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**RPP-RPT-59874 , Rev. 0**

# **Test Report for Cesium and Solids Removal from an 11.5L Composite of Archived Hanford Double Shell Tank Supernate for Off-Site Disposal**

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**Abstract:** The 222-S Laboratory blended supernate waste from Hanford Tanks 241-AN-101, 241-AN-106, 241-AP-105, 241-AP-106, 241-AP-107, and 241-AY-101 from the hot cell archive to create a bulk composite. The composite was blended with 600 mL 19.4 M NaOH, which brought the total volume to approximately 11.5 L (3 gal). The composite was filtered to remove solids and passed through spherical resorcinol-formaldehyde ion-exchange resin columns to remove cesium. The composite masses were tracked as a treatability study. Samples collected before, during, and after the ion-exchange process were characterized for a full suite of analytes (inorganic, organic, and radionuclides) to aid in the classification of the waste for shipping, receiving, treatment, and disposal determinations.

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RPP-RPT-59874

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**S. R. Doll**

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Washington River Protection Solutions LLC

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**RPP-RPT-59874****Executive Summary**

The 222-S Laboratory blended supernate waste from Hanford Tanks 241-AN-101, 241-AN-106, 241-AP-105, 241-AP-106, 241-AP-107, and 241-AY-101 from the hot cell archive to create a bulk composite. The composite was blended with 600 mL 19.4 M NaOH, which brought the total volume to approximately 11.5 L (3 gal). The composite was filtered to remove solids and passed through spherical resorcinol-formaldehyde ion-exchange resin columns to remove cesium. The composite masses were tracked as a treatability study. Samples collected before, during, and after the ion-exchange process were characterized for a full suite of analytes (inorganic, organic, and radionuclides) to aid in the classification of the waste for shipping, receiving, treatment, and disposal determinations.

No cesium breakthrough was observed on the spherical resorcinol-formaldehyde resin columns. The Cs-137 analytical results demonstrated a decontamination factor in excess of 250,000. Because of the high decontamination factor, sampling from the lead column yielded barely detectable or non-detectable cesium. Therefore this work did not yield data for estimation of cesium column breakthrough. Following chemical analysis, the remaining 10.8 L (2.9 gal) of cesium-depleted supernate composite was packaged for shipment to Perma-Fix in 12 1-L bottles and arranged in a 30-gal drum. Dose rates outside the drum were <0.5 mRem/hr.

This work was performed in part to support low activity waste treatment technologies. Information about exchange column performance was obtained as a byproduct of this work.

## RPP-RPT-59874

## CONTENTS

1	Introduction .....	9
2	Composite.....	10
2.1	Archived Material.....	10
2.2	Composite Preparation and Filtration.....	10
3	Ion Exchange Procedures .....	12
3.1	Ion Exchange Resin Pretreatment and Column Preparation .....	12
3.2	Ion Exchange Column and Manifold System.....	13
3.3	Composite Processing and Sampling .....	14
4	Packaging and Shipping .....	17
4.1	Packaging.....	17
4.2	Analytical Requirements .....	17
5	Results and Discussion .....	19
5.1	Aqueous Analysis .....	19
5.1.1	Aqueous Pre-Column Composite Chemistry .....	19
5.1.2	Aqueous Column Performance Chemistry .....	25
5.1.3	Aqueous Post-Column Composite Chemistry.....	25
5.2	Ion Exchange Resin Breakthrough.....	33
5.3	Solid Analysis.....	33
5.3.1	Pre-Column Settled Solids Chemical Analysis .....	34
5.3.2	Suspended Solids SEM Analysis.....	40
5.3.3	Precipitated Solids PLM Analysis.....	41
6	Quality Assurance.....	42
6.1	Laboratory Analyses.....	42
6.2	Holding Times .....	45
6.3	Sample Digestions .....	45
6.3.1	Acid Digestion.....	45
6.3.2	Fusion Digestion.....	45
6.3.3	Water Digestion.....	45
6.4	Data Summary Report .....	45
7	Quality Control .....	46
7.1	Analytical Quality Control Criteria .....	46

## RPP-RPT-59874

7.2	Quality Control Summary for Analytical Data.....	47
7.2.1	Inorganic Constituents.....	47
7.2.2	Radiochemical Constituents .....	50
7.2.3	Organic Constituents .....	52
7.2.4	IC-Anions/Organic Acids .....	61
7.2.5	Mass and Charge Balance.....	61
8	Process Knowledge.....	62
8.1	Organic Process Knowledge and Calculations .....	62
8.2	Radionuclide Process Knowledge .....	64
9	Discussion.....	67
10	Exceptions to Test Plan .....	68
10.1	Precipitate Formation after Filtration .....	68
10.2	Resin Bed Volume.....	68
10.3	Cesium Breakthrough Curve .....	68
10.4	Additional Constituents Requested for Analysis.....	69
10.5	Product Consumption for Analysis .....	69
10.6	Dry Resin Mass Analysis Not Performed.....	69
11	Acknowledgements .....	69
12	Lessons Learned .....	71
13	References .....	73
14	Bibliography .....	76
	<b>APPENDIX A. Tank Archive Supernate Samples Used and Treatability Records.....</b>	<b>A-1</b>
	<b>APPENDIX B. Sample Breakdown Diagram.....</b>	<b>B-1</b>
	<b>APPENDIX C. Tank Waste Archive Photos .....</b>	<b>C-1</b>
	<b>APPENDIX D. Data Summary Reports .....</b>	<b>D-1</b>
	<b>APPENDIX E. Quality Assurance Review Document.....</b>	<b>E-1</b>
	<b>APPENDIX F. WRPS 222-S activity timeline associated with the Test Bed project .....</b>	<b>F-1</b>

**LIST OF FIGURES**

Figure 2-1. Composite Filtration Apparatus.....	11
Figure 2-2. Composite Filtration.....	12
Figure 3-1. H <sup>+</sup> -Form (left) and Na <sup>+</sup> -Form (right) of sRF Resin Used .....	12
Figure 3-2. Polarized Light Microscopy Image of sRF Resin in the H <sup>+</sup> -Form.....	13
Figure 3-3. Ion Exchange Column and Manifold System. .....	14
Figure 3-4. sRF Resin Expansion From the H <sup>+</sup> to Na <sup>+</sup> Form. .....	15

## RPP-RPT-59874

Figure 3-5. Final Product .....	17
Figure 4-1. Product Packaging.....	17
Figure 5-1. SEM Image of Aggregated Sub-Micron Sized Particles in Suspended Solids. ....	41
Figure 5-2. SEM Image of Individual Particles in Suspended Solids.....	41
Figure 5-3. PLM Image of Natrophosphate (left) and Phosphate Needles (right) in Precipitated Solids.....	41

## LIST OF TABLES

Table 5-1. Aqueous Pre-Column Organic Analytes. (2 pages) .....	20
Table 5-2. Aqueous Pre-Column Composite Inorganic Analytes. (2 pages).....	22
Table 5-3. Aqueous Pre-Column Radiological Analytes. (2 pages).....	23
Table 5-4. Aqueous Post-Column Organic Analytes. (2 pages).....	27
Table 5-5. Aqueous Post-Column Inorganic Analytes. (2 pages) .....	29
Table 5-6. Aqueous Post-Column Radiological Analytes. (2 pages) .....	31
Table 5-7. Inorganic Analysis of Pre-Column Settled Solids. (2 pages).....	34
Table 5-8. Radiological Analysis of Pre-Column Settled Solids. (2 pages).....	35
Table 5-9. Constituent Mass of Pre-Column Settled Solids. (2 pages) .....	36
Table 5-10. Phase Composition of Pre-Column Settled Solids. ....	39
Table 6-1. Liquid Analytical Procedures .....	43
Table 6-2. Solids Analytical Procedures.....	44
Table 7-1. Analysis Qualifier Flag Definitions.....	47
Table 8-1. Bounding Cl-36 to Tc-99 Ratio from Fuel Processing Estimates.....	65
Table 8-2. Estimated Tc-99, Cl-36 and Ni-59 Pre-column Composite Concentrations. ....	66
Table 12-1. Lessons Learned – Successes. ....	71
Table 12-2. Lessons Learned – Potential Improvements.....	72

## LIST OF TERMS

## Abbreviations and Acronyms (3 pages)

AEA	alpha energy analysis
AES	atomic emission spectroscopy
ALARA	as low as reasonably achievable
AN-101	Tank 241-AN-101
AN-106	Tank 241-AN-106
AP-105	Tank 241-AP-105
AP-106	Tank 241-AP-106
AP-107	Tank 241-AP-107
AY-101	Tank 241-AY-101
BBI	Best Basis Inventory
BFB	4-bromofluorobenzene
BV	bed volume
CCV	continuing calibration verification

## RPP-RPT-59874

**Abbreviations and Acronyms (3 pages)**

CL	centrifuged liquid
CMBST	RCRA technology code for combustion
Comp	composite
CVAA	cold vapor atomic absorption
DI	deionized
d <sub>max</sub>	maximum diameter
d <sub>min</sub>	minimum diameter
DOT	Department of Transportation
ECD	electron capture detector
ESD	energy dispersive X-ray spectrometer
EQL	estimated quantitation limit
FWF	Federal Waste Disposal Facility
GC/MS	gas chromatography/mass spectrometry
GEA	gamma energy analysis
GL	grab liquid
GPC	gas proportional counter
HASQARD	Hanford Analytical Services Quality Assurance Requirements Documents
HDPE	high-density polyethylene
IC	ion chromatography
IC/MS	ion chromatography/mass spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
ICP	inductively coupled plasma spectroscopy
ICSAB	interference check standard
ICV	initial calibration verification
LAWPS	Low Activity Waste Pretreatment System
LDR	land disposal restrictions
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
Liq	liquid
LLS	low level standard
LSC	liquid scintillation counting
MDA	minimum detectable activity
MDL	method detection limit
MQC	middle quality control
MS	matrix spike
MSD	matrix spike duplicate
N/A	not available
NR	not needed for regulatory requirements
NR	not needed for regulatory requirements
NRC	Nuclear Regulatory Commission
NWW	nonwastewater

## RPP-RPT-59874

**Abbreviations and Acronyms (3 pages)**

OmniLIMS	laboratory information management system
PCB	polychlorinated biphenyl
PDS	post-digestion spike
PFNW	Perma-Fix Northwest Richland, Inc.
PLM	polarized light microscopy
PNNL	Pacific Northwest National Laboratory
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	The Resource Conservation and Recovery Act
RDL	required detection limit
RML	Radioactive Material License
RPD	relative percent difference
RSD	relative standard deviation
SEM	scanning electron microscopy
sRF	spherical resorcinol-formaldehyde
StDev	standard deviation
SVOA	semivolatile organic analysis
TC	total carbon
TCLP	toxicity characteristic leaching procedure
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
TSCA	Toxic Substances Control Act of 1976
TWINS	Tank Waste Information System
UHC	underlying hazardous constituent
UTS	universal treatment standards
VOA	volatile organic analysis
VOC	volatile organic compounds
WAC	Washington Administrative Code
WCS	Waste Control Specialists LLC
WDOH	Washington State Department of Health
WIR	Waste Incidental to Reprocessing
WHL	Wastren Advantage, Inc. Hanford Laboratory
WRPS	Washington River Protection Solutions LLC

## RPP-RPT-59874

**Units**

$^{\circ}\text{C}$	degrees Celsius
$\text{Ci}$	curies
$\text{cm}$	centimeter
$^{\circ}\text{F}$	degrees Fahrenheit
$\text{g}$	gram
$\text{gal}$	gallon
$\text{hr}$	hour
$\text{in.}$	inch
$\text{kg}$	kilogram
$\text{L}$	liter
$\mu\text{Ci}$	microcuries
$\mu\text{g}$	microgram
$\mu\text{m}$	micrometer
$\text{mg}$	milligram
$\text{mEq}$	milliequivalent
$\text{mm}$	millimeter
$\text{mmol}$	millimole
$\text{mRem}$	millirem
$\text{min}$	minute
$\text{M}$	molar
$\text{mL}$	milliliters
$\text{mol}$	moles
$\text{n}$	sample size
$\text{nCi}$	nanocuries
$\text{ppm}$	parts per million
$\text{ppmv}$	parts per million by volume
$\text{psi}$	pounds per square inch

## 1 INTRODUCTION

This test report describes the 222-S Laboratory Washington River Protection Solutions LLC (WRPS) and WAI Hanford Laboratory (WHL) tasks performed in support of the Radioactive Test Bed Initiative. LAB-PLN-16-00003, Rev. 0, “Test Plan for the Preparation of a Supernate Archive Composite from Hanford Tanks 241-AN-101, 241-AN-106, 241-AP-105, 241-AP-, 241-AP-107, and 241-AY-101 for Shipment to Perma Fix,” was written and revised to direct the work described in this report. Similar testing has been safely performed using actual tank wastes from both Hanford and Savannah River Site (PNWD-3697, PNWD-3751, PNWD-3752, PNNL-18007, SRNL-STI-2009-00594<sup>1</sup>). Test methods and protocols described herein are based on those described in these prior tests. Project direction and additional information regarding the Radioactive Test Bed Program required to write and execute the test plan was provided by the WRPS One System organization.

The objectives for the 222-S Laboratory included:

1. Composite 11 L of archived tank waste (Tanks 241-AN-101 [AN-101], 241-AN-106 [AN-106], 241-AP-105 [AP-105], 241-AP-106 [AP-106], 241-AP-107 [AP-107], and 241-AY-101 [AY-101])
2. Ensure molarity of the composite waste is similar to the Low Activity Waste Pretreatment System (LAWPS) design (4.0 to 6.0 M Na)
3. Treat the composite waste through filtration for solids removal and spherical resorcinol-formaldehyde resin (sRF) ion exchange for cesium depletion
4. Develop sRF Cs-137 breakthrough curves
5. Provide sufficient characterization data to ensure waste acceptability requirements are met for Perma-Fix Northwest Richland, Inc. (PFNW), Washington Administrative Code (WAC), and Waste Incidental to Reprocessing (WIR)
6. Package the treated waste for shipment to PFNW

The decontamination process is mainly driven by ALARA (as low as reasonably achievable) concerns and shipping requirements. Waste characterization was performed at several points throughout the pretreatment process to provide data relevant to the regulatory framework, shipping requirements, and the test bed process. Once characterized and shipped, WRPS was tasked with producing a final report covering the WRPS and WHL activities in this plan. This is that final report.

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<sup>1</sup> PNWD-3697, “Spherical Resorcinol-Formaldehyde Resin Testing for <sup>137</sup>Cs Removal from Simulated and Actual Hanford Waste Tank 241-AP-101 Diluted Feed (Envelope A) Using Small Column Ion Exchange.”

PNWD-3751, “Small Column Ion Exchange Testing of Spherical Resorcinol-Formaldehyde Resin for <sup>137</sup>Cs Removal from Pre-Treated Hanford Tank 241-AN-102 Waste (Envelope C).”

PNWD-3752, “Spherical Resorcinol-Formaldehyde Resin Analysis Following Actual Hanford Tank Waste Processing.”

PNNL-18007, “Laboratory Demonstration of the Pretreatment Process with Caustic and Oxidative Leaching Using Actual Hanford Tank Waste.”

SRNL-STI-2009-00594, “Real Waste Testing of Spherical Resorcinol-Formaldehyde Ion Exchange Resin.”

## RPP-RPT-59874

This test report documents the tasks performed to prepare approximately 11 L of aqueous, cesium-depleted Hanford tank waste from the 222-S Laboratory hot cell archive to meet the requirements for transportation to the PFNW and the necessary characterization as outlined by the regulatory framework (Appendix 5.2-1: Waste Acceptance Plan Revision 9, and “Federal Waste Disposal Facility (FWF) Generator Handbook.”) It is important to note that operations in the 222-S Laboratory are separated by contract to WRPS and to WHL, which defined the resources required to perform each of the tasks described below.

## 2 COMPOSITE

### 2.1 Archived Material

A 13,359 g composite (S16R000307) was generated from archived supernate sample material originating from tanks AN-101, AN-106, AP-105, AP-106, AP-107, and AY-101. (See APPENDIX A.) These supernate samples were originally retrieved between 2010 – 2014. Since that time, they have been stored in the hot cell archive located at the 222-S Laboratory. Upon retrieval from storage, the supernate samples were weighed prior to opening to determine the effect of evaporation over time. Very little weight loss was observed. (See net weight difference in APPENDIX A.) With the exception of Jar Number 20657, archive samples were composed of clear, yellow liquids with few residual solids on the bottom of the jar. (Jar Number 20657 was dark grey.) Based on the lack of weight loss over the storage time period and the hydrated state of the supernate samples, the composite integrity was deemed to be representative of current tank waste.

### 2.2 Composite Preparation and Filtration

Prior to preparing the composite, the clean, 13-L carboy was rinsed multiple times with 300 mL of 1 M NaOH to leach any potential contaminants associated with the plastic. Next, the contents of each archive jar identified in Appendix A was transferred to the carboy. No effort was made to re-hydrate any solid residue, however, effort was made to transfer the entire contents. The liquid contents of one jar was occasionally transferred from jar to jar to mobilize residual solids for addition to the composite.

