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Disclaimer:

Key Words:
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ISDP

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Permanent

ISDP SALT BATCH #2 SUPERNATE QUALIFICATION

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JANUARY 5, 2009

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REVIEWS AND APPROVALS

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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

1.0 EXECUTIVE SUMMARY

This report covers the methods and data for Integrated Salt Disposition Project Salt Batch 2 feed sample qualification. This revision 1 contains additional sample results not available at the time of the revision 0 report. A second revision is planned to include future sample results. The following observations are made from the work.

- The composite solution mimicking the planned composite in Tank 49H shows trace precipitation of solids (i.e., < 5 ppm) within the first 23 days after preparation.

- A demonstration of the monosodium titanate removal of strontium and actinides provided acceptable 24 hour decontamination values for Pu and Sr of 5.64 and 70.9, respectively.

- A demonstration of cesium extraction, scrubbing and stripping – prototypical of the Modular Caustic-Side Solvent Extraction Unit – yielded cesium mass transfer behavior within acceptable norms. The measured distribution values are: 14.64, 1.51, 2.13, 0.74, 0.09 and 0.031 for Extraction, Scrub #1, Scrub #2, Strip #1, Strip #2, and Strip #3, respectively. Adjusting the experiment organic-aqueous values to match the planned operational values yields distribution values of 12.50, 1.51, 1.60, 0.38, 0.09 and 0.031 for Extraction, Scrub #1, Scrub #2, Strip #1, Strip #2, and Strip #3, respectively.

- Requested chemical and radionuclide compositions are reported within for the samples from Tank 22H and Tank 41H.

Analyses of a sample from Tank 49H collected after all additions provide the following conclusions.

- The physical measurements of the Tank 49H confirmatory sample (density and turbidity) are within the expected range of results.

- The total plutonium content was 3.35E+05 pCi/mL for ^{238}Pu, 5.63E+03 pCi/mL for $^{239/40}$Pu, and 7.73E+04 pCi/mL for ^{241}Pu.

- The ^{235}U content was measured to be 0.250 pCi/mL.

- There were less than detectable amounts of organic analytes (i.e., butanol, isopropanol, tetraphenylborate, tributyl phosphate, ethylenediaminetetraacetate), except for formate, which was present at a 326 mg/L concentration.

- The measured insoluble solids content was 0.753 wt %.

2.0 INTRODUCTION

This report covers the laboratory testing and analyses of the second Integrated Salt Disposition Project (ISDP) salt supernate samples, 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) characterizing Tank 22H supernate, (2) characterizing Tank 41H supernate, (3) verifying actinide and strontium adsorption with a standard laboratory-scale test using monosodium titanate (MST) and filtration, and (4) checking cesium mass transfer behavior for the MCU solvent performance when contacted with the liquid produced from MST contact.

This study also includes characterization of a post-blend Tank 49H sample as part of the Nuclear Criticality Safety Evaluation (NCSE).[1]

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

Details for the work are contained in controlled laboratory notebooks.[5]

3.0 EXPERIMENTAL

3.1 RECEIPT AND EXAMINATION OF SAMPLES

The Tank Farm pulled samples from both Tank 22H and 41H on 7/22/2008 and 8/28/08, respectively. The Tank 22H material arrived at SRNL in two dip bottles (HTF-22-08-108 and -110) and one 4L carboy (HTF-22-08-107). The Tank 41H material arrived at SRNL in two dip bottles (HTF-41-08-129 and -130) and one 4L carboy (HTF-41-08-128).

Each of the samples was optically clear, with no or virtually no visible solids present.

The researchers measured the density of the solution in each bottle to make sure that the samples pulled from each tank were relatively homogenous. The results are listed in Table 1.

Table 1. Density of Each Tank Sample

Sample ID	Sample Depth (“)	Density (g/mL)
HTF-22-08-107	100	1.019
HTF-22-08-108	135	1.021
HTF-22-08-110	62	1.033
Tank 22H average		1.024
HTF-41-08-128	210	1.398
HTF-41-08-129	250	1.411
HTF-41-08-130	190	1.428
Tank 41H average		1.412

Given the low variance in samples, SRNL combined the samples for each tank into their respective 4L carboys.

After transfer of the waste from Tanks 22H and 41H into Tank 49H, Operations added the required volume of sodium hydroxide (50 wt %) to meet the desired final hydroxide concentrations based on the analyses of the individual tanks.[6] They collected samples from the tank for analyses by SRNL. Three doorstops from Tank 49H were delivered to SRNL on December 2, 2008, containing Sample HTF-49-08-166, -167, and -168. The technicians measured the density on each of the three samples as shown in Table 2.

Table 2. Tank 49H Confirmatory Sample Densities

Sample	Sample Depth (“)	Density
HTF-49-08-166	192	1.280
HTF-49-08-167	115	1.275
HTF-49-08-168	38	1.265
Average		1.273

Sample HTF-49-08-168 was slightly colored. During sample preparation, we found that a 0.45 mm syringe filter was sufficient to remove the coloration (entrained particles).

As the densities were sufficiently close, we combined the three samples into a single bottle. From this bottle samples were removed and sent to Analytical Development (AD) for analyses.

The testing requirements for the Tank 49H sample derive from a customer document,[29] as well as the following additional requests from subsequent verbal communications: tributyl phosphate, tritium, mercury, IC-Anions, ammonium, butanol and isopropanol.

3.2 SAMPLE PREPARATION AND ANALYSES

Once combined, each composite tank sample was filtered through a 0.45 μm nylon membrane filter cup to remove any solids. The solids were retained for analysis.

Tank Farm personnel derived the proper mixing ratios between the Tank 22H, 41H and 49H (from a previous study) samples.[6] Furthermore, the Tank Farm decided to add 50 wt % caustic to the composite to reach a free hydroxide to 2.0 M (to inhibit aluminum compound precipitation). Table 3 lists the constituents and volumes of the materials to comprise the ISDP Composite.

Table 3. Constituents of the ISDP Composite

Component	Volume Ratio
Tank 22H	2.47
Tank 41H	1.74
Tank 49H	7.77
50 wt % NaOH	1

3.2.1 Methods of Sample Preparation

Tank 22H samples were sent to AD without preparation or dilution due to the low radioactivity. Tank 41H samples possess a moderate ^{137}Cs activity and were sent undiluted in small quantities where possible, or diluted ~5:1 otherwise. Dilution volumes used 5 M nitric acid in general, and 0.5 M NaOH for acid sensitive samples, such as TIC/TOC. Sludge/slurry samples were dissolved using a preparation consisting of nitric acid and hydrogen peroxide, allowing titanate dissolution without the addition of any metallic elements. Samples were provided to AD in triplicate.

Table 4 below provides the names and procedure citations for the methods used in this work. A detailed description of the methods is provided by Bannochie and Bibler.[7]

3.3 ACTINIDE REMOVAL PROCESS DEMONSTRATION

For the ARP Demonstration Tests, technicians generated 200 mL of the ISDP Composite (without filtration), placing half into each of two 250-mL polyethylene bottles. We did not observe gross formation of solids and did not measure the turbidity. We used one of the bottles to conduct the MST addition 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 two bottles at 0, 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 initially provided by an actively controlled water bath. Unfortunately, the water bath controller failed (i.e., electronic components have a limited, unpredictable lifespan in the Shielded Cells). However, records of the measured temperature over the duration of the experiment showed that the temperature stayed at 22-23 ºC, which caused no disruption to the experiment.

