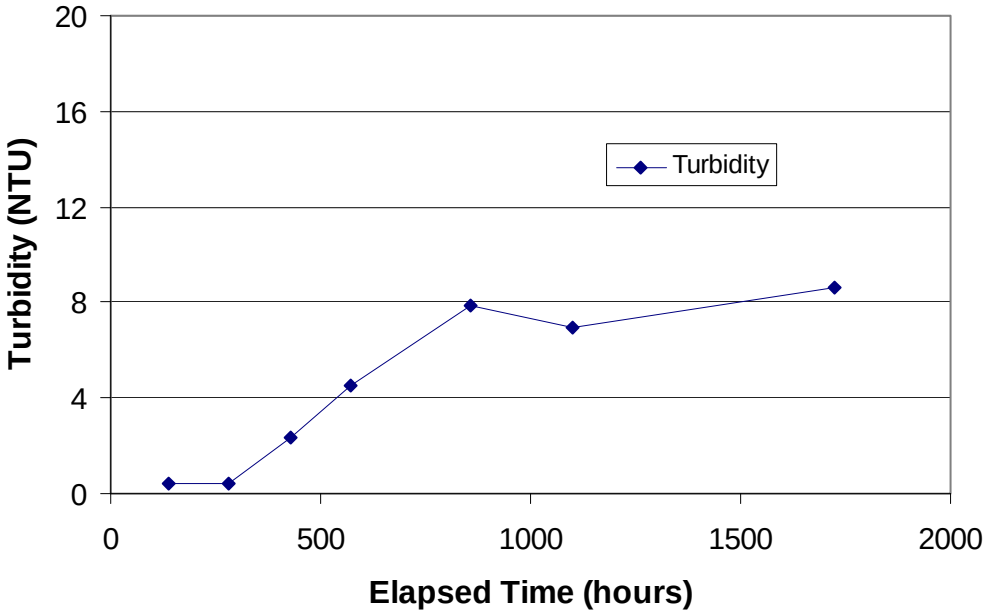
Method	Prep	Average	Standard deviation, 1-sigma, units
MS, (stable) ^{59}Co	2	1.04E-01	8.41E-03 mg/L
MS, Total Rb	2	8.68E-01	9.21E-02 mg/L
MS, Mass 99 (^{99}Tc)	2	4.39E+00	1.22E-01 mg/L
MS, Mass 99 (^{99}Tc)		7.44 E+04	0.207 E+04 pCi/mL
MS, Mass 133 (^{133}Cs)	2	2.01E+00	5.26E-02 mg/L
MS, Mass 135	2	2.97E-01	1.45E-02 mg/L
MS, Mass 137	2	7.93E-01	2.65E-02 mg/L
MS total lead (Pb)	2	1.59E-01	1.60E-02 mg/L
MS ^{233}U	2	<9.99E-03	mg/L
MS ^{233}U		<9.67 E+01	pCi/mL
MS ^{234}U	2	1.52E-02	1.85E-03 mg/L
MS ^{234}U		9.50 E+01	1.16 E+01 pCi/mL
MS ^{235}U	2	3.77E-02	3.58E-03 mg/L
MS ^{235}U		8.15 E-02	7.74 E-03 pCi/mL
MS ^{236}U	2	<2.50E-02	mg/L
MS ^{236}U		<1.62	pCi/mL
MS ^{238}U	2	5.86E-01	1.02E-02 mg/L
MS ^{238}U		1.97 E-01	3.43 E-03 pCi/mL
MS ^{237}Np	2	<2.00E-02	mg/L
MS ^{237}Np		<1.41 E+01	pCi/mL
MS ^{230}Th	2	<9.99E-03	mg/L
MS ^{230}Th		<2.11 E+02	pCi/mL
MS ^{232}Th	2	<6.99E-02	mg/L
MS ^{232}Th		< 7.67 E-03	pCi/mL
MS ^{239}Pu	2	<2.50E-02	mg/L
MS ^{239}Pu		<1.55 E-03	pCi/mL
MS ^{240}Pu	2	<9.99E-03	mg/L
MS ^{240}Pu		<2.28 E+03	pCi/mL
MS ^{241}Pu, ^{241}Am	2	<9.99E-03	mg/L
MS ^{242}Pu	2	<1.50E-02	mg/L
MS ^{242}Pu		<5.73 E+01	pCi/mL
MS ^{243}Am, ^{243}Cm	2	<9.99E-03	mg/L
MS ^{244}Pu, ^{244}Cm	2	<1.50E-02	mg/L