The composite in the carboy was manually shaken for approximately 8 min, then recirculated using a Masterflex<sup>®</sup><sup>2</sup> Model 77200-62 Easy-Load peristaltic pump with Masterflex 6429-24 Tygon LFL tubing (7/16 in. outer diameter, 1/4 in. internal diameter) for a total of 60 min to ensure homogeneity. After recirculation, the composite was allowed to settle for approximately 24 hr. A significant amount (about 10 to 15 mm) of fine solids settled to the bottom of the carboy.

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<sup>2</sup> Masterflex is a registered trademark of the Cole-Parmer Instrument Company, Vernon Hills, Illinois.,

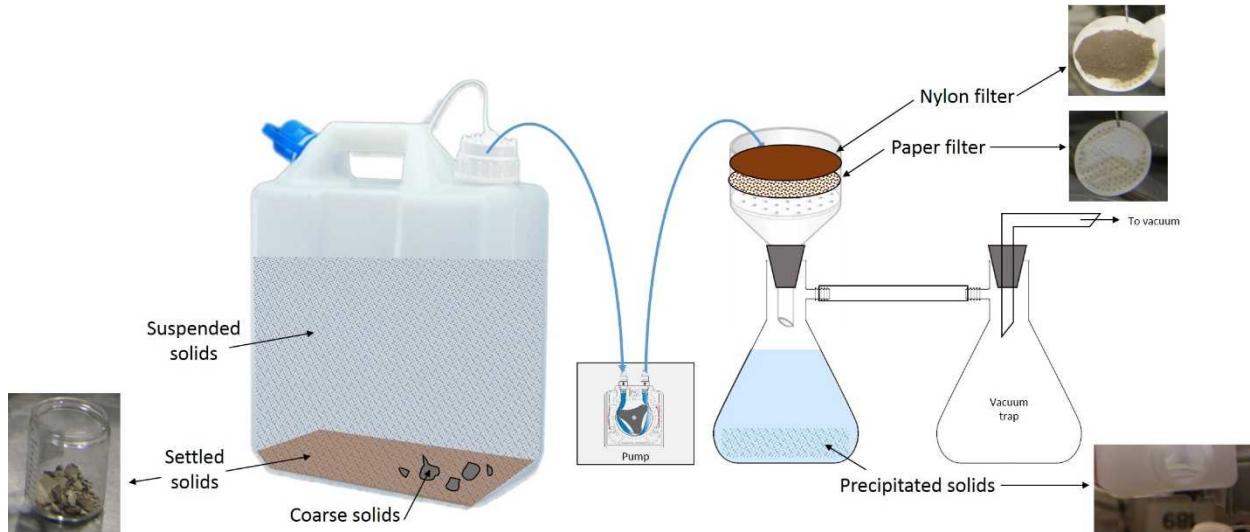
## RPP-RPT-59874

In order to ensure the volume of sample was adequate, the sodium molarity was greater than 4.0 M Na, and soluble solids that had precipitated during archive storage were redissolved, 600 mL (918 g) of 19.4 M NaOH was added to the composite. The composite was again agitated, circulated and allowed to settle as before. The settled solids were reduced considerably in volume, occupying a layer approximately 5 mm thick. The average sodium molarity of the aqueous composite was measured at 4.7 M.

Prior to filtering the composite, twelve 1-L high density polyethylene (HDPE) bottles (S16R000308 through S16R000318, S16R000342) were tared, then rinsed with approximately 100 mL of 1 M NaOH. Next, aqueous composite containing suspended solids was transferred to a filter funnel stand using the peristaltic pump with an input line floating at the top of the composite to minimize disturbance of the settled solids. The composite was filtered through a Millipore nylon filter (0.45- $\mu$ m pore size) with a paper filter backing inside of a Buchner funnel. A total of four nylon/paper filter sets were used. The first three filter sets were used to filter the majority of the aqueous composite, removing 2.65 g of suspended solids. The last filter set removed 18.25 g of residual suspended solids and the settled solids. Remaining in the carboy after filtration was 0.3 g of coarse solids. (See Figure 2-1.)

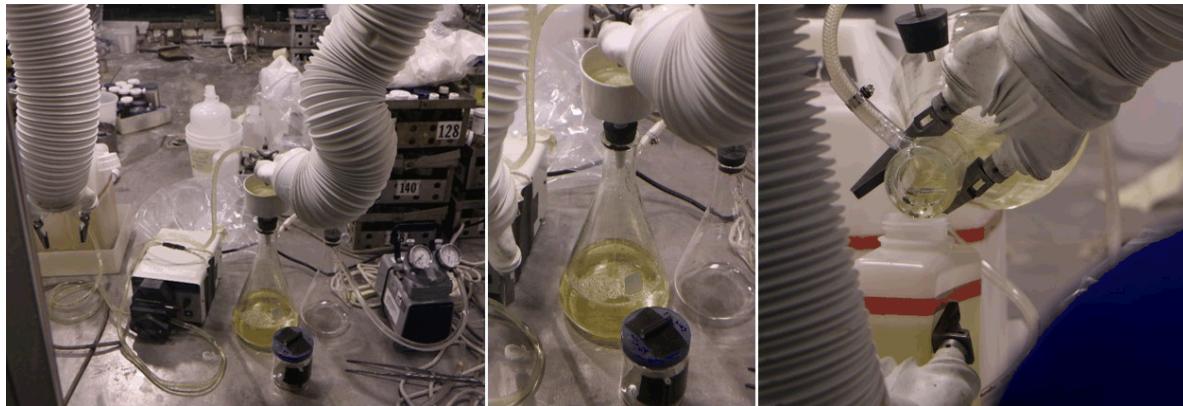
The filtered composite was initially collected into a 2-L flask and was intermittently transferred into twelve NaOH-rinsed 1-L HDPE bottles (S16R000308 through S16R000318 and S16R000342). (See Figure 2-2.) After the filtration process was completed, approximately 12.5 mL (15 g) of filtered composite was sub-sampled from each of the twelve collection bottles and combined to make a 150 mL (180 g) pre-column composite sample (S16R000348). The remaining filtrate was processed through sRF ion exchange resin columns.

During the ion exchange process, one 2-mL blank, twenty-two 2-mL lead column samples, and thirteen 2-mL lag column samples were collected.



**Figure 2-1. Composite Filtration Apparatus.**

## RPP-RPT-59874



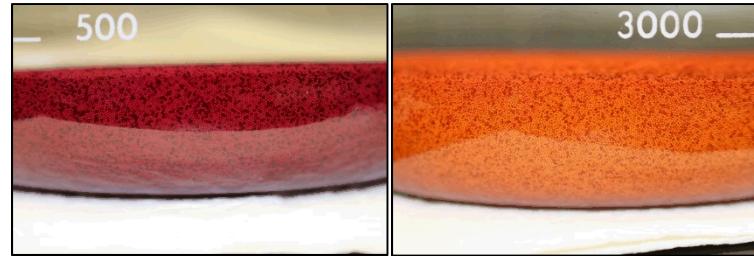
**Figure 2-2. Composite Filtration.**

### 3 ION EXCHANGE PROCEDURES

#### 3.1 Ion Exchange Resin Pretreatment and Column Preparation

Microbeads<sup>TM3</sup> sRF resin (Lot Number: 1F-370-1392; canister serial number: S532312-1-017; production year: 2011; Chemical Information Tracking System barcode: SL029644) was received from Pacific Northwest National Laboratory (PNNL) in the hydrogen form.

Approximately 500 mL of H<sup>+</sup> form resin was regenerated as per test plan LAB-PLN-16-00003, Rev. 0. This regeneration resulted in cycling the resin through the following forms: H<sup>+</sup> (original form), Na<sup>+</sup>, H<sup>+</sup>, and Na<sup>+</sup>. (See Figure 3-1.) Resin subsamples were collected during the final H<sup>+</sup> and Na<sup>+</sup> conversion.

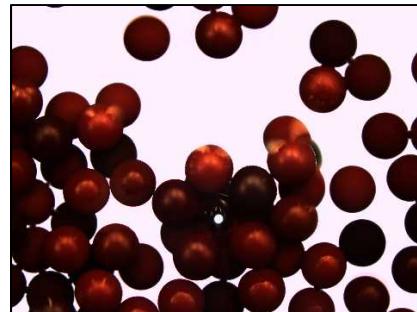


**Figure 3-1. H<sup>+</sup>-Form (left) and Na<sup>+</sup>-Form (right) of sRF Resin Used.**

Polarized light microscopy (PLM) analyses were performed on the Na<sup>+</sup> and H<sup>+</sup> resin subsamples at the 222-S Laboratory using procedure ATS-LT-519-107, “222-S Laboratory Polarized Light Microscopy.” The uniformity of the H<sup>+</sup>-form sRF resin beads is shown in Figure 3-2, which were measured to have a mean diameter of 398  $\mu\text{m}$  ( $n = 23$ ,  $d_{\min} = 385 \mu\text{m}$ ,  $d_{\max} = 407 \mu\text{m}$ , StDev = 5.0). The Na<sup>+</sup>-form sRF resin beads were measured to have a mean diameter of 441  $\mu\text{m}$ . ( $n = 8$ ,  $d_{\min} = 435 \mu\text{m}$ ,  $d_{\max} = 448 \mu\text{m}$ , StDev = 5.3).

<sup>3</sup> Microbeads is a trademark of Microbeads AS, Skedsmokorset, Norway.

## RPP-RPT-59874



**Figure 3-2. Polarized Light Microscopy Image of sRF Resin in the H<sup>+</sup>-Form.**

Three sets of Lead/Lag columns were prepared with sRF resin in the Na<sup>+</sup> form, as per test plan LAB-PLN-16-00003, Rev. 1. To each of the Lead 1, Lag 1, Lead 2, and Lag 2 columns, approximately 40 mL of sRF resin in the Na<sup>+</sup> form was added. This allowed for approximately 20 mL of fluid to set above the resin bed. These columns were then regenerated to the H<sup>+</sup> form to allow for more robust transport conditions. (When converted to the H<sup>+</sup> form, the resin volume contracts to 30 mL.) To each of the Lead 3 and Lag 3 columns, approximately 60 mL of Na<sup>+</sup> form sRF resin was added and contained approximately 10 mL of fluid above the resin bed. The Lead 3 and Lag 3 columns were not regenerated in the H<sup>+</sup> form for transport due to the time requirement of Na<sup>+</sup> form regeneration inside the hot cell.

### 3.2 Ion Exchange Column and Manifold System

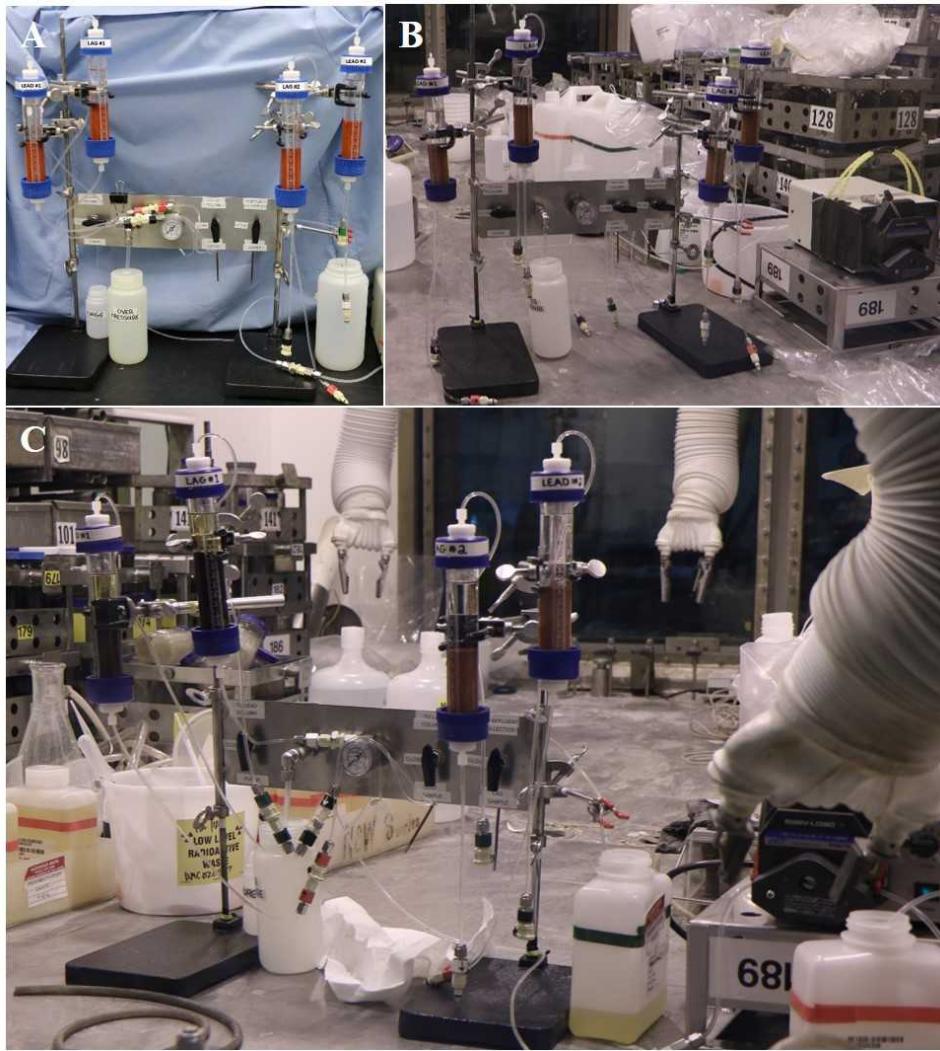
The ion exchange column and manifold system included a Masterflex Model 77200-60 Easy-Load peristaltic pump with Masterflex L/S 14 Tygon Chemical tubing (1/8 in., outer diameter, 1/16 in. internal diameter); three Swagelok<sup>®4</sup> stainless steel 3-way ball valves; one Swagelok 10 psi overpressure release valve; one 15 psi maximum pressure gauge; valved CPC<sup>™5</sup> quick-disconnect couplings (1/8 in. flow); and two standard Chromaflex<sup>®6</sup> chromatography columns (2.5 cm inner diameter by 15 cm tall, 74 mL). Photos of the ion exchange column and manifold system are shown in Figure 3-3. (Figure 3-3A shows the manifold and columns on the cold bench top; Figure 3-3B shows the manifold and columns inside the hot cell before processing; and Figure 3-3C shows the manifold connected with Lead 1/Lag 1 columns during processing in the hot cell.)

<sup>4</sup> Swagelok is a registered trademark of Swagelok Company, Solon, Ohio.

<sup>5</sup> CPC is a trademark of Colder Products Company, St. Paul, Minnesota.,

<sup>6</sup> Chromaflex is a registered trademark of Kimble Kontes LLC, Vineland, New Jersey.

## RPP-RPT-59874

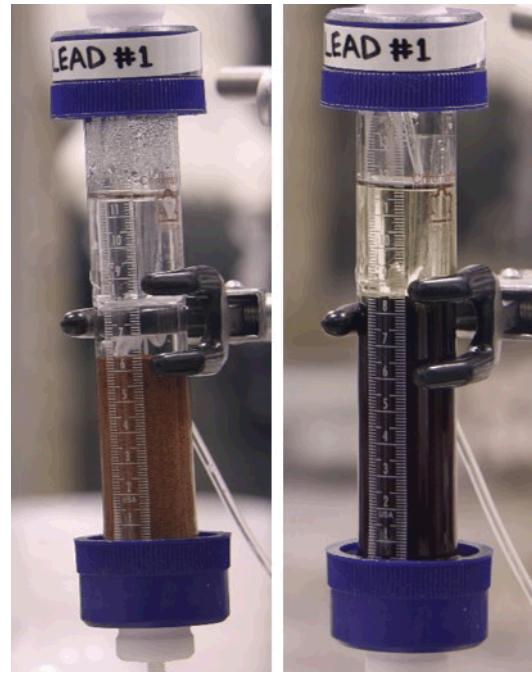


**Figure 3-3. Ion Exchange Column and Manifold System.**

### 3.3 Composite Processing and Sampling

The Lead 1 and Lag 1 columns, containing approximately 30 mL of  $H^+$  form sRF resin, were connected to the valve manifold. The void volume in the feed line was primed with deionized (DI) water. A flow rate of 2.26 mL/min was established through the purge output by gravimetric measurements. The feed line was then transferred to the 0.5 M NaOH reagent bottle. Without adjusting the pump's flow rate, 345 mL (8+ bed volume [BV]) of NaOH was sequentially processed through the entire system to remove air from the system and to convert the resin from the  $H^+$  to  $Na^+$  form. (The volume of the resin in the column when converted to the  $Na^+$  form was approximately 40 mL.) The flow rate through the column and manifold system was measured to be 2.15 mL/min. The processing of NaOH through the ion exchange columns resulted in the resin changing color (from orange-red to dark maroon) and swelling (from approximately 32 mL to 42 mL). (See Figure 3-4.) An 11-mL process blank sample (S16R000359) was collected in a 20-mL glass vial at the effluent output.