Technicians filtered the samples using 0.45 µm Versapor ™ syringe filters, diluted them with acid, removed them from the cells for analysis, and analyzed for plutonium (PuTTA), ^{90}Sr (beta scintillation), ^{137}Cs (gamma scan), and ^{237}Np (ICPMS).

3.4 EXTRACTION, SCRUB, STRIP (ESS) EXPERIMENTAL DEMONSTRATION

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 ISDP Composite 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 4.4.3) of the batch of solvent that most closely matched what is in the MCU facility ("S2-D1-YESBOB-T-WI"). In previous documents,[8,9] we measured the D values from this same batch of solvent. Those results are presented here for comparison (section 4.5, Table 27).

Table 4. 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

4.0 RESULTS AND DISCUSSION

This report provides results in sections corresponding to required analyses described in the TTQAP. Data are generally grouped into radiochemical (pCi/mL) and chemical (mg/L) results.

AD reported some radiochemical results with "upper limit" values rather than "below detection" results with detection limits. Results qualified as "upper limits" are results 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.

Some analytes can be determined by multiple methods. However, due to the customer request, SRNL is providing only a single result – the most appropriate and accurate result as determined by SRNL. For example, 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 (PuTTA plus ^{241}Pu liquid scintillation providing ^{238}Pu, $^{239 \text{ plus } 240}$Pu, and ^{241}Pu) and ^{99}Tc. ^{99}Tc by counting is preferred over ^{99}Tc by MS.

4.1 TANK 22H RESULTS

The testing requirements for the Tank 22H sample are derived from a customer document.[29] Tank 22H has more requirements than Tank 41H. As Tank 22H contains DWPF Recycle, it is expected that the tank is relatively dilute in most analytes.

4.1.1 Tank 22H Radiochemical Results

Tables 5 and 6 list the results of the radiochemical analyses for the Tank 22H samples. The bulk of the results are "less-than" values caused by the low analyte concentrations in the samples. The most notable results are the ^{137}Cs activity (2.31E+05 pCi/mL) and the total ^{238}Pu activity (1.53E+01 pCi/mL).

4.1.2 Tank 22H Chemical Results

Tables 7 and 8 list the results of the cold chemical analyses for the Tank 22H samples. Many of the results are "less-than" values caused by the low analyte concentrations in the samples. The most notable results are the sodium concentration (9400 mg/L or 0.41 M) and the free hydroxide value of 0.141 M.

Table 5. Tank 22H Radiochemical Results (pCi/mL)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Total Alpha	radiocounting	<9.09E+03	<9.23E+03	<8.87E+03	<8.87E+03	NA
Total Alpha, Cs-removed	radiocounting	<1.04E+04	<1.13E+03	<9.81E+02	<9.81E+02	NA
Total Beta	radiocounting	6.75E+05	6.84E+05	6.66E+05	6.75E+05	1.33
Total Beta, Cs-removed	radiocounting	4.17E+05	4.38E+05	4.33E+05	4.29E+05	2.54
^{14}C	radiocounting	<1.17E+01	<1.17E+01	<1.17E+01	<1.17E+01	NA
^{59}Ni	radiocounting	<2.26E+01	<2.53E+01	<2.50E+01	<2.26E+01	NA
^{60}Co	radiocounting	<2.75E+00	<2.61E+00	<4.36E+00	<2.61E+00	NA
^{63}Ni	radiocounting	<4.43E+00	<5.09E+00	<5.00E+00	<4.43E+00	NA
^{79}Se	radiocounting	<4.32E+00	<4.03E+00	<3.56E+00	<3.56E+00	NA
^{90}Sr	radiocounting	2.34E+05	2.02E+05	2.19E+05	2.18E+05	7.32
^{90}Y	calculated	2.34E+05	2.02E+05	2.19E+05	2.18E+05	7.32
^{93}Mo	calculated	<1.24E+04	<1.24E+04	<1.24E+04	<1.24E+04	NA
^{93m}Nb	calculated	<2.68E+06	<2.68E+06	<2.68E+06	<2.68E+06	NA
^{94}Nb	radiocounting	<3.31E+00	<3.44E+00	<5.40E+00	<3.31E+00	NA
^{99}Tc	radiocounting	7.11E+02	7.02E+02	6.66E+02	6.93E+02	3.44
^{106}Ru	radiocounting	<2.41E+01	<2.39E+01	<3.48E+01	<2.39E+01	NA
^{106}Rh	calculated	<2.41E+01	<2.39E+01	<3.48E+01	<2.39E+01	NA
^{125}Sb	radiocounting	<1.41E+01	<1.43E+01	<2.16E+01	<1.41E+01	NA
^{125m}Te	calculated	<1.41E+01	<1.43E+01	<2.16E+01	<1.41E+01	NA
^{126}Sn	radiocounting	<2.48E+01	<2.50E+01	<3.75E+01	<2.48E+01	NA
^{129}I	radiocounting	2.64E+00	2.45E+00	1.99E+00	2.36E+00	14.11
^{134}Cs	radiocounting	<7.07E+03	<8.55E+03	<7.34E+03	<7.07E+03	NA
^{135}Cs	ICPMS	3.66E+00	<2.88E+00	3.87E+00	3.76E+00	3.91
^{137}Cs	radiocounting	2.45E+05	2.11E+05	2.37E+05	2.31E+05	7.70
^{137m}Ba	calculated	2.32E+05	2.00E+05	2.24E+05	2.19E+05	7.70
^{144}Ce	radiocounting	<5.45E+01	<5.49E+01	<8.06E+01	<5.45E+01	NA
^{144}Pr	calculated	<5.45E+01	<5.49E+01	<8.06E+01	<5.45E+01	NA
^{147}Pm	radiocounting	<4.45E+01	<3.87E+01	<1.57E+02	<3.87E+01	NA
^{151}Sm	radiocounting	<1.30E+01	<1.13E+01	<7.70E+01	<1.13E+01	NA
^{154}Eu	radiocounting	<8.78E+00	<8.73E+00	<1.37E+01	<8.73E+00	NA
^{155}Eu	radiocounting	<2.85E+01	<2.88E+01	<4.32E+01	<2.85E+01	NA
^{226}Ra	radiocounting	<1.29E+02	<1.27E+02	<2.20E+02	<1.27E+02	NA