Table 41. CSS MS Raw Data (all entries mg/L)

Method	First Result	Second Result	Third Result	Fourth Result	Fifth Result
MS, (stable) ^{59}Co	1.14 E-01	1.12 E-01	9.50E-02	1.01 E-01	9.87 E-02
MS, Total Rb	9.93 E-01	9.21 E-01	8.86 E-01	7.78 E-01	7.82 E-01
MS, Mass 99 (^{99}Tc)	4.47 E+00	4.45 E+00	4.41 E+00	4.17 E+00	4.44 E+00
MS, Mass 133 (^{133}Cs)	2.05 E+00	2.04 E+00	2.05 E+00	1.92 E+00	2.01 E+00
MS, Mass 135	3.06 E-01	2.97 E-01	3.01 E-01	2.72 E-01	3.09 E-01
MS, Mass 137	7.93 E-01	8.32 E-01	7.85 E-01	7.58 E-01	7.95 E-01
MS total lead	1.60 E-01	1.77 E-01	1.71 E-01	1.46 E-01	1.40 E-01
MS ^{233}U	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03
MS ^{234}U	1.50 E-02	1.50 E-02	1.75 E-02	1.25 E-02	1.62 E-02
MS ^{235}U	4.25 E-02	4.00 E-02	3.37 E-02	3.50 E-02	3.75 E-02
MS ^{236}U	<2.50 E-02	<2.50 E-02	<2.50 E-02	<2.50 E-02	<2.50 E-02
MS ^{238}U	5.71 E-01	5.90 E-01	5.99 E-01	5.89 E-01	5.82 E-01
MS ^{237}Np	<2.00 E-02	<2.00 E-02	<2.00 E-02	<2.00 E-02	<2.00 E-02
MS ^{230}Th	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03
MS ^{232}Th	<6.99 E-02	<6.99 E-02	<6.99 E-02	<6.99 E-02	<6.99 E-02
MS ^{239}Pu	<2.50 E-02	<2.50 E-02	<2.50 E-02	<2.50 E-02	<2.50 E-02
MS ^{240}Pu	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03
MS ^{241}Pu, ^{241}Am	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03
MS ^{242}Pu-242	<1.50 E-02	<1.50 E-02	<1.50 E-02	<1.50 E-02	<1.50 E-02
MS ^{243}Am, ^{243}Cm	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03	<9.99 E-03
MS ^{244}Pu, ^{244}Cm	<1.50 E-02	<1.50 E-02	<1.50 E-02	<1.50 E-02	<1.50 E-02

The turbidity of the CSS solution was observed for 72 days (Figure 15). The measurements showed that while the turbidity slowly increased over the time period (~3 months), it did not exceed 8.6 NTU, which indicates only gradual solids formation (using a previous formalism, [30] roughly estimated to be ~3.5 mg/L). Solids formation continues for ~5 weeks.

Figure 15. CSS Turbidity Measurements



3.4 ESS (Extraction, Scrub, Strip) Solvent Extraction Tests

Material from the ARP (MST strike) test was used in an ESS test. For this test, we used a starting volume of 90 mL of aqueous feed, and 30 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI). Table 42 shows the strip effluent pH and gamma data, corrected for any dilutions.

Table 42. Strip Effluent pH and Gamma Data from the ESS Test

Analyte	Strip#1 (pCi/mL)	Strip#2 (pCi/mL)	Strip#3 (pCi/mL)
Co-60	<6.87E+03	<9.01E+03	<1.03E+04
Ru-106	<6.10E+05	<1.13E+06	<5.32E+05
Sb-125	<4.96E+05	<9.24E+05	<4.46E+05
Cs-134	<3.23E+04	<5.87E+04	<1.23E+04
Cs-137	2.46E+08 (±2.63E+06)	2.35E+08 (±2.56E+06)	1.25E+08 (±1.35E+06)
Ce-144	<5.55E+05	<1.03E+06	<4.73E+05
Eu-154	<4.96E+04	<9.05E+04	<3.79E+04
Eu-155	<2.22E+05	<4.13E+05	<1.76E+05
Am-241	<5.41E+05	<1.00E+06	<3.58E+05
pH	3 (pH units)	3 (pH units)	3 (pH units)