## RPP-RPT-59874



**Figure 3-4. sRF Resin Expansion From the H<sup>+</sup> to Na<sup>+</sup> Form.**

Prior to processing the filtered composite through the ion exchange columns, twelve 1-L HDPE bottles (S16R000319 through S16R000329, S16R000343) were tared and then rinsed with approximately 100 mL of 1 M NaOH to remove residual organics. On September 19, 2016, the feed line was transferred into the first of twelve filtered composite bottles (S16R000308), and the manifold's effluent output line was placed in the first of twelve effluent collection bottles (S16R000319). For the next 32 hr (with a second shift to allow continuous operation), approximately 5 L of the filtered composite was processed from the sequential filtered composite bottles through the Lead 1/Lag 1 columns at an average rate of 2.2 mL/min (3.3 BV/hr) and collected into sequential effluent collection bottles. At regular intervals, the effluent from the lead column was tapped, and a sample was collected for gamma energy analysis (GEA). This was done to monitor for cesium breakthrough of the column.

The active processing of the filtered composite was temporarily halted to exchange the ion exchange columns with the Lead 3 and Lag 3 columns. However, to maximize the amount of resin-treated effluent collected, air was introduced into the system until the fluid level was just above the Lead 1 column resin bed. The Lead 1 column was removed from the valve manifold and set aside. The Lag 1 column was connected to the manifold in the "lead column position," with the overpressure release valve, pressure gauge, and "lag column position" being bypassed. By introducing air to the system, the fluid level was reduced to where it no longer remained above the Lag 1 column's resin bed. The Lag 1 column was removed from the valve manifold and set aside. The evacuated volume from the Lead 1 and Lag 1 columns was added to the respective effluent collection bottle.

The Lead 3 and Lag 3 columns, containing approximately 60 mL of Na<sup>+</sup> form sRF resin, were connected to the valve manifold. (During this process, the overpressure collection bottle was

## RPP-RPT-59874

removed to allow for improved access to column connections.) The feed line was thoroughly rinsed with DI water, placed into the 0.5 M NaOH reagent bottle, and primed. Once all air was visibly removed from the system, 90 mL of 0.5 M NaOH was processed through the Lead 3 and Lag 3 column and disposed of. A flow rate of 4.35 mL/min (4.35 BV/hr) of 0.5 M NaOH was established through the purge output by gravimetric measurements.

The feed line was then transferred to the respective filtered composite bottle. Without adjusting the pump's flow rate, filtered composite was processed through the system at an average measured flow rate of 4.2 mL/min (4.2 BV/hr). The appearance of a yellow color in the fluid above the Lag 3 resin bed became apparent 9 min after the start of processing. A yellow color in the effluent began 16 min after the start of processing. The initial 85 mL of effluent was collected and discarded, as it theoretically consisted of 0.5 M NaOH.

In the process of positioning the effluent collection bottle (S16R000323), the pump was stopped. The columns sat undisturbed for 1 hr until it was realized that the pump was not restarted. At this point, the pump's flow rate was increased slightly to 4.35 mL/min. After 1 hr, it was noticed that yellow product was leaking out of the overpressure release valve and was collecting in a pool underneath the manifold. The pump was immediately stopped. The pool of liquid on the floor of the hot cell was absorbed and disposed of. The overpressure collection bottle was replaced. The flow rate was reduced back to 4.2 mL/min. The overpressure might have resulted from line plugging or from the presence of air in the lines and valves.

After 34 hr of (mostly) continuous pumping, the pump tubing collapsed inside of the pump head, and as a result, the flow stopped. This was immediately observed and corrected by moving a fresh section of the tubing into the pump head. Less than 3 minutes of time was lost performing this corrective action.

Over the course of the three days of column processing, the feed bottles accumulated a clear white precipitate. The feed tubing was positioned to keep the particulate from entering the column system. As mentioned above, the single overpressure event experienced during column processing may have resulted from line plugging due to this precipitate. A total of 63 g of precipitate was found in the 12 feed bottles. A sample of the precipitate was loaded out for PLM analysis. No significant precipitation was observed in the final product bottles.

The last of the filtered composite was processed by mid-day September 22, 2016. After all the filtered composite was processed through the sRF ion exchange resin, the post-column effluent was composited in a 13-L carboy (S16R000330) and recirculated. Twelve 1-L HDPE bottles (S16R000331 through S16R000341; S16R000413) were tared and then rinsed with approximately 100 mL of 0.05 M NaOH to remove residual organics. The filtered composite was then distributed amongst the 1-L bottles. (See Figure 3-5).

In total, 6 solid samples and 50 aqueous samples were loaded out of the hot cell and analyzed as shown in the sample breakdown diagrams. (See APPENDIX B).

## RPP-RPT-59874



**Figure 3-5. Final Product.**

## 4 PACKAGING AND SHIPPING

### 4.1 Packaging

The outsides of the 12 product bottles (S16R000331 through S16R000341; S16R000413) were thoroughly rinsed with DI water and allowed to dry before being loaded out. During the load out process, each bottle was individually dosed, bagged, wrapped in absorbent matting, and labeled with the appropriate reference number. Each product bottle was then placed inside of a plastic bag, forming a single layer inside of a 55 gal drum. (See Figure 4-1.)



**Figure 4-1. Product Packaging.**

### 4.2 Analytical Requirements

The list of constituents to be analyzed was determined by examination of the regulatory limits required. However, the number of constituents identified for analysis grew over time and their respective limits altered. Analyses of a number of inorganic constituents were obtained opportunistically, although they were not specifically requested.

## RPP-RPT-59874

The cesium-depleted waste product was assigned the characteristic waste codes and listed waste codes previously given to the parent tank waste archive material used to make the composite. Since the cesium-depleted waste does not meet the criteria for wastewater in 40 CFR 268.2, “Definitions Applicable in This Part,” as waste that contains <1% by weight total organic carbon (TOC) and <1% by weight total suspended solids, it is classified as a nonwastewater (NNW).

Information from the analytical data will be used to characterize and designate the treated cesium-depleted waste in accordance with WAC 173-303, “Dangerous Waste Regulations.” The data will be used to determine if the waste contains contaminants at concentrations at or above the dangerous waste threshold identified in the table, “Toxicity Characteristics List,” found in the WAC 173-303-090, “Dangerous Waste Characteristics.” If so, the applicable dangerous waste number will be applied.

As part of the designation to determine if the waste meets land disposal and treatment standard requirements, waste constituents’ concentrations will be compared to the table, “Treatment Standards for Hazardous Wastes,” found in 40 CFR 268.40, “Applicability of Treatment Standards.” Those constituents that exceed the concentration-based standards from the table, “Technology Codes and Descriptions of Technology-Based Standards,” specified in 40 CFR 268.42, “Treatment Standards Expressed as Specified Technologies,” will require additional treatment.

Prior to land disposal, all underlying hazardous constituents (UHCs) in the waste (defined in section (i) of 40 CFR 268.2) must meet Universal Treatment Standards (UTS) specified in 40 CFR 268.48, “Universal Treatment Standards.” The waste constituents’ concentrations will be reviewed against the UTS concentration-based values to ensure that the Resource Conservation and Recovery Act of 1976 (RCRA) requirements will be met at the treatment facility. The Nuclear Regulatory Commission waste disposal regulations are identified in 10 CFR 61, “Licensing Requirements for Land Disposal of Radioactive Waste.” Waste classification of low-level radioactive wastes for near surface disposal is determined using the requirements of 10 CFR 61.55, “Waste Classification.” Waste is divided into three classes, A through C. Class A low-level waste is the least radioactive and may be deposited near the surface for disposal, whereas Classes B and C have to be buried progressively deeper. The analytical data and radionuclide concentrations provided by the laboratory will be used to generate radionuclide calculations to determine the radiological classification of the waste for disposal requirements, as well as determining shipment requirements.

Once designation and radiological characterization are completed, a draft waste profile and a pre-note will be generated and sent to PFNW. The information in the profile is compared against the PFNW criteria, which is based on their RCRA permits and Washington State Department of Health (WDOH) Radiological License acceptance limits. PNFW will review the data and the draft profile to verify that the concentrations of the hazardous/radiological constituents are within their waste acceptance limits. Once PFNW determines that the waste profile meets their waste acceptance criteria and treatment capabilities, PFNW will sign and approve the final profile, indicating their concurrence for receipt and treatment of the waste.

The waste acceptance paperwork will then be provided to a Department of Transportation (DOT) Certified Mixed Waste Shipper for review. The shipper verifies that the radiological data is

**RPP-RPT-59874**

sufficient and performs verification of radiological calculations to determine the radiological classification of the waste. Packaging of the waste is determined using 49 CFR “Transportation,” 49 CFR 172.101, “Purpose and Use of Hazardous Materials Table,” and 49 CFR 173, “Shippers-General Requirements for Shipments and Packaging.” The shipper will determine the proper shipping name and complete the Hazardous Waste Manifest, as well as provide marking and labeling requirements per 49 CFR 172, “Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans,” Subpart D, “Marking,” and Subpart E, “Labeling,” respectively. A container inspection is performed prior to shipment of the container to the offsite treatment and disposal facility to ensure that DOT requirements are met.

## **5 RESULTS AND DISCUSSION**

Method detection limits (MDL) were based on the sample size, dilution, and other necessary correction factors. This limit was not necessarily the instrument detection limit. Constituents that were analyzed for but not detected have a less than (<) value equal to the MDL. Some analytical results are flagged with a data qualifier. Data qualifier flags are defined in Section 7, Quality Control.

### **5.1 Aqueous Analysis**

All liquids were clear and showed no evidence of a floating organic liquid phase. The pre-column and post-column aqueous liquid was analyzed for volatile organics, semi-volatile organics, polychlorinated biphenyl (PCB), metals, anions, small organic acids, free hydroxide, total inorganic carbon (TIC)/total organic carbon (TOC), density, weight % water, and radionuclides.

#### **5.1.1 Aqueous Pre-Column Composite Chemistry**

Constituents of interest specified in the test plan and those measured opportunistically are summarized in Table 5-1, Table 5-2, and Table 5-3. These tables contain the constituent’s primary result, duplicate result, NWW limit (if regulated), and WHL’s MDL. All results have been fully evaluated relative to Quality Control (QC). Results of those constituents deemed important will be discussed in order of their importance, including the total cesium, Cs-137, sodium, potassium, aluminum, and hydroxide.

RPP-RPT-59874

Table 5-1. Aqueous Pre-Column Organic Analytes. (2 pages)

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (µg/kg unless noted as "µg/L TCLP")	RCRA §268.48 <sup>†</sup> Nonwastewater Standard (µg/kg unless noted as "µg/L TCLP")	Result (µg/L)	Duplicate (µg/L)	Method Detection Limit (µg/L)
2,4,5-Trichlorophenol	D041	7,400	7,400	<686	<686	686
Hexachlorobutadiene	D033	5,600	5,600	<751 <sup>j</sup>	<751 <sup>j</sup>	751
Nitrobenzene	D036	14,000	14,000	<609	<609	609
Pyridine	D038	16,000	16,000	<731	<731	731
1,1-Dichloroethylene	D029	6,000	6,000	<6.87	<6.87 <sup>b</sup>	6.87
1,2-Dichloroethane	D028	6,000	6,000	<0.972	<0.972	0.972
Methyl ethyl ketone	D035	36,000	36,000	<18.3	<18.3 <sup>b</sup>	18.3
Benzene	D018	10,000	10,000	<0.652	<0.652	0.652
Carbon tetrachloride	D019	6,000	6,000	<1.92	<1.92	1.92
Tetrachloroethylene	D039	6,000	6,000	<2.33	<2.33 <sup>b</sup>	2.33
Trichloroethylene	D040	6,000	6,000	<1.51	<1.51 <sup>b</sup>	1.51
Vinyl chloride	D043	6,000	6,000	<1.94	<1.94	1.94
Methylene Chloride	F001-2	30,000	30,000	<1.58	<1.58	1.58
1,1,1-trichloroethane	F001-2	6,000	6,000	<1.73	<1.73	1.73
Chlorobenzene	F002	6,000	6,000	<1.28	<1.28	1.28
1,1,2-trichloro-1,2,2-trifluoroethane	F002	30,000	30,000	<1.58	<1.58	1.58
o-dichlorobenzene	F002	6,000	6,000	<1.82	<1.82	1.82
1,1,2-trichloroethane	F002	6,000	6,000	<0.92	<0.92 <sup>b</sup>	0.92
Xylene (Total)	F003	30,000	30,000	<4.11	<4.11	4.11
Acetone	F003	160,000	160,000	152 <sup>j</sup>	362	22.9
Ethyl Acetate	F003	33,000	33,000	<7.34	<7.34 <sup>b</sup>	7.34
Ethyl Benzene	F003	10,000	10,000	<1.34	<1.34	1.34
Ethyl Ether	F003	160,000	160,000	<1.69	<1.69	1.69
Methyl Isobutyl Ketone	F003	33,000	33,000	<18.1	<18.1 <sup>b</sup>	18.1
n-Butyl Alcohol	F003	2,600	2,600	456 <sup>j,b</sup>	1,050 <sup>b</sup>	46
Cyclohexanone	F003	750 µg/mL TCLP	750 µg/mL TCLP	<651	<651	651
2-Methylphenol	--	5,600	5,600	<631	<631	631
Cresols (m- & p-)	--	--	--	<562	<562	562

## RPP-RPT-59874

Table 5-1. Aqueous Pre-Column Organic Analytes. (2 pages)

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (µg/kg unless noted as "µg/L TCLP")	RCRA §268.48 <sup>T</sup> Nonwastewater Standard (µg/kg unless noted as "µg/L TCLP")	Result (µg/L)	Duplicate (µg/L)	Method Detection Limit (µg/L)
m-cresol	--	5,600	5,600	--	--	--
p-cresol	--	5,600	5,600	--	--	--
Total Cresols	F004	11,200	NA	<1,193	<1,193	1,193
2-Ethoxyethanol	--	NA	NA	<932	<932	932
2,4-Dinitrotoluene	--	140,000	140,000	<364	<364	364
Isobutyl alcohol	--	170,000	170,000	<1,080 <sup>J</sup>	<1,080 <sup>J</sup>	1,080
Methanol	--	NA	0.75 mg/L TCLP	--	--	--
2-Nitropropane	--	NA	NA	<10.6	<10.6	10.6
Trichlorofluoromethane	--	30,000	30,000	<1.83	<1.83	1.83
Chloroform	--	6,000	6,000	<1.66	<1.66	1.66
Hexachloroethane	--	30,000	30,000	<4.44	<4.44	4.44
Toluene	F005	10,000	10,000	4.09	2.89 <sup>J</sup>	2.09
Carbon disulfide	F005	4,800 µg/mL TCLP	4,800 µg/mL TCLP	<0.844	<0.844 <sup>b</sup>	0.844
All Aroclors	--	NA	10,000	<260.1	<260.1	260.1
AR1016	--	--	--	<94.5	<94.5	94.5
AR1221	--	--	--	<17.8	<17.8	17.8
AR1232	--	--	--	<20.5	<20.5	20.5
AR1242	--	--	--	<31.5	<31.5	31.5
AR1248	--	--	--	<17.8	<17.8	17.8
AR1254	--	--	--	<6.5	<6.5	6.5
AR1260	--	--	--	<71.5	<71.5	71.5

TCLP = toxicity characteristic leaching procedure

‡ = Treatment Standards for Hazardous Wastes. (2015). In P. Gallagher (Ed.), McCoy's RCRA Reference (2015 ed., p 857-859). Lakewood, Colorado: McCoy.