Table 6. Tank 22H Actinide Region Radioanalytical Results (pCi/mL)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
^{229}Th	radiocounting	<1.24E+02	<1.24E+02	<1.73E+02	<1.24E+02	NA
^{230}Th	ICPMS	<6.88E-04	<6.88E-04	<6.88E-04	<6.88E-04	NA
^{232}Th	ICPMS	4.11E-04	<2.75E-04	<2.75E-04	4.11E-04	NA
^{232}U	radiocounting	1.43E-01	1.29E-01	1.13E-01	1.28E-01	11.79
^{233}U	ICPMS	<3.63E+01	<3.63E+01	<3.63E+01	<3.63E+01	NA
^{235}U	ICPMS	3.03E-01	3.23E-01	2.70E-01	2.99E-01	8.93
^{236}U	ICPMS	5.37E-01	2.87E-01	4.12E-01	4.12E-01	30.30
^{237}Np	ICPMS	2.80E+00	2.14E+00	1.87E+00	2.27E+00	20.93
^{238}U	ICPMS	7.63E+00	7.52E+00	7.38E+00	7.51E+00	1.70
^{238}Pu (soluble#1)	radiocounting	6.93E+00	1.43E+01	<4.28E+00	3.90E+01	118.6
^{238}Pu (soluble#2)	radiocounting	1.13E+02	5.58E+01	5.00E+00		
$^{239/40}$Pu (soluble#1)	radiocounting	9.95E+00	<1.10E+01	2.21E+01	1.60E+01	53.63
$^{239/40}$Pu (soluble#2)	radiocounting	<7.34E+00	<2.48E+00	<2.43E+00		
^{241}Pu (soluble)	radiocounting	<9.32E+02	<4.20E+02	<4.95E+02	<4.20E+02	NA
^{238}Pu (total#1)	radiocounting	9.14E+00	<6.44E+00	2.16E+01	2.97E+01	101.3
^{238}Pu (total#2)	radiocounting	8.28E+01	1.94E+01	1.55E+01		
$^{239/40}$Pu (total#1)	radiocounting	1.03E+01	<6.57E+00	1.88E+01	1.46E+01	41.31
$^{239/40}$Pu (total#2)	radiocounting	<4.59E+00	<5.63E+00	<2.73E+00		
^{241}Pu (total)	radiocounting	<5.40E+02	<4.05E+02	<4.86E+02	<4.05E+02	NA
^{242}Pu	ICPMS	<9.55E+00	<9.55E+00	<9.55E+00	NA	NA
^{244}Pu	ICPMS	<6.64E-02	<6.64E-02	<6.64E-02	NA	NA
^{241}Am	radiocounting	<2.05E+01	<1.28E+01	<8.64E+00	NA	NA
^{242m}Am	radiocounting	<2.47E-01	<5.72E-01	<3.46E-01	NA	NA
^{243}Am	radiocounting	<1.24E+01	<9.32E+00	<9.32E+00	NA	NA
^{242}Cm	radiocounting	<2.04E-01	<4.73E-01	<2.86E-01	NA	NA
^{243}Cm	radiocounting	<3.83E+01	<3.83E+01	<2.95E+01	NA	NA
^{244}Cm	radiocounting	<5.27E-01	<1.94E-01	<4.00E-01	NA	NA
^{245}Cm	radiocounting	<3.15E+01	<2.35E+01	<2.42E+01	NA	NA
^{247}Cm	radiocounting	<6.48E+01	<4.86E+01	<5.13E+01	NA	NA
^{249}Cf	radiocounting	<6.98E+01	<5.18E+01	<5.27E+01	NA	NA
^{251}Cf	radiocounting	<3.58E+01	<2.80E+01	<2.79E+01	NA	NA

Two sets of ^{238}Pu and $^{239/40}$Pu analyses were performed (#1 and #2). The high variability is due to random low levels from plutonium cross-contamination in the cells.

Table 7. Tank 22H Chemical Results (mg/L unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
As	AA	<0.0278	<0.0280	<0.0280	<0.0279	NA
Se	AA	<0.055	<0.055	<0.055	<0.055	NA
Br^-	IC	<250	<250	<250	<250	NA
Cl^-	IC	<25	<25	<25	<25	NA
F^-	IC	<25	<25	<25	<25	NA
Formate	IC	171	160	161	164	3.71
NO_3^-	IC	4520	4480	4470	4490	0.59
NO_2^-	IC	7320	7350	7390	7353	0.48
Oxalate	IC	<25	<25	<25	<25	NA
PO_4^{3-}	IC	<25	<25	<25	<25	NA
SO_4^{2-}	IC	42	44	45	43.7	3.50
NH_4^+	IC	<200	<200	<200	<200	NA
CO_3^{2-} [ϒ]	TIC	62.9/126	41.8/127	41.3/127	87.7	49.54
Free OH (M)	Wet chem	0.140	0.141	0.142	0.141	0.71
wt% Soluble solids (%) [∂]	measurement	2.52	2.62	2.67	2.60	2.93
TPB	HPLC	<10	<10	<10	<10	NA
Phenol	HPLC	<10	<10	<10	<10	NA
EDTA	HPLC	<100	<100	<100	<100	NA
Butanol	SVOA	<1	<1	<1	<1	NA
Isopropanol	VOA	<0.25	<0.25	<0.25	<0.25	NA
Benzene	VOA	<0.25	<0.25	<0.25	<0.25	NA
toluene	SVOA	<1	<1	<1	<1	NA
TOC[ϒ]	TOC	225/165	157/128	135/142	159	19.48
Methanol [f]	calculated	NA	NA	NA	308	NA

[∂] This was a filtered sample. However, as a filtration produced no solids, this value can be used for total insoluble solids, too.
[ϒ] Submitted 2 sets of samples, for 6 results total
[f] Methanol result is calculated from the TOC result (less formate) – this number is grossly conservative

Table 8. ICPES Results for Tank 22H (mg/L unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Ag	ICPES	<0.0121	<0.0121	<0.0121	<0.0121	NA
Al	ICPES	15.80	16.30	16.30	16.13	1.79
B	ICPES	3.23	8.09	8.12	6.48	43.44
Ba	ICPES	<0.115	<0.0764	<0.0764	<0.0764	NA
Ca	ICPES	1.36	1.36	1.38	1.37	0.84
Cd	ICPES	<0.0909	<0.0909	<0.0909	<0.0909	NA
Ce	ICPES	<0.290	<1.15	<1.15	<0.290	NA
Co	ICPMS	<7.50E-03	<7.50E-03	<7.50E-03	<7.50E-03	NA
Cr	ICPES	0.164	0.141	0.141	0.149	8.93
Cu	ICPES	<0.106	<0.0449	<0.0449	<0.0449	NA
Fe	ICPES	0.257	0.206	0.205	0.223	13.36
K	ICPES	8.27	<5.89	<5.89	8.27	NA
La	ICPES	<0.0890	<0.0890	<0.0890	<0.0890	NA
Li	ICPES	6.35	3.92	3.91	4.73	29.74
Mg	ICPES	<0.0194	<0.0194	<0.0194	<0.0194	NA
Mn	ICPES	<0.0282	<0.0282	<0.0282	<0.0282	NA
Mo	ICPES	<0.322	<0.322	<0.322	<0.322	NA
Na	ICPES	9420	9390	9390	9400	0.18
Ni	ICPES	<0.227	<1.11	<1.11	<0.227	NA
P	ICPES	1.20	1.33	1.28	1.27	5.16
Pb	ICPES	<0.538	<0.538	<0.538	<0.538	NA
S	ICPES	<15.90	<15.90	<15.90	<15.90	NA
Si	ICPES	114	112	112	112.7	1.02
Sr	ICPES	<0.010	<0.0483	<0.0483	<0.0483	NA
Ti	ICPES	<0.0533	<0.0179	<0.0179	<0.0179	NA
Zn	ICPES	<0.106	<0.106	<0.109	<0.106	NA
Zr	ICPES	<0.0354	<0.0354	<0.0354	<0.0354	NA

4.2 TANK 41H RESULTS

The testing requirements for the Tank 41H sample are derived from a customer document.[29] Tank 41H has fewer requirements than Tank 22H.

4.2.1 Tank 41H Radiochemical Results

Table 9 lists the results of the radiochemical analyses for the Tank 41H samples. The most notable results are the high ^{137}Cs activity (9.56E+07 pCi/mL) and the relatively low ^{90}Sr activity (5.79E+03 pCi/mL – less than observed in the Tank 22H samples).