Table 43 shows the test results from the ESS test, corrected to the normal process operating temperatures (23 ºC for extraction, and 33 ºC for scrubbing and stripping). As a comparison, the results from an ESS test (using the same solvent) in August, 2007 are displayed.[34]

Table 43. Cesium Distribution Values for the Solvents

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
Acceptable Range	>8	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
S2-D1-YES BOB-T-WI, August Test[34]	9.53	2.54	1.72	0.147	0.0607	0.0615
S2-D1-YESBOB-T-WI, Current Cells Test	9.07	1.60	1.29	0.070	0.046	0.042

The tests show acceptable cesium distribution values for each step of the process for the solvents, except for one of the scrub results in the August test.[33] The out of range scrub result was not declared an issue at the time. Prior to the test, personnel used the projected composition to estimate the extraction cesium distribution value for this waste.[35] (Reference 35 contains data at higher Na concentrations. For lower concentrations, see Appendix C.) For 4.5 M Na waste – roughly equivalent to the 4.67 M Na measured concentration – the models predicted an extraction D_{Cs} value of 9.5 in reasonable agreement with the measured value of 9.07.

Finally, the researchers performed an extraction test on the Tank 49H salt solution to concentrate any organic impurities. Taking 53.7 mL of the same Tank 49H material used in the ESS test, the researchers contacted this with 5.34 mL of CSSX solvent (10.1:1 volume ratio) that contained no BOBCalixC6 extractant (Calix[4]arene-bis(t-octylbenzo-crown-6), from batch S2-NOBOB 9/05). After 24 hours of contact, the organic phase was removed and analyzed by GC-MS for any impurities (presumably introduced from the Tank 49H material). The GC-MS analyses found the correct ratio of Isopar® L to Modifier ((2,2,3,3-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol)), and found no detectable levels of impurities.

3.5 ANALYSES OF DSS

The product of the first ESS test provided DSS having the analyses given in Table 44 and Table 45 below. Anions in Table 44 were provided by IC-anions. Mercury was measured by CV-Hg. IC-cations provided ammonium ion.

Table 44. DSS Chemical Data

Component	First Analysis	Second Analysis	Third Analysis	Average mg/L	1 sigma, mg/L
Fluoride	<500	<500	<500	<500	
Formate	<500	<500	<500	<500	
Chloride	<500	<500	<500	<500	
Nitrite	11800	11400	11500	11600	208
Bromide	<500	<500	<500	<500	
Nitrate	162000	161000	166000	163000	2646
Phosphate	<1250	<1250	<1250	<1250	
Sulfate	11100	10500	10500	10700	346
Oxalate	<500	<500	<500	<500	
Mercury	1.94	1.87	2.00	1.94	0.06
TPB (HPLC)	<6	<6	<6	<6	
Ammonium ion**	<500	<500	<500	<500	
pH by paper	14*	14*	14*	14	

*pH paper with a range of 12.5 to 14 provided these values.

**Refined work reduced the ammonium detection limit over that of past reporting.

Table 45. DSS Radiochemical Data (pCi/mL)

Isotope	First Result	Second Result	Third Result	Average, pCi/mL	1-sigma, pCi/mL
Gamma, ^{137}Cs	2.92E+07	2.91E+07	2.97E+07	2.93E+07	3.3E+05
^{137m}Ba	Calculated from ^{137}Cs			2.77E+07	3.1E+05
Gamma, ^{134}Cs	<5.13E+04	<4.32E+04	<5.74E+04	<5.06E+04	
Gamma, ^{94}Nb	<8.61E+01	<8.27E+01	<8.72E+01	<8.54E+01	
Gamma, ^{106}Ru	<8.72E+02	<8.44E+02	<7.54E+02	<8.24E+02	
Gamma, ^{125}Sb	<2.59E+02	<2.49E+02	<2.50E+02	<2.53E+02	
Gamma, ^{126}Sb	2.10E+02	1.99E+02	2.00E+02	2.03E+02	6.1E+00
Gamma, ^{126}Sn	<3.20E+02	<3.43E+02	<4.20E+02	<3.61E+02	
Gamma, ^{144}Ce	<4.35E+02	<4.43E+02	<4.12E+02	<4.30E+02	
Gamma, ^{154}Eu	<1.20E+02	<1.18E+02	<1.10E+02	<1.16E+02	
Gamma, ^{155}Eu	<3.61E+02	<3.87E+02	<4.74E+02	<4.07E+02	
Gamma, ^{226}Ra	<2.04E+03	<1.60E+03	<1.86E+03	<1.83E+03	
Gamma, ^{241}Am	<4.24E+02	<4.28E+02	<4.20E+02	<4.24E+02	
Gamma, ^{60}Co	<1.04E+02	<1.05E+02	<1.01E+02	<1.03E+02	
Total Alpha	<9.06E+04	<8.39E+04	<1.27E+05	<1.01E+05	
Total Beta	3.83E+07	3.77E+07	3.54E+07	3.71E+07	1.5E+06