T = Universal Treatment Standards. (2015). In P. Gallagher (Ed.), McCoy's RCRA Reference (2015 ed., p 929-931). Lakewood, Colorado: McCoy.

a = The laboratory control sample (LCS) has a percent recovery outside the customer or analytical method specified range

b = The matrix spike (MS) or post-digestion spike (PDS) is outside the customer or analytical method defined range

J = The result is considered an estimate

## RPP-RPT-59874

**Table 5-2. Aqueous Pre-Column Composite Inorganic Analytes. (2 pages)**

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (mg/L TCLP)	RCRA §268.48 <sup>T</sup> Nonwastewater Standard (mg/L TCLP)	Result (mg/L unless noted otherwise)	Duplicate (mg/L unless noted otherwise)	Method Detection Limit (mg/L unless noted otherwise)
Mercury	D009	0.025	0.025	0.206 <sup>J</sup>	0.210 <sup>J</sup>	0.00044
Arsenic	D004	5	5	<1.5	<1.5	1.5
Barium	D005	21	21	0.355 <sup>J</sup>	0.385 <sup>J</sup>	0.100
Cadmium	D006	0.11	0.11	0.700 <sup>J</sup>	0.726 <sup>J</sup>	0.100
Chromium	D007	0.6	0.6	237	237	0.2
Lead	D008	0.75	0.75	2.48 <sup>J</sup>	2.37 <sup>J</sup>	1.30
Selenium	D010	5.7	5.7	<3.0	<3.0	3.0
Silver	D011	0.14	0.14	14.0 <sup>J</sup>	16.5 <sup>J</sup>	3.0
Antimony	UHC	--	1.15	<0.18	<0.18	0.18
Beryllium	UHC	--	1.22	0.344 <sup>J</sup>	0.354 <sup>J</sup>	0.100
Nickel	UHC	--	11	34.4	34.3	0.2
Thallium	UHC	--	0.2	<1.5	<1.5	1.5
Vanadium	UHC	--	1.6	0.201 <sup>J</sup>	0.233 <sup>J</sup>	0.100
Aluminum	NR	--	--	4,090	4,120	1.4
Sodium	NR	--	--	98,200	116,000	92
Potassium	NR	--	--	867	871	2.2
Cesium	--	--	--	2.08	2.18	0.0075
Uranium	--	--	--	21.7 <sup>J</sup>	23.5 <sup>J</sup>	2.9
Fluoride	--	--	--	1,850	1,880	5
Glycolate	--	--	--	164	167	2.5
Acetate	--	--	--	567	569	5
Formate	--	--	--	656	659	3.5
Chloride	--	--	--	1,250	1,250	2
Nitrite	--	--	--	29,500	29,700	45
Sulfate	--	--	--	4,170	4,160	4.5
Oxalate	--	--	--	952	944	4.5
Bromide	--	--	--	40.4 <sup>J</sup>	40.8 <sup>J</sup>	3
Nitrate	--	--	--	52,600	53,100	105

RPP-RPT-59874

**Table 5-2. Aqueous Pre-Column Composite Inorganic Analytes. (2 pages)**

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (mg/L TCLP)	RCRA §268.48 <sup>T</sup> Nonwastewater Standard (mg/L TCLP)	Result (mg/L unless noted otherwise)	Duplicate (mg/L unless noted otherwise)	Method Detection Limit (mg/L unless noted otherwise)
Phosphate	--	--	--	2,930	2,660	7
Thiosulfate	--	--	--	<3.0	<3.0	3.0
TIC	NR	--	--	5,110	5,130	35
TOC	NR	--	--	1,380	1,400	100
Hydroxide	--	--	--	24,900	24,500	553
Percent water (Wt. %)	--	--	--	77.2	75.5	0.01
Density (g/mL)	--	--	--	1.202	1.199	0.001

NR = Not needed for regulatory requirements

J = The result is considered an estimate

**Table 5-3. Aqueous Pre-Column Radiological Analytes. (2 pages)**

Constituent <sup>1,2</sup>	§61.55(a) Class A Limit ( $\mu$ Ci/mL)	Result	Result's Method Detection Limit	Duplicate	Duplicate's Method Detection Limit	Unit
(Long-Lived) Alpha-emitting transuramics with half-lives greater than 5 years	N/A	8.83E-04 <sup>J</sup>	4.60E-04	1.09E-03 <sup>J</sup>	4.60E-04	$\mu$ Ci/mL
H-3	40	1.36E-03	1.80E-05	1.53E-03	1.80E-05	$\mu$ Ci/mL
C-14	0.8	5.90E-04	7.76E-07	5.93E-04	7.76E-07	$\mu$ Ci/mL
Cl-36 <sup>3</sup>	N/A	N/A	N/A	N/A	N/A	N/A
Ni-59	N/A	N/A	N/A	N/A	N/A	N/A
Co-60	700	<1.69E-03	1.69E-03	<3.69E-03	3.69E-03	$\mu$ Ci/mL
Ni-63	3.5	1.94E-02	3.06E-06	1.95E-02	3.14E-06	$\mu$ Ci/mL
Sr-89/90	0.04	8.08E-02	7.86E-04	8.17E-02	7.79E-04	$\mu$ Ci/mL
Nb-94 <sup>4,5</sup>	N/A	<2.35E-03	2.35E-03	<3.41E-03	3.41E-03	$\mu$ Ci/mL
Tc-99	0.3	5.13E-02	3.40E-05	4.49E-02	3.03E-05	$\mu$ Ci/mL
Sn-126 <sup>4,5</sup>	N/A	N/A	N/A	N/A	N/A	N/A

## RPP-RPT-59874

**Table 5-3. Aqueous Pre-Column Radiological Analytes. (2 pages)**

Constituent <sup>1,2</sup>	§61.55(a) Class A Limit ( $\mu\text{Ci}/\text{mL}$ )	Result	Result's Method Detection Limit	Duplicate	Duplicate's Method Detection Limit	Unit
I-129	0.0083	1.11E-04	1.62E-05	1.22E-04	9.86E-06	$\mu\text{Ci}/\text{mL}$
Cs-137	1	49.3	0.024	49.9	0.0154	$\mu\text{Ci}/\text{mL}$
Ra-226 <sup>5</sup>	N/A	<0.405	0.405	<1.24	1.24	$\mu\text{Ci}/\text{mL}$
Th-229 <sup>4</sup>	N/A	<0.028 <sup>I</sup>	0.028	<0.028 <sup>I</sup>	0.028	$\mu\text{g}/\text{mL}$
U-233	N/A	<2.50E-04	2.50E-04	3.33E-04 <sup>J</sup>	2.50E-04	$\mu\text{g}/\text{mL}$
U-234 <sup>4</sup>	N/A	$\leq$ 0.0235 <sup>I</sup>	0.0135	0.0324 <sup>B,I,J</sup>	0.0135	$\mu\text{g}/\text{mL}$
U-235	N/A	0.635 <sup>B</sup>	0.0135	0.696 <sup>B</sup>	0.0135	$\mu\text{g}/\text{mL}$
U-238 <sup>4</sup>	N/A	26.3	0.104	28.1	0.104	$\mu\text{g}/\text{mL}$
Np-237 <sup>4</sup>	N/A	8.27E-03	5.60E-05	7.85E-03	5.60E-05	$\mu\text{g}/\text{mL}$
Pu-238	N/A	1.04E-04	5.07E-07	1.07E-04	4.86E-07	$\mu\text{Ci}/\text{mL}$
Pu-239/240	N/A	1.22E-03	5.53E-07	1.21E-03	4.32E-07	$\mu\text{Ci}/\text{mL}$
Pu-241	0.4165 <sup>F</sup>	9.67E-04	2.83E-05	8.97E-04	2.81E-05	$\mu\text{Ci}/\text{mL}$
Pu-242 <sup>7</sup>	N/A	1.24E-05 <sup>Y</sup>	2.67E-07	4.26E-06 <sup>Y</sup>	1.89E-07	$\mu\text{Ci}/\text{mL}$
Am-241 <sup>4</sup>	N/A	1.79E-05	2.81E-07	1.86E-05	1.97E-07	$\mu\text{Ci}/\text{mL}$
Am-243 <sup>4,6</sup>	N/A	N/A	N/A	N/A	N/A	N/A
Cm-242	2.38 <sup>F</sup>	<2.15E-07 <sup>J</sup>	1.05E-07	<1.14E-07	1.14E-07	$\mu\text{Ci}/\text{mL}$
Cm-243/244 <sup>4</sup>	N/A	<4.30E-07 <sup>J</sup>	1.94E-07	2.43E-07 <sup>J</sup>	1.16E-07	$\mu\text{Ci}/\text{mL}$

1) U-233, U-235, and all Pu isotopes are identified for reporting requirements due to their classification as “special nuclear material” as defined by the US Nuclear Regulatory Commission (NRC) and called out in the Waste Control Specialists, LLC (WCS) waste acceptance criteria.

2) All radionuclides listed require reporting of the presence and activity concentrations for acceptance to the WCS facility or for the WIR application.

3) Cl-36 supports the RML R04100 performance assessment updates as denoted in the WCS waste acceptance criteria.

4) Radionuclides specific only to the WIR application and not the WCS waste acceptance criteria.

5) These radionuclides have not been detected by sample analysis. Quantification in the tank waste is based on process knowledge.

6) Used as tracer for Am-241 and Cm isotopes. Not an expected isotope from waste processing history. If necessary, run the Am separation twice, one with Am-243 tracer, one without the Am-243 tracer. Use the indigenous Am-241 as tracer for Am-243.

7) Results contain interferences of the Pu-239/240 peak scattering into the Pu-242 region of interest. The results should be considered as conservative and not to be used for reliable quantification of the Pu-242.

N/A = Not available

F = Limit converted from nCi/g to  $\mu\text{Ci}/\text{mL}$  using density = 1.19 g/mL

B = Preparation blank/method blank exceeded the criteria specified in the analytical method or project- specific work instruction

I = Result was determined via indirect calibration

J = Result is considered an estimate

Y = Data questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations

## RPP-RPT-59874

From the analysis of the aqueous pre-column composite, cesium's concentration was measured at 1.60E-05 M by ion chromatography/mass spectrometry (ICP/MS), and Cs-137 was measured at 4.18E-6 M by GEA, resulting in a cesium:Cs-137 ratio of 3.83. In theory, this ratio should be similar to what was calculated using Best Basis Inventory (BBI) values from AP-107 and AY-101, which had an average of 2.55. (The 2.55 value agrees with the cesium:Cs-137 ratio of 2.59 previously determined in Tank 341-AN-102 [PNWD-3229, "Chemical Analysis and Physical Property Testing of 241-AN-102 Tank Waste—Supernatant and Centrifuged Solids"]). The sodium concentration fell within the target range of 4 to 6 M, at 4.66 M. The potassium, aluminum, and hydroxide concentrations were measured at 2.23E-2 M, 1.52E-1 M, and 1.45 M, respectively.

### 5.1.2 Aqueous Column Performance Chemistry

A total of 22 lead column and 13 lag column performance samples were taken during processing. Of these, only one sample, S16R000361, suggested the presence of cesium. This sample was taken after the Lag 1 column, and was analyzed to contain 0.0116  $\mu\text{Ci}/\text{mL}$  of Cs-137. Since the preceding and following lead column performance samples were measured at background levels for Cs-137, it was concluded that the detected Cs-137 was either the result of external contamination or instrumentation error. For precautionary measures, the contents of the product bottle (S16R000320) that contained this potential contamination was re-processed through the resin columns.

### 5.1.3 Aqueous Post-Column Composite Chemistry

Constituents of interest specified in the test plan and those measured opportunistically are summarized in Table 5-4, Table 5-5, and Table 5-6. These tables contain the constituent's primary result, NWW limit (if regulated), and WHL's MDL. All results have been fully evaluated relative to QC. In addition to discussing those constituents deemed important (cesium, Cs-137, sodium, potassium, aluminum, and hydroxide), the plutonium isotopic constituents will also be discussed.

Cesium's concentration was measured at 1.76E-7 M by ICP/MS, and Cs-137 was measured at 1.73E-4  $\mu\text{Ci}/\text{mL}$  (1.46E-11 M) by GEA. By comparing these values to the pre-column composite concentrations, the cesium decontamination factor would be 98.89%, and the Cs-137 decontamination would be >99.99%. Although these decontamination factors might appear to be similar, the cesium:Cs-137 ratio was calculated to be 12,100. This ratio does not agree with the pre-column value of 3.83. Since it is implausible for isotopes to be selectively removed, error must be associated in one or both of these values. To determine which value(s) contain error, the measured dose rate of a 20 mL sample was compared to the anticipated dose rate using MicroShield<sup>7</sup> software. Assuming that the pre-column composite cesium:Cs-137 ratio of 3.83 is correct, and when applied to the measured post-column composite ICP/MS value, a total of 4.60E-8 M Cs-137 (0.55  $\mu\text{Ci}/\text{mL}$ ) would be anticipated in the sample. Next, the anticipated dose rates were calculated for a 20 mL sample using MicroShield software for the Cs-137 activity associated with the ICP/MS (11  $\mu\text{Ci}$ ) and GEA (0.0035  $\mu\text{Ci}$ ). The MicroShield dose rates (with

<sup>7</sup> MicroShield is a registered trademark of Grove Software, Inc., Lynchburg, Virginia.

## RPP-RPT-59874

buildup) for the associated ICP/MS and GEA measured values at 11.43 cm were projected to be 1.01 mRem/hr and 0.0003 mRem/hr, respectively. The dose rate measured at 11.43 cm for the 20 mL sample, S16R000396, was <0.5 mRem/hr. This would suggest that the measured post-column composite ICP/MS value is the source of the error.

The sodium concentration dropped slightly, but remained within the target range of 4-6 M, at 4.33 M, respectively. The potassium concentration decreased by 14%, with a final concentration of 1.91E-2 M. Aluminum and hydroxide concentrations dropped slightly, and were measured at 1.44E-1 M, and 1.39 M, respectively.

All plutonium isotopic constituents decreased in value when compared to the pre-column composite, however, the magnitudes of these values were not similar: 6% for Pu-238, 1% for Pu-239/240, 12% for Pu-241, and 49% for Pu-242. Of these values, Pu-242 contained a “Y” qualifier flag due to interferences of the Pu-239/240 peak scattering into the Pu-242 region of interest; therefore, the Pu-242 results should be considered as conservative and are not to be used for reliable quantification. The error associated with the method of analysis should also be considered. For example, variance is observed in the alpha energy analysis (AEA) results (Pu-238, 6%; Pu-239/240, 1%) and liquid scintillation counting (LSC) analysis result (Pu-241, 12%). From these factors, it cannot be definitively proven that plutonium was retained on the ion exchange resin.

No other constituents will be discussed at length, as their change in concentration was either determined as negligible ( $\leq 10\%$ ), was measured to be below estimated quantitation limit (EQL), or contained qualitative analysis flag(s) in their measured value. Results from these analyses can be used for guidance in anticipating TCLP values when the appropriate correction factors are used.

## RPP-RPT-59874

**Table 5-4. Aqueous Post-Column Organic Analytes. (2 pages)**

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (µg/kg unless noted as "µg/L TCLP")	RCRA §268.48 <sup>T</sup> Nonwastewater Standard (µg/kg unless noted as "µg/L TCLP")	Result (µg/L)	Method Detection Limit (µg/L)
2,4,5-Trichlorophenol	D041	7,400	7,400	<196	196
Hexachlorobutadiene	D033	5,600	5,600	<215 <sup>b,J</sup>	215
Nitrobenzene	D036	14,000	14,000	<174	174
Pyridine	D038	16,000	16,000	<209 <sup>a,b</sup>	209
1,1-Dichloroethylene	D029	6,000	6,000	<6.87	6.87
1,2-Dichloroethane	D028	6,000	6,000	<0.972	0.972
Methyl ethyl ketone	D035	36,000	36,000	<18.3 <sup>b</sup>	18.3
Benzene	D018	10,000	10,000	<0.652	0.652
Carbon tetrachloride	D019	6,000	6,000	<1.92	1.92
Tetrachloroethylene	D039	6,000	6,000	<2.33	2.33
Trichloroethylene	D040	6,000	6,000	<1.51	1.51
Vinyl chloride	D043	6,000	6,000	<1.94	1.94
Methylene Chloride	F001-2	30,000	30,000	<1.58	1.58
1,1,1-trichloroethane	F001-2	6,000	6,000	<1.73	1.73
Chlorobenzene	F002	6,000	6,000	<1.28	1.28
1,1,2-trichloro-1,2,2-trifluoroethane	F002	30,000	30,000	<1.58	1.58
o-dichlorobenzene	F002	6,000	6,000	<1.82	1.82
1,1,2-trichloroethane	F002	6,000	6,000	<0.92	0.92
Xylene (Total)	F003	30,000	30,000	<4.11	4.11
Acetone	F003	160,000	160,000	209 <sup>J</sup>	22.9
Ethyl Acetate	F003	33,000	33,000	<7.34	7.34
Ethyl Benzene	F003	10,000	10,000	<1.34	1.34
Ethyl Ether	F003	160,000	160,000	<1.69	1.69
Methyl Isobutyl Ketone	F003	33,000	33,000	<18.1 <sup>b</sup>	18.1
n-Butyl Alcohol	F003	2,600	2,600	1340 <sup>b</sup>	46
Cyclohexanone	F003	750 µg/mL TCLP	750 µg/mL TCLP	<186	186
2-Methylphenol	--	5,600	5,600	<180	180

## RPP-RPT-59874

**Table 5-4. Aqueous Post-Column Organic Analytes. (2 pages)**

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (µg/kg unless noted as "µg/L TCLP")	RCRA §268.48 <sup>T</sup> Nonwastewater Standard (µg/kg unless noted as "µg/L TCLP")	Result (µg/L)	Method Detection Limit (µg/L)
Cresols (m- & p-)	--	--	--	<161	161
m-cresol	--	5,600	5,600	--	--
p-cresol	--	5,600	5,600	--	--
Total Cresols	F004	11,200	NA	<341	341
2-Ethoxyethanol	--	NA	NA	<266 <sup>a</sup>	266
2,4-Dinitrotoluene	--	140,000	140,000	<104	104
Isobutyl alcohol	--	170,000	170,000	<308 <sup>b,J</sup>	308
Methanol	--	NA	0.75 mg/L TCLP	--	--
2-Nitropropane	--	NA	NA	<10.6	10.6
Trichlorofluoromethane	--	30,000	30,000	<1.83	1.83
Chloroform	--	6,000	6,000	<1.66	1.66
Hexachloroethane	D034	30,000	30,000	<4.44	4.44
Toluene	F005	10,000	10,000	10.7 <sup>B,J,b</sup>	2.09
Carbon disulfide	F005	4,800 µg/mL TCLP	4,800 µg/mL TCLP	<0.844	0.844
All Aroclors	--	NA	10,000	<260.1	260.1
AR1016	--	--	--	<94.5	94.5
AR1221	--	--	--	<17.8	17.8
AR1232	--	--	--	<20.5	20.5
AR1242	--	--	--	<31.5	31.5
AR1248	--	--	--	<17.8	17.8
AR1254	--	--	--	<6.5	6.5
AR1260	--	--	--	<71.5	71.5

<sup>‡</sup> = Treatment Standards for Hazardous Wastes. (2015). In P. Gallagher (Ed.), *McCoy's RCRA Reference* (2015 ed., p 857-859). Lakewood, Colorado: McCoy.