Table 9. Tank 41H Radiochemical Results (pCi/mL)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Total Alpha	radiocounting	<4.05E+05	<4.37E+05	<4.55E+05	<4.05E+05	NA
Total Alpha, Cs-removed	radiocounting	3.80E+04	3.67E+04	3.67E+04	3.71E+04	2.06
^{90}Sr	radiocounting	5.63E+03	5.85E+03	5.90E+03	5.79E+03	2.50
^{93}Mo	calculated	<9.61E+04	<9.61E+04	<9.61E+04	<9.61E+04	NA
^{93m}Nb	calculated	<2.09E+07	<2.09E+07	<2.09E+07	<2.09E+07	NA
^{134}Cs	radiocounting	<6.84E+03	<7.47E+03	<6.03E+03	<6.03E+03	NA
^{137}Cs	radiocounting	9.18E+07	9.95E+07	9.54E+07	9.56E+07	4.01
^{229}Th	radiocounting	<1.03E+06	<1.06E+06	<1.04E+06	<1.03E+06	NA
^{232}U	radiocounting	8.01E+00	3.87E+00	3.02E+00	4.97E+00	53.77
^{233}U	ICPMS	<1.21E+02	<1.21E+02	<1.21E+02	<1.21E+02	NA
^{235}U	ICPMS	2.27E-01	2.13E-01	1.97E-01	2.12-01	7.00
^{236}U	ICPMS	2.73E+00	2.65E+00	2.33E+00	2.57E+00	8.24
^{238}U	ICPMS	5.70E-01	5.58E-01	5.83E-01	5.70E-01	2.18
^{238}Pu (soluble)	radiocounting	3.34E+04	3.57E+04	3.61E+04	3.46E+04	4.08
$^{239/40}$Pu (soluble)	radiocounting	6.80E+02	7.52E+02	7.29E+02	7.04E+02	5.12
^{241}Pu (soluble)	radiocounting	<8.24E+03	<9.41E+03	<7.25E+03	<7.25E+03	NA
^{238}Pu (total)	radiocounting	4.95E+04	5.18E+04	5.36E+04	5.15E+04	3.93
$^{239/40}$Pu (total)	radiocounting	7.43E+02	7.97E+02	7.70E+02	7.56E+02	3.51
^{241}Pu (total)	radiocounting	<1.05E+04	<1.28E+04	<1.09E+04	<1.05E+04	NA

4.2.2 Tank 41H Chemical Results

Table 10 lists the results of the chemical analyses for the Tank 41H samples. The most notable results are the high sodium concentration (188,336 mg/L or 8.19 M), and the free hydroxide concentration of 1.05 M.

Table 10. Tank 41H Chemical Results (mg/L unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Al	ICPES	17454	17701	18103	17752	1.85
Cr	ICPES	49.65	50.36	51.52	50.51	1.87
Fe	ICPES	10.06	9.48	9.08	9.54	5.15
K	ICPES	521	518	555	532	3.87
Na	ICPES	184955	189001	191051	188336	1.65
P	ICPES	594	611	629	611	2.90
S	ICPES	5887	5881	6071	5947	1.82
Si	ICPES	<137	<150	<146	<137	NA
Br^-	IC	<500	<500	<500	<500	NA
Cl^-	IC	<500	<500	<500	<500	NA
F^-	IC	<500	<500	<500	<500	NA
Formate	IC	<500	<500	<500	<500	NA
NO_3^-	IC	385000	317000	357000	353000	9.68
NO_2^-	IC	11000	9760	9740	10167	7.10
Oxalate	IC	480	435	402	439	8.92
PO_4^{3-}	IC	<2500	<2500	<2500	<2500	NA
SO_4^{2-}	IC	19700	16800	16400	17633	10.21
CO_3^{2-} [ר]	TIC	4162/4271	4126/4173	4194/3945	4145	2.64
Free OH (M)	Wet chem	1.05	1.03	1.06	1.05	1.46
TPB	HPLC	<10	<10	<10	<10	NA
EDTA	HPLC	<100	<100	<100	<100	NA
Butanol	SVOA	<1	<1	<1	<1	NA
Isopropanol	VOA	<0.250	<0.250	<0.250	<0.250	NA
Benzene	VOA	<0.250	<0.250	<0.250	<0.250	NA
toluene	SVOA	<1	<1	<1	<1	NA
TOC[ר]	TOC	1776/778	1552/989	1641/1121	1310	30.68
Methanol [ק]	calculated	NA	NA	NA	3177	NA

4.2.3 Tank 41H Solids Results

The Tank 41H sample (sample ID: "Tank 41 composite 8-4-08 T.B.P.") was filtered and the solids retained for analysis. The solids were digested (aqua regia) and sent forth for analysis by ICPES, ICPMS, PuTTa, Sr-90, and gammascan. The results are shown in Table 11.

[ר] Submitted 2 sets of samples, for 6 results total

[ק] Methanol result is calculated from the TOC result (less oxalate) – this number is grossly conservative

Table 11. Tank 41H Solids Analyses Results (μg/g unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Al	ICPES	165000	177000	171000	171000	3.51
Ba	ICPES	18.8	21.6	19.5	20.0	7.30
Ca	ICPES	395	442	414	417	5.67
Cd	ICPES	<1.77	<1.69	<1.74	<1.69	NA
Cr	ICPES	290	327	303	307	6.12
Cu	ICPES	15.9	17.7	16.8	16.8	5.36
Fe	ICPES	1440	1660	1510	1537	7.31
K	ICPES	236	255	254	248	4.31
Li	ICPES	<3.64	<3.47	<3.47	<3.47	NA
Mg	ICPES	34.9	39.3	35.8	36.7	6.34
Mn	ICPES	219	253	232	235	7.31
Na	ICPES	111000	103000	102000	105333	4.68
Ni	ICPES	39.9	45.5	41.7	42.4	6.75
P	ICPES	309	340	320	323	4.87
S	ICPES	2480	2940	2540	2653	9.42
Si	ICPES	755	906	784	815	9.83
Sr	ICPES	1.18	1.33	1.24	1.25	6.04
Ti	ICPES	4.88	5.55	5.22	5.22	6.42
U	ICPES	<69.6	<66.4	<68.1	<66.4	NA
Zn	ICPES	79.4	86.0	81.7	82.4	4.07
^{235}U (pCi/g)	ICPMS	2.04E+01	1.94E+01	1.88E+01	1.95E+01	4.22
^{238}U (pCi/g)	ICPMS	1.89E+01	2.11E+01	1.96E+01	1.99E+01	5.66
^{237}Np (pCi/g)	ICPMS	1.83E+03	1.57E+03	1.99E+03	1.80E+03	11.92
^{238}Pu (pCi/g)	radiocounting	8.83E+06	1.06E+07	9.73E+06	9.73E+06	9.26
$^{239/40}$Pu (pCi/g)	radiocounting	<4.01E+04	<4.50E+04	<3.02E+04	<3.02E+04	NA
^{241}Pu (pCi/g)	radiocounting	<1.03E+07	<1.47E+07	<1.04E+07	<1.03E+07	NA
^{60}Co (pCi/g)	radiocounting	<9.41E+03	<7.93E+03	<7.43E+03	<7.43E+03	NA
^{90}Sr (pCi/g)	radiocounting	1.87E+06	1.25E+06	2.00E+06	1.71E+06	23.50
^{137}Cs (pCi/g)	radiocounting	5.32E+07	6.26E+07	5.59E+07	5.72E+07	8.52
^{154}Eu (pCi/g)	radiocounting	<2.30E+04	<2.70E+04	<2.37E+04	<2.30E+04	NA
^{241}Am (pCi/g)	radiocounting	<1.65E+05	<1.74E+05	<1.68E+05	<1.65E+05	NA

4.3 TANK 49H CONFIRMATORY SAMPLE RESULTS

4.3.1 Tank 49H Confirmatory Sample Radiochemical Results

Table 12 lists the results of the radiochemical analyses for the combined Tank 49H confirmatory samples.