All samples here were acid preparations (Prep 2).

4.0 CONCLUSIONS

- Density measurements of samples from three elevations in Tank 49H showed that the solution was well mixed for both the Sample A (December 7, 2007), and Sample C (January 18, 2008) events. A transfer of over 400,000 gallons of liquid out of Tank 49H before the second sampling did not appreciably disturb the settled solids.
- Sample A solids demonstrated significant removal through settling overnight and decantation the next day.
- Tests using MST with the Tank 49H material gave acceptable decontamination factors for plutonium (14 and 21.1) and strontium (59.4 and >54.4).
- A standardized demonstration of cesium extraction, scrubbing and stripping yielded expected and acceptable cesium distribution values of: 9.07, 1.60, 1.29, 0.07, 0.046 and 0.042 for extraction, scrub #1, scrub #2, strip #1, strip #2, and strip #3, respectively. Furthermore, a test to look for solvent impurities found no traces of any contaminants.

It is recommended that density and in-cell solids settling continue to be used to evaluate samples in the future. These methods are simple and were found effective in the qualification of Tank 49H feed.

It is recommended that the number of analyses for future feed qualification efforts be reduced by using the bulk chemical analyses from feed for the follow-on salt solution streams (i.e., CSS, DSS) and omitting analyses of those later streams. Bulk chemical analyses include IC anions, and the ICP-ES suite. Some radiochemical analytes like tritium, ^{14}C, ^{147}Pm, ^{151}Sm, and ^{129}I may also best be handed the same way.

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APPENDIX A. COMMUNICATION OF SUITES OF ANALYSES

SALT BATCH QUALIFICATION MEETING – 7/25/07 0930

- **Radionuclide Reporting**

EM-WAPS 1.2 (Radionuclide Inventory Spec) and 1.6 (IAEA Safeguards) requires reporting of radionuclides in Production Records

Strategy for radionuclides determined indirectly from Tank Farm samples (WSRC-IM-91-116-4 and WSRC-TR-94-0505):

$WDF_{salt} = [Ti]_{Concentrated\ MST/Sludge\ Solids} / [Ti]_{SME\ Product}$

$WDF_{sludge} = [C]_{Tank\ 40} / [C]_{SME\ Product}$ where C is Fe, Al, Mn and Ca

An SDF is calculated and is the average of the WDF_{sludge} for the four elements

^{238}Pu "salt" contribution to the final DWPF glass product:
$[^{238}Pu]_{glass(salt)} = [^{238}Pu]_{Concentrated\ MST/Sludge\ Solids} / WDF_{salt}$

^{238}Pu "sludge" contribution to the final DWPF glass product:
$[^{238}Pu]_{glass(sludge)} = [^{238}Pu]_{Tank\ 40} / SDF$

$^{238}Pu_{glass(total)} = [^{238}Pu]_{glass(salt)} + [^{238}Pu]_{glass(sludge)}$

- **RW-0333P Applicability in SRNL Activities**

RW-0333P will need to apply to various tasks in TTR (SRNL tasks based on Tank 25 TTQAP – WSRC-RP-2007-00402).

Sampling of Tank 49 (Tank Farm activity) – NOT WFA

Task 1 - Compositing the samples and adjusting the sodium molarity – WFA

Task 2 - Performing MST Strike (24 hrs) and filtering - WFA

Task 3 - Performing Extraction, Scrub and Strip (ESS) – NOT WFA

Task 4 – Data analysis from Task 1 (WFA), Task 2 (WFA) and Task 3 (not WFA) MST/Sludge solids concentration will be calculated based on the analysis of the incoming feed and the filtrate

- **Required Analytes from the SRNL Characterization**

512-S Requirements (Adjusted Feed to 512-S, MST/sludge solids and CSS)