<sup>T</sup> = Universal Treatment Standards. (2015). In P. Gallagher (Ed.), *McCoy's RCRA Reference* (2015 ed., p 929-931). Lakewood, Colorado: McCoy.

<sup>a</sup> = The laboratory control sample (LCS) has a percent recovery outside the customer or analytical method specified range

<sup>b</sup> = The MS or PDS is outside the customer or analytical method defined range

<sup>B</sup> = Preparation blank/method blank exceeded the criteria specified in the analytical method or project-specific work instruction

<sup>J</sup> = Result is considered an estimate

## RPP-RPT-59874

Table 5-5. Aqueous Post-Column Inorganic Analytes. (2 pages)

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (mg/L TCLP)	RCRA §268.48 <sup>†</sup> Nonwastewater standard (mg/L TCLP)	Result (mg/L unless noted otherwise)	Method Detection Limit (mg/L unless noted otherwise)
Mercury	D009	0.025	0.025	0.118 <sup>J</sup>	0.00044
Arsenic	D004	5	5	<1.5	1.5
Barium	D005	21	21	0.335 <sup>J</sup>	0.1
Cadmium	D006	0.11	0.11	0.658 <sup>J</sup>	0.1
Chromium	D007	0.6	0.6	231	0.2
Lead	D008	0.75	0.75	<1.3	1.3
Selenium	D010	5.7	5.7	<3	3
Silver	D011	0.14	0.14	12.8	3
Antimony	UHC	--	1.15	<0.18	0.18
Beryllium	UHC	--	1.22	0.328 <sup>J</sup>	0.1
Nickel	UHC	--	11	34.6	0.2
Thallium	UHC	--	0.2	<1.5	1.5
Vanadium	UHC	--	1.6	0.171 <sup>J</sup>	0.1
Aluminum	NR	--	--	3890	14
Sodium	NR	--	--	99,500	92
Potassium	NR	--	--	745	2.2
Cesium	--	--	--	0.0234	0.0006
Uranium <sup>T</sup>	--	--	--	26.57	0.131
Fluoride	NR	--	--	1,970	5
Glycolate	NR	--	--	166	2.5
Acetate	NR	--	--	564	5
Formate	NR	--	--	653	3.5
Chloride	NR	--	--	1,240	2
Nitrite	NR	--	--	30,700	45
Sulfate	NR	--	--	4,190	4.5
Oxalate	NR	--	--	953	4.5

RPP-RPT-59874

**Table 5-5. Aqueous Post-Column Inorganic Analytes. (2 pages)**

Constituent	Waste Code	RCRA §268.40 <sup>‡</sup> Nonwastewater Concentration (mg/L TCLP)	RCRA §268.48 <sup>†</sup> Nonwastewater standard (mg/L TCLP)	Result (mg/L unless noted otherwise)	Method Detection Limit (mg/L unless noted otherwise)
Bromide	NR	--	--	41.2 <sup>J</sup>	3
Nitrate	NR	--	--	55,000	105
Phosphate	NR	--	--	2,910	7
Thiosulfate	NR	--	--	<3	3
TIC	NR	--	--	5,030	14
TOC	NR	--	--	1,330	40
Hydroxide	--	--	--	23,700	553
Percent water (Wt. %)	--	--	--	78	0.01
Density (g/mL)	--	--	--	1.192	0.001

<sup>‡</sup> = Treatment Standards for Hazardous Wastes. (2015). In P. Gallagher (Ed.), *McCoy's RCRA Reference* (2015 ed., p 857-859). Lakewood, Colorado: McCoy.

<sup>†</sup> = Summation of IC/MS U-233, U-234, U-235, and U-238 results since ICP analysis failed QC

<sup>J</sup> = Result is considered an estimate

RPP-RPT-59874

**Table 5-6. Aqueous Post-Column Radiological Analytes. (2 pages)**

Constituent <sup>1,2</sup>	§61.55(a) Class A Limit ( $\mu$ Ci/mL)	Result	Method Detection Limit	Unit
(Long-Lived) Alpha-emitting transuranics with half-lives greater than 5 years	N/A	2.54E-04	6.53E-07	$\mu$ Ci/mL
H-3	40	1.36E-03	1.67E-05	$\mu$ Ci/mL
C-14	0.80	5.76E-04	7.96E-07	$\mu$ Ci/mL
Cl-36 <sup>3</sup>	N/A	N/A	N/A	N/A
Ni-59	N/A	N/A	N/A	N/A
Co-60	700	9.52E-04	3.36E-05	$\mu$ Ci/mL
Ni-63	3.5	1.99E-02	3.53E-06	$\mu$ Ci/mL
Sr-89/90	0.042	7.4E-02	1.07E-05	$\mu$ Ci/mL
Nb-94 <sup>4,5</sup>	N/A	<7.51E-05	7.51E-05	$\mu$ Ci/mL
Tc-99	0.3	4.55E-02	3.19E-05	$\mu$ Ci/mL
Sn-126 <sup>4</sup>	N/A	2.12E-04	2.57E-05	$\mu$ Ci/mL
I-129	0.008	1.22E-04	1.45E-05	$\mu$ Ci/mL
Cs-137	1	1.73E-04	3.86E-05	$\mu$ Ci/mL
Ra-226 <sup>5</sup>	N/A	<1.46E-03	5.28E-04	$\mu$ Ci/mL
Th-229 <sup>4</sup>	N/A	<0.028 <sup>1</sup>	0.028	$\mu$ g/mL
U-233	N/A	<2.50E-04	2.50E-04	$\mu$ g/mL
U-234 <sup>4</sup>	N/A	$\leq$ 0.0176 <sup>1</sup>	0.0135	$\mu$ g/mL
U-235	N/A	0.653 <sup>B</sup>	0.0135	$\mu$ g/mL
U-238 <sup>4</sup>	N/A	25.9	0.104	$\mu$ g/mL
Np-237 <sup>4</sup>	N/A	7.67E-03	5.60E-05	$\mu$ g/mL
Pu-238	N/A	9.92E-05	5.46E-07	$\mu$ Ci/mL
Pu-239/240	N/A	1.20E-03	5.44E-07	$\mu$ Ci/mL
Pu-241	0.4165 <sup>F</sup>	8.21E-04	2.76E-05	$\mu$ Ci/mL
Pu-242 <sup>7</sup>	N/A	4.24E-06 <sup>Y</sup>	3.13E-07	$\mu$ Ci/mL

## RPP-RPT-59874

**Table 5-6. Aqueous Post-Column Radiological Analytes. (2 pages)**

Constituent <sup>1,2</sup>	§61.55(a) Class A Limit ( $\mu$ Ci/mL)	Result	Method Detection Limit	Unit
Am-241 <sup>4</sup>	N/A	1.68E-05	2.38E-07	$\mu$ Ci/mL
Am-243 <sup>4,6</sup>	N/A	N/A	N/A	N/A
Cm-242	2.38 <sup>F</sup>	2.15E-07 <sup>J</sup>	1.17E-07	$\mu$ Ci/mL
Cm-243/244 <sup>4</sup>	N/A	3.95E-07 <sup>J</sup>	1.19E-07	$\mu$ Ci/mL

1) U-233, U-235, and all Pu isotopes are identified for reporting requirements due to their classification as “special nuclear material” as defined by the US NRC and called out in the WCS waste acceptance criteria.

2) All radionuclide listed require reporting of the presence and activity concentrations for acceptance to the WCS facility or for the WIR application.

3) Cl-36 supports the RML R04100 performance assessment updates as denoted in the WCS waste acceptance criteria.

4) Radionuclides specific only to the WIR application and not the WCS waste acceptance criteria.

5) These radionuclides have not been detected by sample analysis. Quantification in the tank waste is based on process knowledge.

6) Used as tracer for Am-241 and Cm isotopes. Not an expected isotope from waste processing history. If necessary, run the Am separation twice, one with Am-243 tracer, one without the Am-243 tracer. Use the indigenous Am-241 as tracer for Am-243.

7) Results contain interferences of the Pu-239/240 peak scattering into the Pu-242 region of interest. The results should be considered as conservative and not to be used for reliable quantification of the Pu-242.

F = Limit converted from nCi/g to  $\mu$ Ci/mL using density = 1.19 g/mL

N/A = Not available

B = Preparation blank/method blank exceeded the criteria specified in the analytical method or project-specific work instruction

I = Result was determined via indirect calibration

J = Result is considered an estimate

Y = Data is questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations

## RPP-RPT-59874

### 5.2 Ion Exchange Resin Breakthrough

One objective not met in this scope of work was to develop cesium breakthrough curves for the sRF resin columns.

### 5.3 Solid Analysis

Four distinct types of solids were observed during the study. These were categorized as suspended, settled, coarse, and precipitated solids. The level of analysis performed on each was dependent on its relative importance to the project's primary objectives and its initial screening.

The suspended solids were classified as those which did not settle to the bottom of the composite's jerrican (flat-sided plastic container) prior to filtration. These solids were selectively collected on a 0.45 micron nylon filter with a Wattman 41 paper filter backing. Upon separating the nylon and paper filter, brown dots colored the paper filter, indicating that some of the solids had a particle size less than 0.45 micron. Scanning electron microscopy (SEM) analysis was performed on the suspended solids.

The settled solids were identified as those solids that settled on the bottom of the composite's jerrican prior to filtration. The maximum amount of liquid above the settled solids layer was siphoned off prior to solids collection. Enough residual liquid was left behind to slurry and siphon the solids for filtration and collection. This method resulted in the incorporation of some suspended solids. Chemical analysis was performed on the (primarily) settled solids to estimate their composition.

The coarse solids were those sparse pebbles remaining in the composite's jerrican after completion of the composite's filtration and jerrican rinsing using portions of the filtered liquid as rinsate. The coarse solids were presumed to include rust, exotic gibbsite, and/or natrophosphate from an unknown source. Further analysis was not pursued due to their low abundance (less than 2% of the initial composite solids) and unexplained presence in a liquid grab sample.

The precipitated solids formed in the filtered product collection bottles after composite filtration. These suspended solids were too small in particle size for PLM analysis, but were analyzed by SEM.

Further analyses of the settled and precipitated solids were pursued for the purpose of this study. Analytical results for the suspended, settled, and precipitated solids are expanded upon in the sections below.

## RPP-RPT-59874

### 5.3.1 Pre-Column Settled Solids Chemical Analysis

The filtered, settled solids were analyzed for metals, anions, small organic acids, free hydroxide, TIC/TOC, weight percent H<sub>2</sub>O, and select radionuclides. Inorganic and radiological results are shown in Table 5-7 and Table 5-8, respectively.

**Table 5-7. Inorganic Analysis of Pre-Column Settled Solids. (2 pages)**

Constituent	WHL Result (µg/g unless noted otherwise)	WHL Method Detection Limit (µg/g unless noted otherwise)
Mercury	106 <sup>b</sup>	0.22
Aluminum	70,400	25.3
Arsenic	<27.2	27.1
Barium	126	1.81
Bismuth	232	34.3
Cadmium	33.4	1.81
Calcium	3,580 <sup>b</sup>	217
Cerium	173 <sup>B,J</sup>	45.1
Cesium	4.36	0.0542
Chromium	389	3.61
Copper	59	3.61
Iron	10,500	36.1
Magnesium	874	16.3
Manganese	555	1.81
Phosphorous	21,000	23.5
Sulfur	864 <sup>B</sup>	50.6
Lead	474	23.5
Selenium	<54.3	54.2
Silicon	1,040 <sup>B,b</sup>	21.7
Silver	26.2 <sup>J</sup>	5.42
Antimony	<32.6	32.5
Strontium	66.4	3.61
Beryllium	4.73 <sup>J</sup>	1.81
Nickel	7,750	3.61
Thallium	<27.2	27.1
Vanadium	8.31 <sup>J</sup>	1.81
Sodium	183,000	166
Potassium	359 <sup>J</sup>	39.7
Uranium	15,300	52.4
Fluoride	12,100	28.7
Glycolate	<93.4	92
Acetate	305 <sup>J</sup>	161
Formate	501 <sup>J</sup>	241
Chloride	528 <sup>B</sup>	92
Nitrite	14,300 <sup>b</sup>	402
Sulfate	2,260 <sup>B</sup>	264
Oxalate	484 <sup>B,J</sup>	149
Bromide	<87.5	86.2

## RPP-RPT-59874

**Table 5-7. Inorganic Analysis of Pre-Column Settled Solids. (2 pages)**

Constituent	WHL Result (µg/g unless noted otherwise)	WHL Method Detection Limit (µg/g unless noted otherwise)
Nitrate	28,100	362
Phosphate	106,000	115
Thiosulfate	<152	149
TIC	9,750	59.8
TOC	1,600 <sup>c</sup>	171
% Water (Wt%)	29.7	0.01

b = The MS or PDS is outside the customer or analytical method defined range  
 B = Preparation blank/method blank exceeded the criteria specified in the analytical method or project-specific work instruction  
 c = RPD was greater than method defined range  
 J = Result is considered an estimate

**Table 5-8. Radiological Analysis of Pre-Column Settled Solids. (2 pages)**

Constituent	Result	Method Detection Limit	Unit
(Long-Lived) Alpha-emitting transuramics with half-lives greater than 5 years	1.68	0.00507	µCi/g
H-3	1.42E-04 <sup>J</sup>	7.83E-05	µCi/g
C-14	2.96E-04	4.07E-06	µCi/g
Cl-36 <sup>1</sup>	N/A	N/A	N/A
Co-60	0.0462	0.0256	µCi/g
Ni-59	N/A	N/A	N/A
Ni-63	36.3	0.00131	µCi/g
Sr-89/90	98.6	0.00922	µCi/g
Nb-94 <sup>2</sup>	<0.0713	0.0713	µCi/g
Tc-99	0.114	0.00153	µCi/g
Sn-126 <sup>2</sup>	<0.156	0.156	µCi/g
I-129	7.39E-05	1.46E-05	µCi/g
Cs-137	110	0.0556	µCi/g
Ra-226 <sup>2</sup>	<3.48	3.48	µCi/g
Th-229	<2.03 <sup>I</sup>	2.03	µg/g
U-233	7.38 <sup>J</sup>	0.903	µg/g
U-234	≤6.30 <sup>I</sup>	3.90	µg/g
U-235	463 <sup>B</sup>	3.90	µg/g
U-238	15,900 <sup>B</sup>	7.55	µg/g
Np-237	0.966 <sup>J</sup>	0.202	µg/g
Pu-238	0.037	2.26E-04	µCi/g
Pu-239/240	1.33	2.77E-04	µCi/g
Pu-241	1.13 <sup>c</sup>	9.87E-03	µCi/g
Pu-242 <sup>3</sup>	5.17E-03 <sup>Y,c</sup>	1.36E-04	µCi/g

## RPP-RPT-59874

**Table 5-8. Radiological Analysis of Pre-Column Settled Solids. (2 pages)**

Constituent	Result	Method Detection Limit	Unit
Am-241	0.415	1.8E-04	$\mu\text{Ci/g}$
Am-243 <sup>4</sup>	N/A	N/A	N/A
Cm-242	1.5E-04 <sup>J</sup>	1.08E-04	$\mu\text{Ci/g}$
Cm-243/244	1.09E-03	1.52E-04	$\mu\text{Ci/g}$

1) Cl-36 supports RML R04100 performance assessment updates as denoted in the WCS waste acceptance criteria.

2) These radionuclides have not been detected by sample analysis. Quantification in the tank waste is based on process knowledge.

3) Results contain interferences of the Pu-239/240 peak scattering into the Pu-242 region of interest. The results should be considered as conservative and not to be used for reliable quantification of the Pu-242.

4) Used as tracer for Am-241 and Cm isotopes. Not an expected isotope from waste processing history. If necessary, run the Am separation twice, one with Am-243 tracer, one without the Am-243 tracer. Use the indigenous Am-241 as tracer for Am-243.

N/A = Not available

B = Preparation blank/method blank exceeded the criteria specified in the analytical method or project-specific work instruction

c = The relative percent difference (RPD) between duplicate samples, laboratory control sample duplicates (LCSD) or matrix spike duplicates (MSD), was greater than the customer or analytical method defined range

I = Result was determined via indirect calibration

J = Result is considered an estimate

Y = Data questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations

In order to obtain more precise estimates of the concentration of the various phases present in the solid sample, the qualitative estimates of the phases present may be combined with the quantitative analyses performed by WHL. To accomplish this, measured constituents were converted into units of moles per 100 g solids. (See Table 5-9.)