Table 12. Tank 49H Confirmatory Sample Radiochemical Results (pCi/mL)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Total Alpha	radiocounting	<1.08E+05	<1.02E+05	<1.02E+05	<1.02E+05	NA
Total Alpha, Cs-removed	radiocounting	<2.90E+04	<2.94E+04	<2.86E+04	<2.86E+04	NA
Total Beta	radiocounting	6.39E+07	6.35E+07	6.39E+07	6.38E+07	0.41
Total Beta, Cs-removed	radiocounting	6.08E+05	6.08E+05	6.03E+05	6.06E+05	0.43
^{3}H	radiocounting	7.49E+02	<7.75E+02	<6.82E+02	7.49E+02	NA
^{14}C	radiocounting	5.63E+02	5.63E+02	6.21E+02	5.82E+02	5.80
^{90}Sr	radiocounting	2.97E+05	2.93E+05	2.87E+05	2.93E+05	1.78
^{99}Tc	radiocounting	6.89E+04	6.89E+04	6.89E+04	6.89E+04	0.00
^{129}I	radiocounting	<5.00E+01	<3.38E+01	<3.90E+01	<3.38E+01	NA
^{134}Cs	radiocounting	<3.30E+03	<3.32E+03	<3.31E+03	<3.30E+03	NA
^{137}Cs	radiocounting	5.36E+07	5.45E+07	5.22E+07	5.34E+07	2.12
^{233}U	ICPMS	<1.45E+03	<1.45E+03	<1.45E+03	<1.45E+03	NA
^{234}U	ICPMS	<3.13E+02	<3.13E+02	3.24E+02	3.24E+02	NA
^{235}U	ICPMS	2.72E-01	2.56E-01	2.22E-01	2.50E-01	10.14
^{236}U	ICPMS	<4.85E+00	5.18E+00	<4.85E+00	5.18E+00	NA
^{238}U	ICPMS	1.69E+00	1.67E+00	1.65E+00	1.67E+00	1.30
^{237}Np	ICPMS	<7.05E+01	<7.05E+01	<7.05E+01	<7.05E+01	NA
^{238}Pu (soluble)	radiocounting	2.55E+04	3.06E+04	2.34E+04	2.65E+04	14.00
$^{239/40}$Pu (soluble)	radiocounting	2.09E+02	2.44E+02	2.07E+02	2.20E+02	9.39
^{241}Pu (soluble)	radiocounting	<2.77E+03	<3.16E+03	<2.52E+03	<2.52E+03	NA
^{238}Pu (total)	radiocounting	3.89E+05	2.97E+05	3.19E+05	3.35E+05	14.37
$^{239/40}$Pu (total)	radiocounting	6.17E+03	5.18E+03	5.54E+03	5.63E+03	8.91
^{241}Pu (total)	radiocounting	9.05E+04	6.80E+04	7.34E+04	7.73E+04	15.21

For the ^{233}U values, the confirmatory samples had a high detection limit. The change in the detection limit is sample dependant and can change over time. However, the higher detection limit gives the impression of a higher than expected quantity of ^{233}U. In this case, it is appropriate to use the previous measurements of the tank constituents [9] and generate a lower value for the ^{233}U content. Using the Tank 22H (<36.3 pCi/mL), 41H (<121 pCi/mL) and the

previous 49H measurement (<161 pCi/mL) [9], we generate a new "average" ^{233}U detection limit of <119 pCi/mL (volume-weighted average of the tank constituents).

4.3.2 Tank 49H Confirmatory Sample Chemical Results

Table 13 lists the results of the chemical analyses for the combined Tank 49H confirmatory samples.

Table 13. Tank 49H Confirmatory Sample Chemical Results (mg/L unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Al	ICPES	6920	7260	7020	7067	2.47
Ca	ICPES	1.68	1.13	1.70	1.50	21.52
Cr	ICPES	58.0	60.9	59.0	59.3	2.48
Fe	ICPES	1.77	2.01	1.92	1.90	6.38
K	ICPES	241	248	236	242	2.49
Mo	ICPES	7.36	7.99	7.46	7.60	4.45
Na	ICPES	125000	131000	127000	127667	2.39
Na (M)	ICPES	5.43	5.70	5.52	5.55	2.48
P	ICPES	373	396	386	385	3.00
S	ICPES	3550	3840	3670	3687	3.95
Si	ICPES	47.20	49.60	47.90	48.23	2.56
Zn	ICPES	1.42	1.45	1.37	1.41	2.86
Hg	CV-Hg	8.97	9.14	9.03	9.05	0.95
Br^-	IC	<250	<250	<250	<250	NA
Cl^-	IC	<250	<250	<250	<250	NA
F^-	IC	<250	<250	<250	<250	NA
Formate	IC	323	330	324	326	1.16
NO_3^-	IC	140000	131000	125000	132000	5.72
NO_2^-	IC	9980	10300	10300	10193	1.81
Oxalate	IC	<250	<250	<250	<250	NA
PO_4^{3-}	IC	797	796	800	798	0.26
SO_4^{2-}	IC	9230	8760	8790	8927	2.95
NH_4^+	IC	<200	<200	<200	<200	NA
TPB	HPLC	<10	<10	<10	<10	NA
EDTA	HPLC	<100	<100	<100	<100	NA
Butanol	SVOA	<1	<1	<1	<1	NA
Isopropanol	VOA	<0.25	<0.25	<0.25	<0.25	NA
Benzene	VOA	<0.25	<0.25	<0.25	<0.25	NA
toluene	SVOA	<1	<1	<1	<1	NA
pH	pH paper	13-14	13-14	13-14	13-14	0.5 units
Free Hydroxide	Wet chem	2.14 M (single sample)				10
Total Solids	wetchem	30.87	31.27	31.54	31.23	1.08
Suspend.(insoluble) Solids	wetchem	0.219	0.86	1.181	0.753	65.0
Turbidity (NTU)	turbidometer	8.97	7.80	8.79	8.52	7.39

4.3.3 Tank 49H Solids Results

The Tank 49H original sample (TS124-07-A-101178) was filtered and the solids were retained for analysis. The solids were digested (aquaregia) and sent forth for analysis by ICPES, ICPMS, PuTTa, Sr-90, and gammascan. The results are shown in Table 14.

Table 14. Tank 49H Solids Analyses Results (μg/g unless otherwise noted)

Analyte	Method	Result 1	Result 2	Result 3	Average	%STDEV
Al	ICPES	202000	208000	202000	204000	1.70
Ba	ICPES	708	742	722	724	2.36
Ca	ICPES	2430	2540	2480	2483	2.22
Cd	ICPES	22.0	22.0	21.6	21.9	1.06
Cr	ICPES	6110	6390	6230	6243	2.25
Cu	ICPES	127	138	130	132	4.32
Fe	ICPES	12600	13100	12700	12800	2.07
K	ICPES	<423	<457	<440	<423	NA
Li	ICPES	173	179	176	176	1.70
Mg	ICPES	540	564	546	550	2.27
Mn	ICPES	1920	2010	1960	1963	2.30
Na	ICPES	18300	18900	18300	18500	1.87
Ni	ICPES	303	322	313	313	3.04
P	ICPES	273	273	270	272	0.64
S	ICPES	<1140	<1230	<1190	<1140	NA
Si	ICPES	6030	6070	5850	5983	1.96
Sr	ICPES	31.5	33	32.2	32.2	2.33
Ti	ICPES	92.4	94.9	91.6	93.0	1.85
U	ICPES	637	656	653	649	1.57
Zn	ICPES	272	280	272	275	1.68
^{235}U (pCi/g)	ICPMS	1.65E+01	1.52E+01	1.50E+01	1.56E+01	5.08
^{238}U (pCi/g)	ICPMS	7.56E+01	7.90E+01	7.76E+01	7.74E+01	2.19
^{237}Np (pCi/g)	ICPMS	2.50E+03	2.45E+03	2.44E+03	2.47E+03	1.35
^{238}Pu (pCi/g)	radiocounting	6.85E+07	7.70E+07	8.96E+07	7.84E+07	13.59
$^{239/40}$Pu (pCi/g)	radiocounting	1.16E+06	8.56E+05	9.46E+05	9.88E+05	15.93
^{241}Pu (pCi/g)	radiocounting	<2.56E+08	<9.19E+07	<4.40E+08	<9.19E+07	NA
^{60}Co (pCi/g)	radiocounting	<4.20E+04	<4.73E+04	3.60E+04	3.60E+05	NA
^{90}Sr (pCi/g)	radiocounting	8.60E+08	9.10E+08	5.63E+08	7.78E+08	24.12
^{137}Cs (pCi/g)	radiocounting	9.10E+07	9.59E+07	8.96E+07	9.22E+07	3.60
^{154}Eu (pCi/g)	radiocounting	1.01E+06	1.03E+06	9.37E+05	9.73E+05	4.98
^{241}Am (pCi/g)	radiocounting	8.83E+05	<6.94E+05	1.10E+06	9.48E+05	9.74