(I) DWPF Rad Program Evaluation **[Priority A – NOT WFA] - feed**
- Radionuclides (^{3}H, ^{90}Sr, ^{106}Ru, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{147}Pm, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{241m}Am ^{244}Cm, ^{125}Sb, ^{125m}Te, total alpha and total beta-gamma)

(II) TSR/WAC Requirements **[Priority A – NOT WFA] - feed**
- Radionuclides (total alpha, total beta, total gamma, ^{3}H, ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{125m}Te, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu, ^{155}Eu, ^{233}U, ^{235}U, ^{238}U, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{241}Am and ^{244}Cm)
- Chemicals ([OH^-], [CO_3^{2-}], [NO_2^-], [NO_3^-], [SO_4^{2-}], [Mn], [Hg], [Fe], [Ti], [Cr], [P], [F], [Cl], [Cu] and wt% solids)
- Organic analysis (VOA and SVOA)

(III) WAPS Reporting **[Priority C - WFA] – MST/sludge solids**
- Radionuclides in MST/Sludge Solids with half-lives greater than 10 years and will contribute greater than 0.01% of the total activity up to 1100 years after production
- IAEA radionuclides in MST/sludge solids (^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu and ^{242}Pu
- Elementals present in MST/sludge solids with quantities greater than __ wt% (Ti, etc.)

(IV) Solid Waste Characterization **[Priority B – NOT WFA] – feed, MST/sludge solids and CSS**
- Radionuclides (^{3}H, ^{14}C, ^{90}Sr, ^{90}Y, ^{99}Tc, ^{129}I, ^{137}Cs, ^{137m}Ba, ^{147m}Pm, ^{233}U, ^{234}U, ^{235}U, ^{238}U, ^{237}Np, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu, ^{241}Am, ^{244}Cm, gross alpha and gross beta/gamma)
- Chemicals (Sb, As, Ba, Be, Cd, Cr, Co, Pb, Mn, Hg, Ni, Se and Ag)

Tank Farm/MCU-Related (CSS / Wash Water Stream)

(V) TSR/WAC Requirements for Clarified Salt Solution/Wash Water **[Priority A]**
pH, specific gravity, [OH^-], [NO_2^-], [NO_3^-], [Cl^-], [F^-], [SO_4^{2-}], ^{233}U, ^{235}U, ^{239}Pu, ^{241}Pu, [K], TBP, DBP, TMA, formate, 1-butanol
- Radionuclides required to calculate IDP (these are not defined in the WAC – total alpha and Sr-90?)
- Radionuclides required for Hazard Cat. calc (bolded isotopes contribute ≥0.01% sum of ratios)
($^{\mathbf{3}}$**H**, ^{14}C, ^{60}Co, ^{59}Ni, ^{63}Ni, ^{79}Se, $^{\mathbf{90}}$**Sr**, ^{90}Y, ^{94}Nb, ^{99}Tc, ^{125}Sb, ^{126}Sn, ^{129}I, $^{\mathbf{134}}$**Cs**, ^{135}Cs, $^{\mathbf{137}}$**Cs**, $^{\mathbf{137m}}$**Ba**, ^{147}Pm, ^{154}Eu, ^{232}Th, ^{232}U, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{237}Np, $^{\mathbf{238}}$**Pu**, $^{\mathbf{239}}$**Pu**, $^{\mathbf{240}}$**Pu**, $^{\mathbf{241}}$**Pu**, ^{242}Pu, $^{\mathbf{241}}$**Am**, ^{242}mAm, $^{\mathbf{244}}$**Cm**, ^{245}Cm, $^{\mathbf{22}}$**Na**, ^{26}Al, ^{126}Sb, ^{126m}Sb, ^{151}Sm, ^{152}Eu, ^{155}Eu, ^{226}Ra, ^{228}Ra, ^{227}Ac, ^{229}Th, ^{230}Th, ^{231}Pa, ^{244}Pu, ^{243}Am, ^{242}Cm, ^{243}Cm, ^{247}Cm, ^{248}Cm, ^{249}Bk, ^{249}Cf, ^{251}Cf and ^{242}Cf)
- Downstream facility acceptance criteria (see Attach. 13.1 of X-SD-G-00001)

Saltstone-Related (DSS Stream)