**Table 5-9. Constituent Mass of Pre-Column Settled Solids. (2 pages)**

Constituent	Constituent Weight Percent (g/100 g solids)	Constituent Molecular Weight (g/mol)	Constituent Concentration (mol/100 g solid)	Digestion/Instrument
Al	7.04	27	2.61E-01	Acid/ICP
Ba	0.01	137.3	9.18E-05	Acid/ICP
Bi	0.02	209	1.11E-04	Acid/ICP
Pb	0.05	207.2	2.29E-04	Acid/ICP
Si	0.10	28.1	3.70E-03	Acid/ICP
Ni	0.78	58.7	1.32E-02	Acid/ICP
U-235	0.05	235	1.97E-04	Acid/ICP
U-238	1.59	238	6.68E-03	Acid/ICP
Ca	0.36	40.1	8.93E-03	Acid/ICP
Ce	0.02	140.1	1.23E-04	Acid/ICP
Cr	0.04	52	7.48E-04	Acid/ICP

## RPP-RPT-59874

**Table 5-9. Constituent Mass of Pre-Column Settled Solids. (2 pages)**

Constituent	Constituent Weight Percent (g/100 g solids)	Constituent Molecular Weight (g/mol)	Constituent Concentration (mol/100 g solid)	Digestion/Instrument
Fe	1.05	55.8	1.88E-02	Acid/ICP
Mg	0.09	24.3	3.60E-03	Acid/ICP
Mn	0.06	54.9	1.01E-03	Acid/ICP
K	0.04	39.1	9.18E-04	Acid/ICP
Na	18.30	23	7.96E-01	Acid/ICP
S	0.09	32.1	2.69E-03	Acid/ICP
P	2.10	31	6.77E-02	Acid/ICP
Acetate	0.03	59	5.17E-04	Water/IC
Formate	0.05	45	1.11E-03	Water/IC
Ox	0.05	88	5.50E-04	Water/IC
Cl	0.05	35.5	1.49E-03	Water/IC
NO <sub>2</sub>	1.43	46	3.11E-02	Water/IC
NO <sub>3</sub>	2.81	62	4.53E-02	Water/IC
PO <sub>4</sub>	10.60	95	1.12E-01	Water/IC
SO <sub>4</sub>	0.23	96.1	2.35E-03	Water/IC
F	1.21	19	6.37E-02	Water/IC
TIC	0.98	12	8.13E-02	Acid Pursulfate
TOC	0.16	12	1.33E-02	Acid Pursulfate
OH*	1.19	17	7.02E-02	Calculated
H <sub>2</sub> O	0.00	wt%	1.65E+00	TGA

IC = ion chromatography

ICP = inductively coupled plasma spectroscopy

TGA = thermogravimetric analysis

\* = assuming that all the NO<sub>2</sub> and NO<sub>3</sub> is associated with the interstitial liquid and that the NO<sub>2</sub>:OH ratio is the same for the interstitial liquid and the settled solids

A 49% discrepancy in the moles of phosphorous measured by the ICP phosphorous (P) and IC phosphate (PO<sub>4</sub>) was observed, with the IC yielding the lower measurement. The ICP value for phosphorous (0.068 mol P/100 g solids) was translated to a phosphate value (0.068 mol PO<sub>4</sub>/100 g solids) and was used in calculations since it contributed to a more representative sample mass (95.2 g vs 88.0 g, with 100 g being ideal) and +/- charge balance (1.11 vs 1.37, with 1 being ideal).

The mass and charge balance calculations provided support for the predicted phases present in the settled solids. Each of the measured constituents was assigned to a representative phase, based on tank waste history. Quantification of the phases were calculated one at a time by delegating a quantity of a constituent to that phase. For each phase that consumed a fraction of a constituent, (e.g., kogarkoite consumed 0.006 mol Na), the remaining moles of that constituent were calculated by subtracting the relevant amount consumed (e.g., the remaining moles of

## RPP-RPT-59874

sodium would be:  $0.796 \text{ mol Na} - 0.006 \text{ mol Na} = 0.790 \text{ mol Na}$ . (See Table 5-10.) After all the constituents were consumed, individual phase substitutions were made to determine if the mass and/or charge balance could be optimized. (For example, gibbsite, dawsonite and aluminate were substituted as the primary Al phase in the calculations before gibbsite was determined to be the best representative.) The rules used to perform these calculations were performed in the below order:

1. All the Al is gibbsite,  $[\text{Al}(\text{OH})_3]$
2. All the Ba is barium hydroxide,  $[\text{Ba}(\text{OH})_2]$
3. All the Bi is bismuth oxide,  $[\text{Bi}_2\text{O}_3]$
4. All the Pb is lead oxide,  $[\text{PbO}_2]$
5. All the Si is cancrinite,  $[\text{Na}_3\text{CaAl}_3\text{Si}_3\text{O}_{12}(\text{CO}_3)]$
6. All the Ni is nickel hydroxide,  $[\text{Ni}(\text{OH})_2]$
7. All the U-235 and U-238 is sodium diuranate,  $[\text{Na}_2\text{U}_2\text{O}_7]$
8. All the Ca is calcium carbonate,  $[\text{Ca}(\text{CO}_3)]$
9. All the Ce is cerium hydroxide,  $[\text{Ce}(\text{OH})_3]$
10. All the Cr grimaldite,  $[\text{CrOOH}]$
11. All the Fe is hematite,  $[\text{Fe}_2\text{O}_3]$
12. All the Mg is magnesium hydroxide,  $[\text{Mg}(\text{OH})_2]$
13. All the Mn is manganese dioxide,  $[\text{MnO}_2]$
14. All the K is potassium nitrate,  $[\text{K}(\text{NO}_3)]$
15. All the  $\text{C}_2\text{H}_3\text{O}_2$  is sodium acetate,  $[\text{Na}(\text{C}_2\text{H}_3\text{O}_2)]$
16. All the  $\text{HCOOH}$  is sodium formate,  $[\text{Na}(\text{HCOO})]$
17. All the Cl is sodium chloride,  $[\text{NaCl}]$
18. All the  $\text{NO}_2$  is sodium nitrite,  $[\text{Na}(\text{NO}_2)]$
19. All the  $\text{C}_2\text{O}_4$  is natroxalate,  $[\text{Na}_2(\text{C}_2\text{O}_4)]$
20. All the  $\text{PO}_4$  is natrophosphate,  $[\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}]$
21. All the  $\text{SO}_4$  is kogarkoite,  $[\text{Na}_3\text{F}(\text{SO}_4)]$
22. Remaining  $\text{NO}_3$  is sodium nitrate,  $[\text{Na}(\text{NO}_3)]$
23. Remaining F is sodium fluoride,  $[\text{NaF}]$
24. Remaining TIC is thermonatrite,  $[\text{Na}_2(\text{CO}_3) \cdot \text{H}_2\text{O}]$
25. Remaining, unidentified TOC is generalized as  $[\text{NaCH}_2\text{O}]$
26.  $[\text{OH}]$  was calculated using the following assumptions: a) all the  $\text{NO}_2$  and  $\text{NO}_3$  is associated with the interstitial liquid; and b) the  $\text{NO}_2:\text{OH}$  ratio is the same for the interstitial liquid and the settled solids
27. Remaining  $\text{H}_2\text{O}$  is presumed to be associated with the dissolved interstitial liquid

## RPP-RPT-59874

**Table 5-10. Phase Composition of Pre-Column Settled Solids.**

Assumed Phase	Phase Molecular Weight (g/mol)	Mass of Phase in Solids (g/100g solids)	Moles of Phase in Solids (mol/100g solid)	Moles of Na Associated to Phase in Solids (mol/100g solid)	Na Remaining (mol) <sup>a</sup>
Na (total)	--	--	--	--	7.96E-01
Al(OH) <sub>3</sub>	78	20.34	2.61E-01	0.00E+00	7.96E-01
Ba(OH) <sub>2</sub>	171.3	0.02	9.18E-05	0.00E+00	7.96E-01
Bi <sub>2</sub> O <sub>3</sub>	466	0.03	5.55E-05	0.00E+00	7.96E-01
PbO <sub>2</sub>	239.2	0.05	2.29E-04	0.00E+00	7.96E-01
Na <sub>3</sub> CaAl <sub>3</sub> Si <sub>3</sub> O <sub>12</sub> (CO <sub>3</sub> )	60	0.22	3.70E-03	3.70E-03	7.92E-01
Ni(OH) <sub>2</sub>	92.7	1.22	1.32E-02	0.00E+00	7.92E-01
Na <sub>2</sub> U <sub>2</sub> O <sub>7</sub> *	634	0.06	9.85E-05	1.97E-04	7.92E-01
Na <sub>2</sub> U <sub>2</sub> O <sub>7</sub> **	634	2.12	3.34E-03	6.68E-03	7.85E-01
CaCO <sub>3</sub>	100.1	0.89	8.93E-03	0.00E+00	7.85E-01
Ce(OH) <sub>3</sub>	191.1	0.02	1.23E-04	0.00E+00	7.85E-01
Cr(O)OH	85	0.06	7.48E-04	0.00E+00	7.85E-01
Fe <sub>2</sub> O <sub>3</sub>	159.7	1.50	9.41E-03	0.00E+00	7.85E-01
Mg(OH) <sub>2</sub>	58.3	0.21	3.60E-03	0.00E+00	7.85E-01
MnO <sub>2</sub>	86.9	0.09	1.01E-03	0.00E+00	7.85E-01
KNO <sub>3</sub>	101.1	0.09	9.18E-04	0.00E+00	7.85E-01
NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	82	0.04	5.17E-04	5.17E-04	7.85E-01
NaHCOO	68	0.08	1.11E-03	1.11E-03	7.84E-01
NaCl	58.5	0.09	1.49E-03	1.49E-03	7.82E-01
NaNO <sub>2</sub>	69	2.15	3.11E-02	3.11E-02	7.51E-01
Na <sub>2</sub> (C <sub>2</sub> O <sub>4</sub> )	134	0.07	5.50E-04	1.10E-03	7.50E-01
Na <sub>7</sub> F(PO <sub>4</sub> ) <sub>2</sub> ·19H <sub>2</sub> O	370.4	20.66	5.58E-02	3.91E-01	3.59E-01
Na <sub>3</sub> F(SO <sub>4</sub> )	184.1	0.43	2.35E-03	7.06E-03	3.52E-01
NaNO <sub>3</sub>	85	3.77	4.44E-02	4.44E-02	3.08E-01
NaF	42	0.23	5.54E-03	5.54E-03	3.02E-01
Na <sub>2</sub> (CO <sub>3</sub> )·H <sub>2</sub> O	106	7.67	7.23E-02	1.42E-01	1.60E-01
TOC (unidentified, NaCH <sub>2</sub> O)	30	0.37	1.23E-02	1.01E-02	1.50E-01
NaOH <sup>f</sup>	40	2.81	7.02E-02	7.02E-02	7.98E-02
H <sub>2</sub> O	18	29.70	1.65E+00	--	--
<b>Sum</b>	--	<b>95.0</b>	--	<b>7.05E-01</b>	--

\* = Accounting for U-235 isotope

\*\* = Accounting for U-238 isotope

f = Assuming that all the NO<sub>2</sub> and NO<sub>3</sub> is associated with the interstitial liquid and that the NO<sub>2</sub>:OH ratio is the same for the interstitial liquid and the settled solids

a = Value obtained by subtracting "Moles of Sodium Associated to Phase in Solids" from the current value of "Sodium Remaining"

## RPP-RPT-59874

The mass balance for the phase composition totaled 95.2% of the settled solid mass. The amount of sodium unaccounted for was 0.080 mol. These values suggest that the assumed phases and their respective concentrations correlate well with the settled solid mass.

### 5.3.2 Suspended Solids SEM Analysis

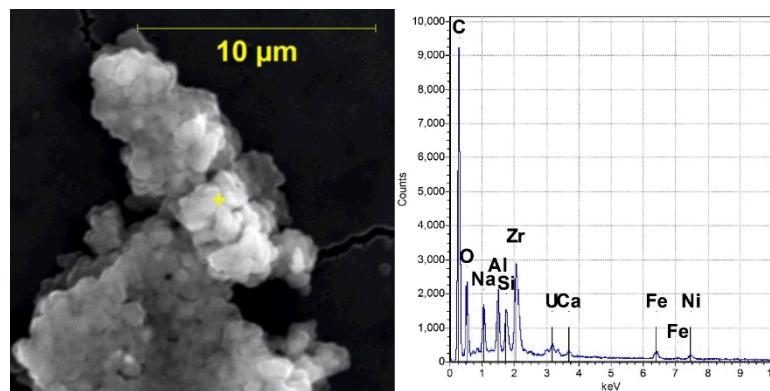
Chemical analysis was not performed on the suspended solids due to their low abundance (12% of the initial composite solids) and difficulty in recovering the suspended solids from the filters. However, a sub-sample was submitted for SEM analysis, which provided sufficient insight to its representative phases.

The suspended particulates that remained in solution after the 24-hr settling period were captured on nylon filters during filtration. The suspended particulates collected from one of the nylon filters were prepared for examination by SEM. Sample preparation was performed by cutting out a section of the nylon filter and placing it in a beaker of water. The beaker was placed in an ultrasonic bath for 5 min to dislodge and break up the particulate adhering to the filter. The suspended particulate was then filtered onto a 45-mm diameter, 0.4- $\mu$ m pore size polycarbonate filter and allowed to dry. A section of the polycarbonate filter was then cut and adhered to a carbon planchet on an aluminum SEM stub and coated with a thin layer of conductive carbon.

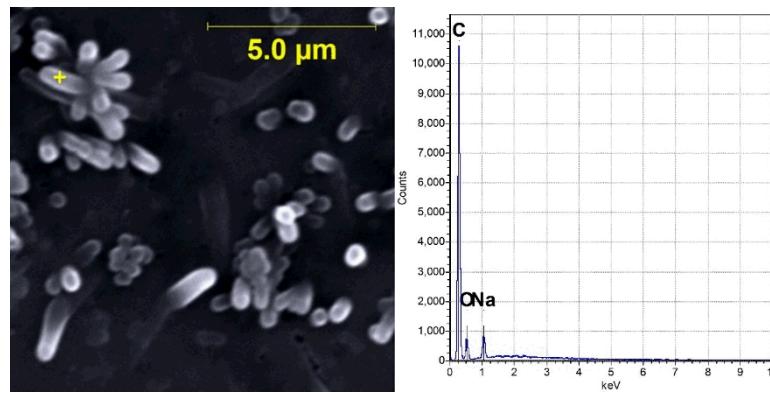
SEM analysis was conducted on the Aspex Explorer SEM in room 1F using the approved laboratory procedure, ATS-LT-161-103, “222-S Laboratory Technology Procedure for the ASPEX Explorer Scanning Electron Microscope.” The sample was imaged in high vacuum mode, allowing imaging with both backscattered and secondary electrons. The SEM was equipped with an energy dispersive X-ray spectrometer (EDS) to collect chemical information on elements heavier than boron. The analysis was conducted by J. S. Lachut on 11/01/16 and 11/02/16. Run details are included in the Laboratory notebook HNF-N-832-1, “Laboratory Controlled Notebook for SEM.”

Two types of particulate were observed. One was aggregates of sub-micron sized particles with a variable chemistry that includes zirconium, aluminum, sodium, silicon iron, nickel and calcium, which is suspect of fine sludge particulate. (See Figure 5-1 of sub-sample S16R000418.) The second particle type consisted of small individual particles, with a spheroidal appearance, often elongate or rod-like. These particles were dominantly composed of carbon and sodium, although it is difficult to tell how much of the carbon is associated with the particle and how much is from filter and carbon planchet of the substrate. (See Figure 5-2 of sub-sample S16R000418.)

## RPP-RPT-59874



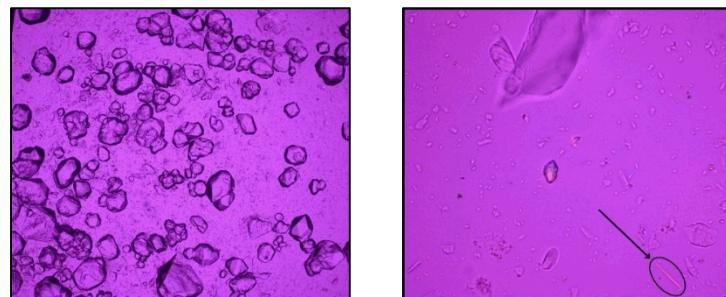
**Figure 5-1. SEM Image of Aggregated Sub-Micron Sized Particles in Suspended Solids.**



**Figure 5-2. SEM Image of Individual Particles in Suspended Solids.**

### 5.3.3 Precipitated Solids PLM Analysis

The solids that precipitated from the post-filtered, pre-column composite were immediately recognizable using PLM. The primary solid identified was the uniformly sized and shaped, isotropic, sodium fluoride phosphate double salt, natrophosphate. In addition, trace amounts of sodium phosphate needles and gibbsite were observed. (See Figure 5-3 of sample S16000501.) No SEM or XRD analysis was performed on this sub-sample, as the PLM morphology and optical properties made the identification certain, and trace phases were not of concern.



**Figure 5-3. PLM Image of Natrophosphate (left) and Phosphate Needles (right) in Precipitated Solids.**

**RPP-RPT-59874**

Solid formation post-filtration is problematic since it could plug the ion exchange column used for cesium removal. Natrophosphate probably derived from mixing highly concentrated fluoride AP tank waste with highly concentrated phosphate AP tank waste. This formation might have been mitigated if sufficient time was allowed for the composite to equilibrate (e.g., for months). Solid formation was not due to temperature fluctuation, as the hot cell temperature remained at  $74 \pm 1^{\circ}\text{F}$ .

## **6    QUALITY ASSURANCE**

### **6.1    Laboratory Analyses**

The samples were analyzed for the analytes listed in LAB-PLN-16-00003, Rev. 1. Quality control measurements required to ensure the quality of the analyses of the test bed composite and solid samples performed at 222-S are specified in ATS-MP-1032, Rev. D-6 and D-7, “222-S Laboratory Quality Assurance Project Plan,” and in WHL-MP-1011, “Quality Assurance Project Plan for 222-S Laboratory.” Results of all QC measurements performed are included with the data summary report (Appendix D).