4.4 ARP RESULTS

After input from the customer, SRNL prepared a composite of the tank samples designed to mimic the estimated composition of Tank 49H after the addition of Tank 22H, 41H, and sodium hydroxide (Table 3). Researchers generated two, 100-mL composites – one for the experiment, and one for the control.

4.4.1 Plutonium Results

For the MST strike, researchers analyzed the filtered samples for ^{238}Pu. Table 15 shows the plutonium results while Figure 1 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. While the TTR requested analysis of samples for 6 hours after the strike, due to time limitations in the cells, personnel pulled the 6 hour samples early at 3 hours. Since filtration times in ARP are currently limiting the ability to operate at strike times less than 12 hours, SRNL has not analyzed the 3 hour samples at this time.

The uncertainty in Table 15 is the analytical uncertainty associated with the measurement and does not include any contribution to uncertainty due to experimental and sampling methods.

Table 15. ^{238}Pu Concentrations in the MST Strike Filtrates

Time (hours)	Experiment	Control
	^{238}Pu (pCi/mL)	^{238}Pu (pCi/mL)
0*	2.46(±0.143)E+04	2.46(±0.143)E+04
12	4.30(±0.223)E+03	2.93(±0.218)E+04
24	4.36(±0.257)E+03	2.82(±0.169)E+04

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

Figure 1. ^{238}Pu in Solution over Time for the MST Strike

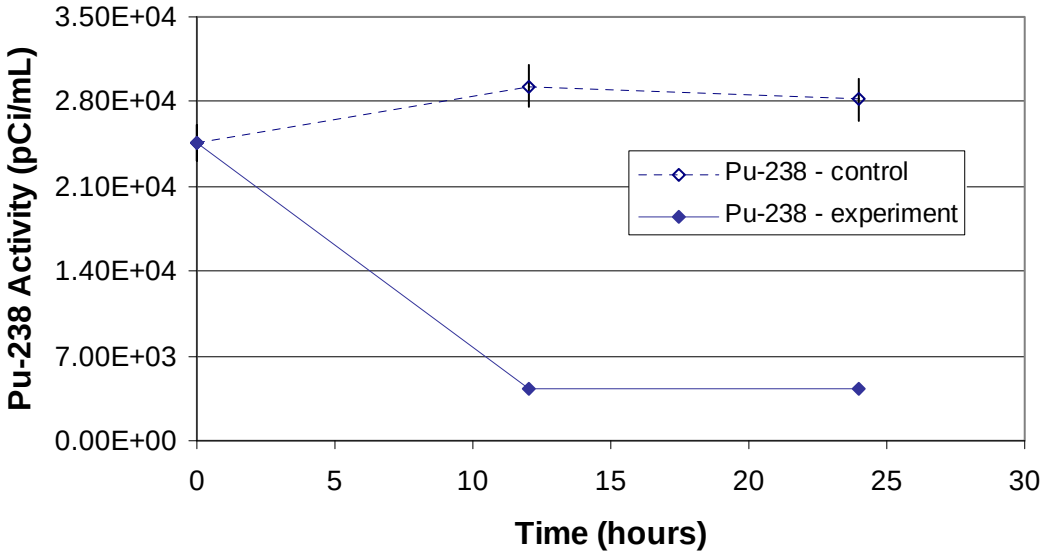

Table 16 lists the decontamination factors (DF) after the first MST strike.

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

Time (hours)	Experiment	Control
	DF	DF
12	5.73	0.84
24	5.64	0.87

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.

4.4.2 Strontium Results

For the MST strike, researchers analyzed the filtered samples for ^{90}Sr. Table 17 shows the plutonium results while Figure 2 shows the graphical results for ^{90}Sr. While the TTR requested analysis of samples for 6 hours after the strike, due to time limitations in the cells, personnel pulled the 6 hour samples early at 3 hours. Since filtration times in ARP are currently limiting the ability to operate at strike times less than 12 hours, SRNL did not analyze the 3 hour samples.

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

Time (hours)	Experiment	Control
	^{90}Sr (pCi/mL)	^{90}Sr (pCi/mL)
0*	1.91(±0.160)E+05	1.91(±0.160)E+05
12	2.15(±0.193)E+03	1.76(±0.142)E+05
24	2.70(±0.262)E+03	1.71(±0.135)E+05

*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 2. ^{90}Sr in Solution over Time for the MST Strike

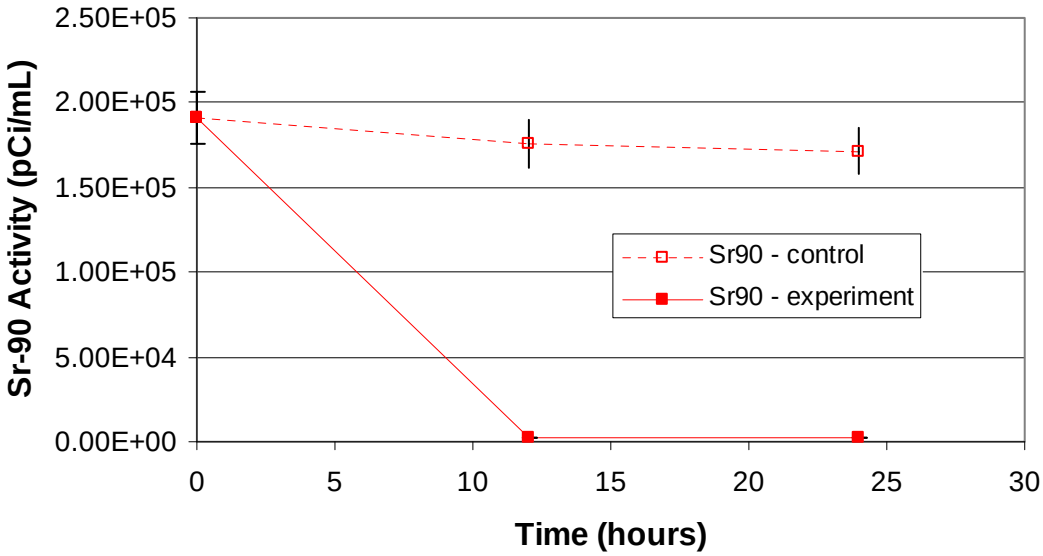

Table 18 lists the decontamination factors (DF) after the first MST strike.