(VI) WAC Requirements (LIMITS) **[Priority A]**
- Radionuclides (^{3}H, ^{14}C, 63Ni, ^{90}Sr, ^{99}Tc, ^{129}I, ^{137}Cs, ^{233}U, ^{235}U, ^{241}Pu and Total Alpha)
- Chemicals ($[NH_4^+]$, $[CO_3^{2-}]$, $[Cl^-]$, $[F^-]$, $[OH^-]$, $[NO_3^-]$, $[NO_2^-]$, $[C_2O_4^{2-}]$, $[PO_4^{3-}]$, $[SO_4^{2-}]$, As, Ba, Cd, Cr, Pb, Hg, Se, Ag, Al, NaC_4H_9OH, C_3H_7OH, C_6H_5OH, TOC and TPB)
- Other (pH and total insoluble solids)

APPENDIX B. COMMUNICATION OF TANK 49H MIXING HISTORY

Caroline Atseff/WSRC/Srs

12/06/2007 12:27 PM

To: Samuel Fink/SRNL/Srs@Srs
CC: Charles Nash/SRNL/Srs@Srs, Byron Neely/WSRC/Srs@Srs, Eric Freed/WSRC/Srs@Srs, David02 Martin/WSRC/Srs@Srs, Jeff Ledbetter/WSRC/Srs@Srs, Adam Orris/WSRC/Srs@Srs, Michael Poirier/SRNL/Srs@Srs

Subject: Tank 49 Slurry Pump Operation to Support Feed Qual Sample

Sam,

Here is the information for the 23-49 transfer that we discussed:

- Start time of T.49 slurry pump: 12/5/07, Wed, 1920 (pump started, some mixing occurring)

-Turntable for slurry pump rotating: 12/5/07, Wed., 2230 (full mixing occurring)

- Time that 23-49 transfer was completed: 12/6/07, Thursday, 0330 (this is the time that Tank 23 material was no longer added; jet no longer operating; this does not include the time spent for vent and drain).

- Slurry Pump shut off: 12/6/07, Thursday, 1047.

- Pump was running continuously since it started.

- 9X volume flush has not yet started as of 1145 12/6/07. Pump was successfully restarted, plan to continue to run the pump throughout the flush. However, per previous discussion, the running of the pump during the flush, while recommended, is not required. If the pump stops again, we may not restart it.

From this information, there was some mixing during the transfer, and full mixing was occurring post transfer from 12/6/07, Thursday, 0330 until 12/6/07, 1047. This is 7 hours and 17 minutes of mixing post transfer.

Per SRNL-CST-2007-00115, Rev. 0, Slurry Pump Mixing Time in SRS Tank 49H, it would appear that 4-7 hours of slurry pump run time after the completion of the transfer would constitute sufficient mixing. The actual time was a little over 7 hours. Per our verbal discussion, SRNL agreed that the 7 hours was sufficient. Please reply to this message to confirm.

Thanks,
Caroline

APPENDIX C. PRETEST PREDICTIONS FOR MST and CSSX PERFORMANCE

Predictive models estimated the effect of the MST addition on the blended Tanks 23F and 49H waste. The models had previously been benchmarked with a wide variety of data.[32] Initial concentrations for models were taken from a Tank Farm material balance where a 440,000 gallon transfer into Tank 49H was considered.[36]

All concentrations (except sodium) is given in micromolar.

ELEMENT	Initial Concentration	Final Concentration	DF	Sodium Concentration
Strontium	30.9	0.531	58.2	5.1 M
Uranium	8.31	4.71	1.76	5.1 M
Plutonium	0.0879	0.00797	11.0	5.1 M
Neptunium	0.154	0.0351	4.39	5.1 M
Strontium	27.3	0.424	64.4	4.5 M
Uranium	7.32	3.53	2.07	4.5 M
Plutonium	0.0775	0.00702	11.0	4.5 M
Neptunium	0.136	0.0307	4.43	4.5 M

The original[35] pretest predictions for solvent performance with this waste assumed dilution to between 5 and 6 M Na. Once project personnel realized the dilution would produce a less concentrated waste, that author extended his calculations as shown in the following tables.

Waste Compositions of Tanks 23 and 49 Blends Along with Predicted D-values Using SXFIT and Neural Net Models.