The analytical methods employed are written to be consistent with SW-846, “Test Methods for Evaluating Solid Waste: Physical/Chemical Methods,” where possible. Due to the hazardous and complex nature of Hanford tank waste samples, most SW-846 test methods performed at the 222 S Laboratory contain deviations that are listed in an appendix to the analytical procedures. All other known deviations or variances from SW-846 are documented in this narrative.

The calibration of each analytical balance used during the test bed operations was verified before use or daily, whichever was less frequent (ATS-LO-140-008, “Routine Use and Quality Assurance for Analytical Balances at 222-S Laboratory Complex”). The verification measurements for each balance were recorded on a “Balance Calibration Verification Check Sheet” (Hanford Site Form A-6005-287) or in laboratory notebook HNF-N-472 1, “General Laboratory Notebook.”

The analytical procedures used for the liquid and solid samples are detailed in Table 6-1 and Table 6-2. Revision 1 of the test plan (LAB-PLN-16-00003, Rev. 1) was issued in September 2016 and included a number of additional constituents that had been inadvertently left off the organic analyte list in Revision 0. All of these additional constituents are included in this report.

## RPP-RPT-59874

**Table 6-1. Liquid Analytical Procedures**

Analysis	Preparation Method	Analysis Procedure
<b>Inorganic Analyses</b>		
Density	Direct	LA-510-112 Rev. 10-3
Anions/Small Org Acids - IC	Direct	LA-533-166 Rev. 2-2
Total Metals – ICP	Direct	LA-505-174 Rev. 1-5
Periodic Table (Cs) - MS	Direct	LA-506-103 Rev. 3-1
OH	Direct	LA-211-102 Rev. 10-2
TGA	Direct	LA-514-115 Rev. 7-2
TIC/TOC	Direct	LA-342-100 Rev. 12-1
Hg – CVAA	Hg Digest - LA-325-110 Rev. 1-2	LA-325-110 Rev. 1-2
<b>Radiochemical Analyses</b>		
Alpha – Separation/GPC	Separation - LA-508-101 Rev. 15-3	LA-508-124 Rev. 3-1
H-3 - Separation /LSC	Separation - LA-218-114 Rev. 8-2	LA-508-122 Rev. 0-2
C-14 - Separation /LSC	Separation - LA-348-104 Rev. 10-5	LA-508-122 Rev. 0-2
Co-60 – Separation/GEA	Separation - LA-548-121 Rev. 10-2	LA-508-167 Rev. 2-1
Ni-63 - Separation /LSC	Separation - LA-285-102 Rev. 7-4 Tracer - LA-505-174 Rev. 1-5	LA-508-122 Rev. 0-2
Sr-90- Separation/GPC	Separation - LA-220-101 Rev. 10-3	LA-508-124 Rev. 3-1
Tc-99 - Separation/LSC	Separation - LA-438-101 Rev. 13-0	LA-508-122 Rev. 0-2
I-129 – Separation/GEA	Separation - LA-378-103 Rev. 14-3	LA-508-167 Rev. 2-1
Cs-137 – Separation/GEA	Separation - LA-548-121 Rev. 10-2	LA-508-167 Rev. 2-1
Actinides (Th-229, U-233, U-234, U-235, U-238, Np-237) – ICP/MS	Direct	LA-506-103 Rev. D-0
Pu (Pu-239/240, Pu-238, Pu-242) – Separation/AEA	Separation - LA-543-102 Rev. 0-2	LA-508-168 Rev. 1-2
Am (Am-241, Am-243, Cm-234/244, Cm-242) – Separation/AEA	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-543-102 Rev. 0-2	LA-508-168 Rev. 1-2
Pu (Pu-241) - Separation/LSC	Separation - LA-543-102 Rev. 0-2	LA-508-122 Rev. 0-2
Dose Hot Cell	Direct	ATS-LO-161-172 Rev. Z-0
<b>Organic Analyses</b>		
VOA-GC/MS	Purge and Trap	LA-523-118 Rev. 8-2 (SW-846 8260C)
SVOA – GC/MS	Extraction – LA-523-115 Rev. 9-2	LA-523-135, Rev. 4-2 (SW-846 8270D)
PCB – GC/ECD	Extraction – LA-523-115 Rev. 9-2	LA-523-140, Rev. 8-1 (SW-846 8280A)

CVAA = cold vapor atomic absorption

ECD = electron capture detector

GPC = gas proportional counter

SVOA = semi-volatile organic analysis

VOA = volatile organic analysis

## RPP-RPT-59874

**Table 6-2. Solids Analytical Procedures.**

Analysis	Preparation Method	Analysis Procedure
<b>Inorganic Analyses</b>		
Anions/Small Org Acids - IC	Water Digest - LA-504-101 Rev. 13-3	LA-533-166 Rev. 2-2
Total Metals - ICP	Acid Digest - LA-505-163 Rev. 9-0	LA-505-174 Rev. 1-5
Periodic Table (Cs) - MS	Acid Digest - LA-505-163 Rev. 9-0	LA-506-103 Rev. 3-1
TGA	Direct	LA-514-115 Rev. 7-2
TIC/TOC	Direct	LA-342-100 Rev. 12-1
Hg - CVAA	Hg Digest - LA-325-110 Rev.1-2	LA-325-110 Rev.1-2
<b>Radiochemical Analyses</b>		
Alpha – Separation/GPC	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-508-101 Rev. 15-3	LA-508-124 Rev. 3-1
H-3 - Separation /LSC	Water Digest Rad - LA-544-133 Rev. 0-3 Separation - LA-218-114 Rev. 8-2	LA-508-122 Rev. 0-2
C-14 - Separation /LSC	Water Digest Rad - LA-544-133 Rev. 0-3 Separation - LA-348-104 Rev. 10-5	LA-508-122 Rev. 0-2
Co-60 – Separation/GEA	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-548-121 Rev. 10-2	LA-508-167 Rev. 2-1
Ni-63 - Separation /LSC	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-285-102 Rev. 7-4 Tracer - LA-505-174 Rev. 1-5	LA-508-122 Rev. 0-2
Sr-90- Separation/GPC	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-220-101 Rev. 10-3	LA-508-124 Rev. 3-1
Tc-99 - Separation/LSC	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-438-101 Rev. 13-0	LA-508-122 Rev. 0-2
I-129 – Separation/GEA	Separation - LA-378-104 Rev. 8-2	LA-508-167 Rev. 2-1
Cs-137 – Separation/GEA	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-548-121 Rev. 10-2	LA-508-167 Rev. 2-1
Actinides (Th-229, U-233, U-234, U-235, U-238, Np-237) – ICP/MS	Acid Digest - LA-505-163 Rev. 9-0	LA-506-103 Rev. D-0
Pu (Pu-239/240, Pu-238, Pu-242) – Separation/AEA	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-543-102 Rev. 0-2	LA-508-168 Rev. 1-2
Am (Am-241, Am-243, Cm-234/244, Cm-242) – Separation/AEA	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-543-102 Rev. 0-2	LA-508-168 Rev. 1-2
Pu (Pu-241) - Separation/LSC	Zr Fusion - LA-549-141 Rev. 13-6 Separation - LA-543-102 Rev. 0-2	LA-543-102 Rev. 0-2
Dose Hot Cell	Direct	ATS-LO-161-172 Rev. Z-0

## RPP-RPT-59874

### 6.2 Holding Times

The test plan did not specify holding times for analyses. Since these samples were archive samples that had been collected several years earlier, specified holding times were deemed unnecessary.

### 6.3 Sample Digestions

Three separate digests were used to digest the solid samples. These are described below.

#### 6.3.1 Acid Digestion

The acid digestion used for metals analysis by ICP/atomic emission spectroscopy (AES) and ICP/MS actinides followed a procedure based on SW-846 Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils," and used nitric and hydrochloric acid (HCl). For the Tc-99 by ICP/MS analysis, the same digestion procedure was used with the exception that HCl was not added because of chlorine interference. Additional HCl was used with this digest for the Sn-126 ICP/MS analysis. For all three digestions, approximately 0.25 g of sample was digested and diluted to a final volume of 50 mL.

#### 6.3.2 Fusion Digestion

The potassium hydroxide fusion digestions were performed using approximately 0.5 g of sample. The digested samples were diluted to a volume of 250 mL with HCl and nitric acids.

#### 6.3.3 Water Digestion

The water digestion was performed using approximately 0.5 g of sample and 50 mL of water. IC was performed using this digestion technique.

### 6.4 Data Summary Report

The Data Summary Report (Appendix D) presents the final analytical results for the constituents identified in the test plan. Some non-requested constituents are also reported. WHL flagged all requested analytes which failed QC parameter results based on Quality Assurance Project Plan (QAPP) requirements.

Required detection limits (RDL) were defined in the test plan and anticipated MDLs specified for select constituents. However, the MDLs listed were, in fact, instrument detection limits and assumed no interferences and no dilutions. Initial results for many analytes did not meet the RDLs requested and had to be re-run at lower dilutions.

## RPP-RPT-59874

The “Det Limit” column in Appendix D contains the MDL for non-radionuclide analyses or the minimum detectable activity (MDA) for radionuclide analyses.

In Appendix D, “A#” indicates the aliquot class or the method used for sample preparation before analysis. For solid samples, the aliquot classes are defined as follows:

- “A” indicates samples were prepared by SW-846 3050B acid digest.
- “HG” indicates samples were prepared by SW-846 7471B acid digest.
- “O” indicates samples were extracted for organic analysis by either SW-846 3540C or SW-846 3545A.
- “W” and “WR” indicate samples were prepared by a water digest.
- “Z” indicates samples were prepared by a fusion digest using a zirconium crucible.

Samples without a letter identifier in the “A#” column were analyzed directly with no separate preparation analysis or with sample preparation performed as a part of the procedure steps.

Manual calculations using rounded results from the Data Summary Report or result calculation forms may differ slightly from the actual results derived from the raw data.

## 7 QUALITY CONTROL

### 7.1 Analytical Quality Control Criteria

WHL calibrated analytical instrumentation and analyzed calibration check standards and blanks per applicable analytical procedures. QC analyses (duplicates, matrix spikes, blanks) were prepared and analyzed per analytical batch, as specified in WRPS's laboratory QAPP (ATS-MP-1032, Rev. D-6 and D-7) and WHL's QAPP (WHL-MP-1011). An analytical batch contains a maximum of 20 samples.

The following guidelines were used in the QC analysis:

- Instrument standards are analyzed at the beginning of each batch, after every ten samples, and at the end of each batch. Acceptance criterion for instrument standards is 90-110% with the exception of TIC/TOC. Acceptance criterion for TIC/TOC instrument standards is 85-115%.
- Instrument blanks are analyzed after instrument standards for ICP, IC, OH, and TIC/TOC; Acceptance criterion is <EQL (estimated quantitation limit).
- A duplicate and matrix spike are analyzed per batch when applicable. (For example, matrix spikes are not applicable to density and TGA).
- Serial dilutions only apply to ICP. A serial dilution is a 5-fold dilution prepared from the sample dilution and only applies to analytes detected at concentrations >50X the MDL. (The MDL is the minimum concentration of an analyte which can be detected above background.)

## RPP-RPT-59874

- Indirect calibration is a standard approach used when a specific element or isotope is not readily available, requiring the use of an isotope thought to behave in a similar fashion, e.g., using the calibration for U-235 to quantitate U-233, U-234, and U-236.

A list of relevant data qualifier flags and their descriptions are outlined in Table 7-1.

**Table 7-1. Analysis Qualifier Flag Definitions.**

Qualifier Flag <sup>1</sup>	Description
B	Applied when the preparation blanks are either >EQL or $\geq 5\%$ of the measured concentration in the sample.
I	Applied when the result was determined via indirect calibration.
J	Applied to results that are considered estimates. Some examples of when a "J" flag may be applied include (but are not limited to): <ul style="list-style-type: none"> <li>Result with concentration <math>\geq</math>MDL, but &lt; the EQL</li> <li>Radiochemical result with counting uncertainty <math>&gt;30\%</math></li> <li>An "unknown" constituent reported for an organic analysis.</li> </ul>
Y	Applied to results that require verbal descriptions or qualifying comments. This flag is used by the chemist, project coordinator, or other technical authority to identify data that is questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations.
a	Applied when the LCS has a percent recovery outside the customer or analytical method specified range. The "a" flag is not applied based on LCSD results.
b	Applied when the MS is outside the acceptance criterion of 75-125%.
c	Applied when the RPD between duplicate samples, LCSDs, or MSDs, is $\geq 20\%$ . The exception is for ICP analysis on fusion digests, where the ICP RPD acceptance criterion for solids is $\leq 35\%$ .
e	Applied when the percent difference between the serial dilution and sample is $\geq 10\%$ .

<sup>1</sup>ATS-GD-1028, "Flagging Data in LIMS."

Due to the large LCS statistical acceptance range for some constituents, some results should be considered qualitative at best. Since WHL did not denote this issue with a qualifier flag, we decided to apply "J" qualifier flags to specific data in the report tables. (Qualifier flags were not modified to the data summary reports in Appendix D.)

## 7.2 Quality Control Summary for Analytical Data

### 7.2.1 Inorganic Constituents

#### Bulk Density

The bulk density was performed on direct aliquots of the aqueous effluent, aqueous pre-column composite, and aqueous post-column composite samples. The standard and duplicate recoveries met the QC criteria.

## RPP-RPT-59874

### Percent Water

The percent weight loss was measured for the aqueous pre-column composite, aqueous post-column composite and solid pre-column composite samples using TGA. The measurement was performed on direct subsamples of each sample. Per procedure, the results were reported as percent water with the assumption that all weight loss below 250 °C was due to water loss. However, this assumption might be incorrect because there could be other volatile products that contribute to weight loss below 250 °C that are not easily differentiated using the current method at the 222-S Laboratory. The standard and duplicate recoveries met the QC criteria.

### ICP Metals

Analysis for the metals and inorganic cations requested was performed by ICP/AES on aliquots of the aqueous pre-column composite and aqueous post-column composites and on an acid digestion of the solid composite sample. Two splits of the pre-column composite were submitted for analysis, and duplicate analytical splits of all three samples were run as part of the analytical batches.

Standard recoveries for the aqueous samples ranged from 93 – 106%. For the solids, the standard recoveries ranged from 88.3 – 99.8%.

Duplicate analyses are usually within 10% for the aqueous samples except where the detection limit is approached. Many of the metals are “J” flagged because the concentration is near the detection limit, including copper (29.8% RPD), lead (15.4% RPD), palladium (31.8% RPD), rhodium (12.4% RPD), silicon (24.8% RPD), and vanadium (23.8% RPD) in the pre-composite sample. In the post-composite sample, barium (10.9% RPD), copper (20.9% RPD), rhodium (23.4% RPD), and vanadium (13.5% RPD) were “J” flagged. RPDs greater than 10% include silicon in the post-column sample with an RPD of 24.8% and phosphorous in the post-column sample with an RPD of 16.1%. The silicon values are flagged with a “c” flag, indicating this RPD is outside the QC limit.

Duplicate analyses for the acid-digested solids are within 20% for most analytes. Again, most RPD’s greater than 20% are due to low concentrations (silver, 20.7% RPD; bismuth, 21.9% RPD; cerium, 21.5% RPD; potassium, 23.2% RPD; lithium, 46.7% RPD; molybdenum, 24.7% RPD; samarium, 24.7% RPD; titanium, 52.6% RPD; yttrium, 33.5% RPD; and zirconium, 29.1% RPD). Phosphorous, with an RPD of 25.5%, and uranium, with a 21.6% RPD, cannot be attributed to low concentration, as the results are significantly higher than the detection limit. Sample inhomogeneity may be the cause of these higher RPDs.

The post-composite liquid and the acid-digested solids were spiked with known amounts of the metals. Spike recoveries were between 83% and 106% for the post-composite liquid. For the solids, spike recoveries were more variable, with most results falling between 80 and 120%.

### TIC/TOC

The TIC/TOC analysis was performed by coulometric detection on an aqueous pre-column composite, aqueous post-column composite, and solid pre-column composite sample. The TOC result for the solid pre-column composite sample exceeded the RPD limit of 20% at 30.5%;

## RPP-RPT-59874

therefore, a “c” flag was applied to the sample result. All other QC and reporting limit requirements in the QAPP were met.

### **Mercury**

Analysis for mercury was performed by CVAA on acid-digested aliquots of the aqueous pre-column composite, aqueous post-column composite, and solid pre-column composite samples. The aqueous pre-column and post-column composite sample results contained a “J” flag since the initial calibration blank was observed to be greater than the detection level. For the solid pre-column composite sample, the spike recovery exceeded the acceptance criteria of 75% to 125% at 264%. Therefore, a “b” flag was applied to the sample result. All other QC and reporting limit requirements in the QAPP were met.

### **Uranium**

Metal analysis for uranium was performed by ICP/AES for the aqueous and solid pre-column composites. For the solid pre-column composite sample (S16000405), the spike recovery exceeded the acceptance criteria of 75% to 125% at 436% and exceeded the RPD limit of 20% at 21.6%.