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

Time (hours)	Experiment	Control
	DF	DF
12	88.9	1.09
24	70.9	1.11

The slight decline in strontium DF from 12 to 24 hours seems to be a real effect, and is behavior SRNL has observed before.[30]

4.4.3 Cesium Results

For the MST strike, researchers analyzed the filtered samples for ^{137}Cs. 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 19 shows the plutonium results while Figure 3 shows the graphical results for ^{137}Cs.

Table 19. ^{137}Cs Concentrations in the MST Strike Filtrates

Time (hours)	Experiment ^{137}Cs (pCi/mL)	Control ^{137}Cs (pCi/mL)
0*	4.74(±0.0531)E+07	4.74(±0.0531)E+07
12	4.81(±0.0534)E+07	5.43(±0.0603)E+07
24	4.82(±0.0540)E+07	5.08(±0.0565)E+07

*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 3. ^{137}Cs in Solution over Time for the MST Strike

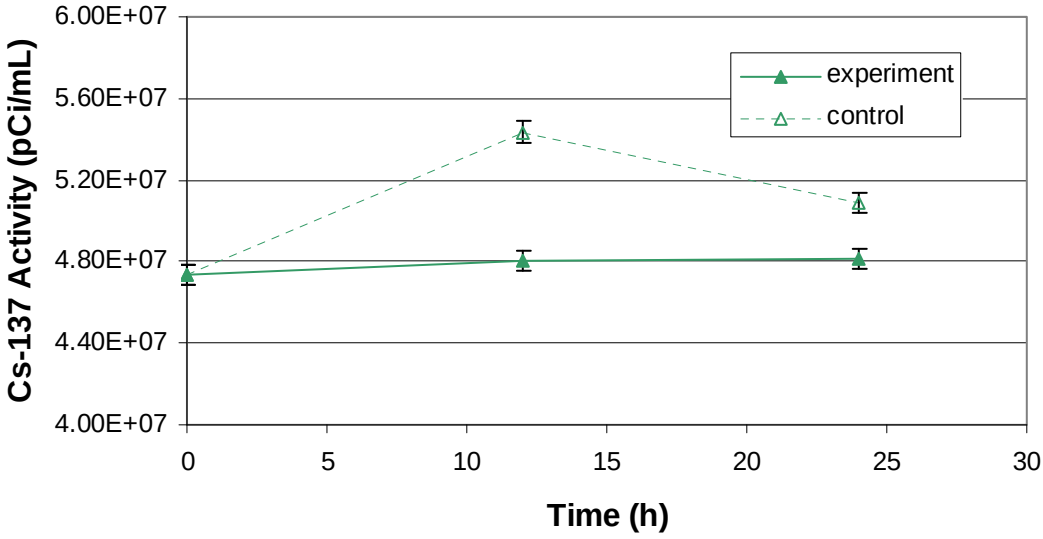

Table 20 lists the decontamination factors (DF) after the first MST strike.

Table 20. ^{137}Cs Decontamination Factors over Time

Time (hours)	Experiment	Control
	DF	DF
12	0.985	0.872
24	0.983	0.931

4.4.4 Analysis of MST Solids

After the MST test completed, personnel digested (sodium peroxide fusion) of the retained solids and sent to AD for analysis. Table 21 lists the results of the MST solids analyses.

Table 21. Tank 49H MST Solids Analyses Chemical Results (mg/L unless otherwise noted)

Analyte	Method	Result 1	% Uncertainty
Ag	ICPES	<0.113	NA
Al	ICPES	24.1	10%
As	AA	<0.0275	NA
B	ICPES	6.61	10%
Ba	ICPES	0.105	10%
Ca	ICPES	20.7	10%
Cd	ICPES	<0.037	NA
Co	ICPMS	<1.25E-02	NA
Cr	ICPES	0.24	10%
Cu	ICPES	<0.0316	NA
Fe	ICPES	0.639	10%
Gd	ICPES	<0.14	NA
Hg	CV-Hg	<0.02	NA
K	ICPES	5.33	10%
La	ICPES	<0.0663	NA
Mg	ICPES	7.9	10%
Mn	ICPES	<0.072	NA
Mo	ICPES	<0.215	NA
Na	ICPES	6.72E+03 [Y]	10%
Ni	ICPES	<0.171	NA
P	ICPES	1.21	10%
Pb	ICPES	<0.522	NA
S	ICPES	1.61E+04	10%
Sb	ICPES	<0.889	NA
Se	AA	<0.055	NA
Si	ICPES	157	10%
Sn	ICPES	<0.619	NA
Ti	ICPES	28.8	10%
U	ICPES	<2.64	NA
Zn	ICPES	<0.137	NA
Zr	ICPES	44.4	10%

[Y] relatively high sodium values are from sodium peroxide fusion and are not likely to be indicative of the MST solids

Table 22. Tank 49H MST Solids Radiological Results (pCi/mL)

Analyte	Method	Result 1	% Uncertainty
^{230}Th	ICPMS	<5.28E+01	NA
^{232}U	ICPMS	<3.85E-03	NA
^{233}U	ICPMS	<2.42E+01	NA
^{234}U	ICPMS	<1.56E+01	NA
^{235}U	ICPMS	1.51E-02	20
^{236}U	ICPMS	<1.63E-01	NA
^{238}U	ICPMS	1.12E-01	20
^{237}Np	ICPMS	3.51E+00	20
^{238}Pu	radiocounting	4.68E+03	7.40
$^{239/40}$Pu	radiocounting	3.25E+01	74.60
^{241}Pu	ICPMS	<3.85E+05	NA
Total Alpha	radiocounting	4.09E+03	12.00
Total beta	radiocounting	2.89E+05	10.00
^{60}Co	radiocounting	<9.41E+00	NA
^{90}Sr	radiocounting	3.40E+04	7.68
^{90}Y	calculated	3.40E+04	7.68
^{94}Nb	radiocounting	<8.96E+00	NA
^{99}Tc	radiocounting	2.04E+02	7.03
^{106}Ru	radiocounting	<5.36E+01	NA
^{125}Sb	radiocounting	<2.43E+01	NA
^{126}Sn	radiocounting	<2.52E+01	NA
^{137}Cs	radiocounting	1.94E+05	1.07
^{144}Ce	radiocounting	<6.35E+01	NA
^{147}Pm	radiocounting	<1.12E+03	NA
^{151}Sm	radiocounting	<2.30E+02	NA
^{154}Eu	radiocounting	<1.72E+01	NA
^{155}Eu	radiocounting	<2.91E+01	NA
^{226}Ra	radiocounting	<2.08E+02	NA
^{229}Th	radiocounting	<1.56E+02	NA
^{241}Am	radiocounting	3.28E+01	18.60
^{242m}Am	radiocounting	<3.75E-01	NA
^{243}Am	radiocounting	<6.62E+00	NA
^{242}Cm	radiocounting	<3.11E-01	NA
^{243}Cm	radiocounting	<2.39E+01	NA
^{244}Cm	radiocounting	4.64E+00	42.20
^{245}Cm	radiocounting	<1.98E+01	NA
^{247}Cm	radiocounting	<2.84E+01	NA
^{249}Cf	radiocounting	<3.29E+01	NA
^{251}Cf	radiocounting	<2.34E+01	NA

4.4.5 Activity Balance in the ARP Test

With the digested MST data, we can compare the results to the solution data (average of the ^{238}Pu control data in Table 15 and average of the control data in Table 17 for ^{90}Sr) 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 solid and liquid streams).

From the MST solids digestion, the amount of plutonium and strontium sorbed by the MST can be calculated. In Table 23, the first MST digestion results are listed.