Blend ID	Na^+, M	K^+, M	Cs^+, M	NO_3^-, M	Cl^-, M	NO_2^-, M	OH^-, M	F^-, M	PO_4^{3-}, M	SO_4^{2-}, M	CO_3^{2-}, M	AlO_2^-, M	Extraction D-values at 23 °C: SXFIT	Extraction D-values at 23 °C: Neural Net
Transfer of 440,000 Gallons of Supernate from Tank 23 into Tank 49 Followed by Dilution to 5.6 M Sodium (initial cesium-137 concentration = 0.26 Ci/gallon)														
440-5.6A	5.6000000	0.0081002	0.0000077	2.8307909	0.0021246	0.2730290	0.7100594	0.0043049	0.0115924	0.1259585	0.2114839	0.7453323	n/a	9.10
440-5.6B	**5.2671952**	0.0081002	0.0000077	2.8307909	0.0021246	0.2730290	0.7100594	0.0043049	0.0115924	0.1259585	0.2114839	0.7453323	9.53	n/a
440-5.6C	**5.4335976**	0.0081002	0.0000077	**2.9971933**	0.0021246	0.2730290	0.7100594	0.0043049	0.0115924	0.1259585	0.2114839	0.7453323	9.31	n/a
440-5.6D	**5.4890651**	0.0081002	0.0000077	**2.9417258**	0.0021246	0.2730290	0.7100594	0.0043049	0.0115924	0.1259585	0.2114839	**0.8562672**	9.39	n/a
												Average	9.41	9.10
												% Relative Standard Deviation	1.20	n/a
Maximum feed or initial cesium-137 concentration that can be processed in the MCU for final DSS cesium-137 concentration to be ≤ 0.1 Ci/gallon (MCU target)													2.35 Ci/gallon	
Transfer of 440,000 Gallons of Supernate from Tank 23 into Tank 49 Followed by Dilution to 5.0 M Sodium (initial cesium-137 concentration = 0.23 Ci/gallon)														
440-5.0A	5.0000000	0.0072323	0.0000069	2.5274919	0.0018970	0.2437759	0.6339816	0.0038437	0.0103503	0.1124629	0.1888249	0.6654753	n/a	9.57
440-5.0B	**4.7028529**	0.0072323	0.0000069	2.5274919	0.0018970	0.2437759	0.6339816	0.0038437	0.0103503	0.1124629	0.1888249	0.6654753	9.63	n/a
440-5.0C	**4.8514264**	0.0072323	0.0000069	**2.6760654**	0.0018970	0.2437759	0.6339816	0.0038437	0.0103503	0.1124629	0.1888249	0.6654753	9.48	n/a
440-5.0D	**4.9009510**	0.0072323	0.0000069	**2.6265409**	0.0018970	0.2437759	0.6339816	0.0038437	0.0103503	0.1124629	0.1888249	**0.7645243**	9.54	n/a
												Average	9.55	9.57
												% Relative Standard Deviation	0.81	n/a
Maximum feed or initial cesium-137 concentration that can be processed in the MCU for final DSS cesium-137 concentration to be ≤ 0.1 Ci/gallon (MCU target)													2.52 Ci/gallon	
Transfer of 440,000 Gallons of Supernate from Tank 23 into Tank 49 Followed by Dilution to 4.5 M Sodium (initial cesium-137 concentration = 0.21 Ci/gallon)														
440-4.5A	4.5000000	0.0065091	0.0000062	2.2747427	0.0017073	0.2193983	0.5705835	0.0034593	0.0093153	0.1012166	0.1699424	0.5989278	n/a	9.94
440-4.5B	**4.2325676**	0.0065091	0.0000062	2.2747427	0.0017073	0.2193983	0.5705835	0.0034593	0.0093153	0.1012166	0.1699424	0.5989278	9.61	n/a
440-4.5C	**4.3662838**	0.0065091	0.0000062	**2.4084589**	0.0017073	0.2193983	0.5705835	0.0034593	0.0093153	0.1012166	0.1699424	0.5989278	9.51	n/a
440-4.5D	**4.4108559**	0.0065091	0.0000062	**2.3638868**	0.0017073	0.2193983	0.5705835	0.0034593	0.0093153	0.1012166	0.1699424	**0.6880719**	9.56	n/a
												Average	9.56	9.94
												% Relative Standard Deviation	0.51	n/a
Maximum feed or initial cesium-137 concentration that can be processed in the MCU for final DSS cesium-137 concentration to be ≤ 0.1 Ci/gallon (MCU target)													2.66 Ci/gallon	

n/a – not applicable

The numbers in bold are the adjusted values.