Table 23. Plutonium and Strontium Sorbed on MST from the Digestion Results

	Ti	MST	^{238}Pu	^{90}Sr	units
In digestate	28.80	60	2.74E-04	2.48E-04	mg/L
Normalize to MST			4.57E-06	4.13E-06	g /g MST
g Pu or Sr			1.85E-07	1.68E-07	g
μg Pu or Sr			1.86E-01	1.68E-01	μg

Due to the fact that MST collection is not perfect, the plutonium and strontium results are normalized to the digestate titanium results, giving gram of Pu/Sr per gram of MST. We analyzed the digestate to have 28.8 mg/L of titanium, which corresponds to 60 mg/L of MST solids. The normalized values are then multiplied by the amount of MST solids introduced (0.0407 g) to give the grams and micrograms of plutonium and strontium sorbed onto the entire added mass of MST.

From the solution results (average of the ^{238}Pu control data in Table 15 and average of the control data in Table 17 for ^{90}Sr), we can calculate the μg of Pu and Sr are in the original Tank 49H material after the addition of MST (Table 24) to the 100 mL of composite solution.

Table 24. Plutonium and Strontium (μg) Left in the Tank 49H Solution

CSS Component	pCi/mL	μg (in 100 mL)
^{238}Pu	4.36E+03	2.55E-02
^{90}Sr	2.70E+03	1.98E-03

The respective sums of ^{238}Pu and ^{90}Sr on the MST and the amounts in the post-Tank49H solution are given in Table 25.

Table 25. Respective Sums of ^{238}Pu and ^{90}Sr in the MST and Tank 49H Solution

CSS Component	μg
^{238}Pu	2.12E-01
^{90}Sr	1.70E-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 26).

Table 26. Activity Balance for the First Test

Component	Amount on MST (μg)	Amount in post-MST solution (μg)	Amount starting in Tank 49H (μg)	Balance (%)
^{238}Pu	0.186	0.0255	0.160	132
^{90}Sr	0.168	0.00198	0.132	129

The activity balance for both ^{238}Pu and ^{90}Sr is above 100%. This is likely due to two factors. First, we are making a comparison of three measurements via a sum. Hence, the uncertainty in the sum is three times the uncertainty in the measurements assuming the same uncertainty in each measurement. If we take the uncertainty from the Pu and Sr measurements of the control, we would expect the Pu balance uncertainty to be ~27% and that of the Sr to be ~17.4%. In actuality, the uncertainty in the digested solids measurement is even greater due to additional handling steps and losses. In fact, at 95% certainty (using 2 sigma), the closure falls within expected bounds. Additionally, the picture is clouded by the presence of an amount of insoluble ^{238}Pu. While we cannot prove conclusively (the composite used in the MST test may or may not physically mimic the Tank 49H confirmatory sample), the Tank 49H confirmatory analyses suggest that 93% of the ^{238}Pu is an insoluble form. With insoluble ^{238}Pu, we cannot clearly discern in the activity balance how much of the insoluble ^{238}Pu was actually bound to the MST, and how much was "along for the ride" when the filtration and collection occurred. Indeed, having the activity balance much higher than 100% suggests the presence of insoluble ^{238}Pu and ^{90}Sr insoluble solids.

4.5 ESS RESULTS

Material from the ARP (MST strike) test was used in an ESS test. For this test, we used a nominal starting volume of 90 mL of aqueous feed, and 30 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI).

Table 27 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.[8]

Table 27. 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, ISDP 1 (Tank 49H test)	9.07	1.60	1.29	0.070	0.046	0.042
S2-D1-YES BOB-T-WI, ISDP 2 (current test)	14.64	1.51	2.13	0.74	0.09	0.031
Current test, volume corrected	12.50		1.60	0.38		

The current test shows acceptable values except for the Scrub #2 and Strip #1 results. This deviation is due to variations in the aqueous volumes for the Extraction, Scrub #2, and Strip #1 steps from the targeted volumes. We can correct the distribution calculations for those steps that deviated from calculated aqueous amounts. If we do so, the extraction value drops slightly (but is still acceptable), the Scrub #2 value falls within specification, and the Strip #1 value is still out of specification, but much closer to the target. Researchers confirmed that the pH values did not deviate from predicted values – indicating minimal carryover of the other liquid phase at each separation.

4.5.1 Strip Effluent and DSS Results

During, and at the end of the ESS test, the researched measured the gamma activity in the strip effluent and the decontaminated salt solution (DSS). The results are shown in Table 28.

Table 28. Strip Effluent and DSS Results

Sample	Measurement	Result
Strip Effluent#1	^{137}Cs activity	1.01E+08 dpm/mL
Strip Effluent#1	pH	1
Strip Effluent#2	^{137}Cs activity	4.10E+08 dpm/mL
Strip Effluent#2	pH	1
Strip Effluent#3	^{137}Cs activity	8.86E+07 dpm/mL
Strip Effluent#3	pH	5
DSS	^{137}Cs activity	1.55E+07 dpm/mL
DSS	pH	14

The analytical uncertainty on the 137 activity is 10% and ±1 pH unit for the pH measurement.

4.6 TURBIDITY MONITORING

After the completion of the MST test, the control bottle was periodically monitored for turbidity values over ~3 weeks (Table 29).

Table 29. Turbidity of the Control Sample Over Time

Time (after MST strike)	Turbidity (NTU)
2	5.07
10	4.89
16	11.1
23	7.70

The measurements show that the turbidity slowly increased over the time period, reaching a peak of 11.1 NTU, which indicates gradual solids formation. (Using a previous formalism,[31] the solids content at the end of this period is estimated as ~4.5 mg/L).

5.0 CONCLUSIONS

This report covers the methods and data for ISDP Salt Batch 2 feed sample qualification. The following observations are made from the work.

- The composite solution mimicking the planned composite in Tank 49H shows trace precipitation of solids (i.e., < 5 ppm) within the first 23 days after preparation.

- A demonstration of the monosodium titanate removal of strontium and actinides provided acceptable 24 hour decontamination values for Pu and Sr of 5.64 and 70.9, respectively.

- A demonstration of cesium extraction, scrubbing and stripping – prototypical of the Modular Caustic-Side Solvent Extraction Unit – yielded cesium mass transfer behavior within acceptable norms. The measured distribution values are: 14.64, 1.51, 2.13, 0.74, 0.09 and 0.031 for Extraction, Scrub #1, Scrub #2, Strip #1, Strip #2, and Strip #3, respectively. Adjusting the experiment organic-aqueous values to match the planned operational values yields distribution values of 12.50, 1.51, 1.60, 0.38, 0.09 and 0.031 for Extraction, Scrub #1, Scrub #2, Strip #1, Strip #2, and Strip #3, respectively.

- Requested chemical and radionuclide compositions are reported within for the samples from Tank 22H and Tank 41H.

Analyses of a sample from Tank 49H collected after all additions provide the following conclusions.

- The physical measurements of the Tank 49H confirmatory sample (density and turbidity) are within the expected range of results.

- The total plutonium content was 3.35E+05 pCi/mL for ^{238}Pu, 5.63E+03 pCi/mL for $^{239/40}$Pu, and 7.73E+04 pCi/mL for ^{241}Pu.

- The ^{235}U content was measured to be 0.250 pCi/mL.

- There were less than detectable amounts of organic analytes (i.e., butanol, isopropanol, tetraphenylborate, tributyl phosphate, ethylenediaminetetraacetate), except for formate, which was present at a 326 mg/L concentration.

- The measured insoluble solids content was 0.753 wt%.

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