Metal analysis for uranium was performed by ICP/MS for the aqueous post-column composite since the uranium was failing low for the opening interference check standard (ICSAB) by ICP/AES. (There are no flagging requirements for ICSAB failures.) The post-column composite sample was reanalyzed by ICP/AES to improve the QC results, but reanalysis produced a failing duplicate RPD for uranium. Therefore, the post-column composite uranium was analyzed by ICP-MS, with the U-233 (<2.5E-04  $\mu\text{g/mL}$ ), U-234 (0.0176  $\mu\text{g/mL}$ ), U-235 (0.653  $\mu\text{g/mL}$ ), and U-238 (25.9  $\mu\text{g/mL}$ ) values being summed for the total uranium concentration. The U-233 result was flagged with an “I” to denote the use of indirect calibration (isotopic substitution). The U-235 result was flagged with a “B” since the amount of U-235 detected in the blank was seven orders of magnitude lower than the result. All other QC requirements in the QAPP were met.

### **Technetium-99**

The ICP/MS Tc-99 analysis was performed on the acid digest without the use of HCl. All sample results for Tc-99 were above the MDL. There were no notable issues with the Tc-99 analysis, and all QC requirements in the QAPP were met.

### **Actinides**

The ICP/MS analysis of actinides was performed on acid digests of each sample. The RDLs were met for all required constituents.

## RPP-RPT-59874

## 7.2.2 Radiochemical Constituents

### Gamma Energy Constituents

GEA was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. All sample results for Cs-137 were above the MDA. The aqueous post-column composite and the pre-column composite solid sample results for Co-60 were above the MDA. All other QC requirements in the QAPP were met.

### Strontium-89/90

The Sr-89/90 analysis was performed by gas proportional counting directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. There were no notable issues with this analysis. All QC requirements in the QAPP were met.

### Carbon-14

The C-14 analysis by LSC was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a water-digested aliquot of the pre-column composite solids. All QC requirements in the QAPP were met.

### Tritium

The H-3 analysis by LSC was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a water-digested aliquot of the pre-column composite solids. All sample results for H-3 met the RPD requirement except for the pre-column solids sample (S16000409), which was above the RPD limit of 20%. A “J” flag was applied to this sample to indicate that this result should be considered an estimate due to the increased uncertainty near the detection limit. All other QC requirements in the QAPP were met.

### Nickel-63

The Ni-63 analysis by LSC was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. All QC requirements in the QAPP were met.

### Technetium-99

The Tc-99 analysis by LSC was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. All QC requirements in the QAPP were met.

### Iodine-129

The I-129 analysis by low energy GEA was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on an iodine-specific, fusion-

## RPP-RPT-59874

digested aliquot of the pre-column composite solids. All QC requirements in the QAPP were met.

### **Americium-241, -243; Curium-242, -243/244**

The Am-241, Cm-242, and Cm-244 analysis by AEA was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. Concerning the Am results, the Am-241 analysis met all QC requirements listed in the QAPP. (No analysis for Am-243 was performed since it is not associated with tank waste processing history. If present, it would be at sample tracer quantities, as it is used as a tracer for Am-241 and Cm isotope analysis.) Concerning the Cm-242 results, the RPD for the pre-column solid sample (S16R000493) exceeded the less than or equal to 20% requirement, at 75%. With the exception of the aqueous pre-column duplicate sample (S16R000377), Cm-242 sample results contained a counting error greater than 30%. With the exception of the pre-column solid sample (S16R000493), sample results for Cm-243/244 contained a counting error greater than 30%. A “J” flag was applied to the results that contained a counting error greater than 30% due to the high uncertainty near the detection limit, which indicates that these results should be considered estimates. The Cm-242 and Cm-243/244 results met all other QC requirements listed in the QAPP.

### **Isotopic Plutonium-238, 239/240, 242**

The isotopic plutonium analysis by AEA was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. The aqueous pre-column sample (S16R000376) and solid sample (S16R000493) analysis result for Pu-242 exceeded the RPD requirement of less than or equal to 20%, at 90.1% and 23.7%, respectively. Therefore, a “c” flag was applied to the sample results. Additionally, all Pu-242 analysis results contained interferences of the Pu-239/240 peak scattering into the Pu-242 region of interest; a “Y” qualifier flag was applied to the results due to their uncertainty, indicating that these results should be considered as conservative and are not to be used for reliable quantification. All other QC requirements in the QAPP were met.

### **Plutonium-241**

The Pu-241 analysis by LSC was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on a fusion-digested aliquot of the pre-column composite solids. The Pu-241 result for the solid sample (S16R000493) exceeded the less than or equal to 20%, at 31.1%; therefore, a “c” flag was applied to the sample result. All other QC requirements in the QAPP were met for the samples.

### **Thorium-229**

The Th-229 analysis by ICP/MS was performed directly on an aliquot of the aqueous pre-column composite and aqueous post-column composite, and on an acid-digested aliquot of the pre-column composite solids. All sample results for Th-229 were flagged with an “I” to denote the use of indirect calibration (isotopic substitution). All sample results for Th-229 were below the MDL. All other QC requirements in the QAPP were met for the samples.

## RPP-RPT-59874

### 7.2.3 Organic Constituents

Organic analyses were run on both the pre-column and post-column aqueous composite samples.

#### 7.2.3.1 Polychlorinated Biphenyls

The PCB analysis by GC/ECD was performed on a methylene chloride extraction of approximately 1 g from each sample. All results were below detection limits. All QC requirements in the QAPP were met for the samples.

#### 7.2.3.2 Semivolatile Organics

SVOA by GC/MS was performed on methylene chloride extractions of a direct sample aliquot. This method was met with some challenges, as the sample density (1.2 g/mL) was similar to that of methylene chloride, resulting in poorly defined layers. In addition, difficulties were encountered in achieving the post-column composite SVOA detection limits requested in the test plan. Therefore, the composite was extracted with additional sample material to achieve better detection limits and the 5X dilution data for the post-column composite were reported.

#### Initial Calibration

##### Pre-Column Composite

The initial calibration was completed on 09/29/16. The calibration passed at <20% relative standard deviation (RSD) for all constituents which used average response factor and passed at a minimum of  $r^2 \geq 0.99$  for all constituents which used a linear regression fit. The following constituents required linear regression fits:

- Hexachlorocyclopentadiene
- 2,4-Dinitrophenol
- 4-Nitrophenol
- 4,6-Dinitro-2-methylphenol
- Tributylphosphate
- Pentachlorophenol
- Butylbenzylphthalate
- Bis(2-ethylhexyl)phthalate
- Di-n-octylphthalate

For the MQC1 (LLS-1) level, all of the linear fit constituents above (except for 2,4-Dinitrophenol) met recovery limits of 70 to 130%. 2,4-Dinitrophenol failed the low level standard (LLS) high at 146% recovery. The MQC2 (LLS-2) passed for all constituents listed above. All calibrated constituents passed the initial calibration verification (ICV) criteria.

## RPP-RPT-59874

### **Post-Column Composite**

The initial calibration for the improved project detection limits was completed on 11/02/2016. The calibration passed at <20% RSD for all constituents which used average response factor and passed at minimum  $r^2 \geq 0.99$  for all constituents which used a linear regression fit. The following constituents required linear regression fits:

- 2,4,5-Trichlorophenol
- Hexachlorobutadiene
- Hexachloroethane
- Cyclohexanone
- Cresols (all)
- Nitrobenzene
- Pyridine
- Isobutyl alcohol
- 2,4-dinitrotoluene
- 2-Ethoxyethanol

For the MQC1 (LLS-1) level all of the linear fit constituents met recovery limits of 70 to 130%. All calibrated constituents passed the Initial Calibration Verification (ICV) criteria. All target constituents were below the detection limit. The ICAL received a first level review but did not have a second peer review.

### **Daily Tuning Check**

#### **Pre-Column Composite**

The decafluorotriphenylphosphine (DFTPP) tune verification standard passed required tuning criteria for mass ratios, resolution, response, component degradation, and tailing. All analytical runs were injected within 12 hr of an acceptable tune verification.

#### **Post-Column Composite**

The DFTPP tune verification standard passed required tuning criteria for mass ratios, resolution, response, and component degradation. The tailing factor was greater than 2.0 at 2.08. However, this did not affect the ability to properly quantitate and identify target constituents as indicated by the passing continuing calibration verification (CCV). Therefore, the data is acceptable. The verbiage in both SW-846 and the laboratory SOP use the work “should”, not “shall” when referring to tailing factor requirements. A tailing factor greater than 2 indicates that the GC may need front end maintenance including trimming the column and changing out the injection port liner and gold seal. All analytical runs were injected within 12 hours of an acceptable tune verification.

**RPP-RPT-59874****Continuing Calibration Verification % Drift****Pre-Column Composite**

All calibrated constituents passed CCV criteria.

**Post-Column Composite**

The CCV passed for all target analytes. The percent drift for the constituents were less than 20%.

**Internal Standard Recoveries****Pre-Column Composite**

Internal standard response and retention time requirements passed required criteria for the CCV. All samples and other standards also had normal internal standard responses.

**Post-Column Composite**

Internal standard response and retention time requirements passed the required acceptance criteria with the exception of Perylene-d12. For the samples and the MS/MSD, the internal standard Perylene-d12 had a very low response due to matrix effects, with recoveries of 27 – 40%. Only the field blank sample does not have this effect.

**Surrogate Recoveries****Pre-Column Composite**

Acid surrogates (2-Fluorophenol, Phenol-d6, and 2,4,6-Tribromophenol) had no recoveries in the samples due to conversion of phenols to nitrophenols by the highly nitrated sample matrix. This is a known artifact of SVOA acid surrogates with Hanford tank samples. All other sample surrogates, as well as both the acid and base/neutral surrogates for the batch QC, passed criteria method criteria.

**Post-Column Composite**

There were several surrogate recoveries that were outside the acceptable recovery limits on the low side. These samples and the associated MS/MSD samples showed the same pattern, and the blank and laboratory control sample (LCS) were within specification; therefore, reanalysis was not required. This pattern of the sample and associated MS/MSD failure of the surrogate recoveries indicate a matrix effect. The spikes were almost completely eliminated due to “nitration” by the matrix. This sample was a 5x dilution. The adjusted % recoveries were also included.

The below constituents were not recovered for the MS/MSD spike samples. This was most likely due to degradation by the sample matrix.

**RPP-RPT-59874**

- 2,4,5-Trichlorophenol
- 3- & 4-Methylphenol
- Isobutyl alcohol
- Pyridine

**Prep Blank****Pre-Column Composite**

All target analytes in the blank were below the detection limit.

**Post-Column Composite**

All target analytes in the blank were below the detection limit.

**Laboratory Control Sample Recoveries****Pre-Column Composite**

All target constituents were within the administrative LCS limits of 70% – 130% with the exception of hexachlorobutadiene (14.62% recovery). Therefore, an “a” flag was applied to the hexachlorobutadiene result. Statistical limits have not been developed for this constituent.

Isobutyl alcohol had a low recovery (39.58%) but the result was not “a” flagged as the recovery fell within the LCS statistical limits of 0% – 122%. With the large statistical acceptance range, a great deal of uncertainty is expected in the analysis. Since the result should be considered qualitative at best and WHL decided not to flag the result, we applied a “J” flag to the result in Table 5-1.

(The SVOA hexachloroethane result was not reported in Table 5-1. Instead, the VOA result was reported due to the lower method detection limit obtained.)

**Post-Column Composite**

All target constituents were within the administrative LCS limits of 70% – 130% with the exception of 2-Ethoxyethanol (64.85% recovery) and pyridine (62.83% recovery). An “a” flag was applied to the 2-Ethoxyethanol and pyridine result. Statistical limits have not been developed for these constituents.

Low recoveries were obtained for isobutyl alcohol (30.80%) and hexachlorobutadiene (17.65%). These results were not “a” flagged as their recoveries fell within the LCS statistical limits of 0% – 122% and 0% – 125%, respectively. With the large statistical acceptance range, a great deal of uncertainty is expected in the analysis. Since the result should be considered qualitative at best and WHL decided not to flag the result, we applied a “J” flag to the result in Table 5-4.

## RPP-RPT-59874

(The SVOA hexachloroethane result was not reported in Table 5-4. Instead, the VOA result was reported due to the lower method detection limit obtained.)

### **Quality Control Issues**

#### **Pre-Column Composite**

None of the QC issues discussed below adversely impact conclusions reached in this study.

#### **Post-Column Composite**

None of the QC issues discussed below adversely impact conclusions reached in this study.

#### **7.2.3.3 Volatile Organics**

Volatile organic compounds (VOC) analysis by GC/MS was performed on a direct subsample. Approximately 0.5 g of sample was used for VOC analysis.

Samples S16R000374 and S16R000375 were initially analyzed on 09/22/16. The MS/MSD was prepared using sample S16R000374. All of the polar analytes failed to pass the CCV and several failed the LCS.

The system was recalibrated on 9/27/16 and the samples were rerun on 09/29/16. An additional “Post-column” sample S16R000394 was made available for analysis at this time and was added to the batch. The MS/MSD was prepared from sample S16R000375. Although the QC standards passed acceptance requirements for all samples, internal standards and surrogates failed extremely low (or not at all) on S16R000375, S16R000375MS, and S16R000375MSD. It is believed that the loss of internal standards was due to a stuck valve or other malfunction in the purge and trap instrument, since all samples and QC were spiked at the same time with the same internal standard mix.

A second rerun was performed on 10/04/16. For this run, vinyl chloride and trichlorofluoromethane (along with the remaining non-target constituents in the gas mix) failed to pass CCV, LCS, and MS/MSD recovery criteria. Each constituent appeared to be high by a factor of two. All remaining constituents passed required criteria.

It was decided for samples S16R000374 and S16R000375 to report only vinyl chloride and trichlorofluoromethane from the 9/22/16 analysis and all other constituents from the 10/04/16 analysis. Results from samples S16R000374 and S16R000375 were merged together in OmniLIMS batch 00067733.

Sample S16R000394 (Post-column) was reported from the 9/29/16 analysis because the 10/04/16 run failed the gases and the 9/22/16 run did not include this sample. Although the 9/29/16 run did not have an acceptable MS/MSD, it was assumed that the matrix of the “PRE” and “POST” samples were the same, and therefore, the MS/MSD from 9/22/16 was applicable for the vinyl chloride result and trichlorofluoromethane. The MS/MSD from 10/04/16 was applicable for the

**RPP-RPT-59874**

remaining constituents. Sample S16R000394 was reported in a separate batch 00067870, discussed below.

**Initial Calibration****06/14/16 Calibration**

This calibration data was used for analyses performed on 9/22/16 (vinyl chloride and trichlorofluoromethane). The initial calibration was completed on 06/14/16. All calibrated constituents passed the linearity requirement of 20% RSD or minimum  $r^2 = 0.99$ . All calibrated constituents passed the ICV criteria of 70% to 130% with the exception of Acetone at 135.10%.

**09/27/16 Calibration**

This calibration was used for analyses performed on 10/04/16 (all remaining constituents). The initial calibration was completed on 09/27/16. All calibrated constituents passed the linearity requirement of 20% RSD or minimum  $r^2 = 0.99$ . All calibrated constituents passed the ICV criteria of 70% to 130%.

**Daily Tuning Check****09/22/16 Runs**

The daily 4-bromofluorobenzene (BFB) tuning check passed for all criteria. All samples were injected within 12 hr of the tuning standard.

**10/04/16 Runs**

The daily BFB tuning check passed for all criteria. All samples were injected within 12 hr of the tuning standard.

**Daily Calibration Verification Check****09/22/16 Runs**

The following target constituents failed the daily calibration verification:

- 2-Butanone
- Acetone
- 4-Methyl-2-pentanone (MIBK)
- N-Butanol

These constituents were reported from the 10/04/16 analysis.

## RPP-RPT-59874

**10/04/16 Runs**

- All target constituents passed the daily calibration verification check except for vinyl chloride and trichlorofluoromethane. These constituents were reported from the 9/22/16 analysis.

**Internal Standard Recoveries****9/22/16 Run**

Internal standards passed acceptance criteria for all samples and QC.

**10/04/16 Run**

Internal standards passed acceptance criteria for all samples and QC.

**Surrogate Recoveries****9/22/16 Run**

Surrogate recoveries passed for all samples and standards.

**10/04/16 Run**

Surrogate recoveries passed for all samples and standards.

**Prep Blank****9/22/16 Run**

The prep blank met acceptance requirements for all target constituents reported from this run. Toluene was detected at 0.23 µg/L but was reported from the 10/04/16 run.

**9/22/16 Run**

The prep blank met acceptance requirements for all target constituents reported from this run. No constituents were detected above their respective detection limits.

**Blank Spike Recoveries (LCS)****09/22/16 Run**

The LCS (blank spike) passed required criteria for all target constituents except the polars (n-butanol, MIBK, MEK). The 9/22/16 run was only used to report vinyl chloride and trichlorofluoromethane. All polars and other non-gases were reported from the 10/04/16 run.

**RPP-RPT-59874****10/04/16 Run**

The LCS (blank spike) passed required criteria for all target constituents except the gases (vinyl chloride and trichlorofluoromethane). Vinyl chloride and trichlorofluoromethane were reported from the 9/22/16 run.

**Matrix Spike Recoveries**

All constituents passed MS/MSD recoveries except for the following:

- 1,1,2-Trichloroethane
- 1,1-Dichloroethene
- Carbon disulfide
- Ethyl acetate
- n-Butanol
- 2-Butanone (MEK)
- 4-Methyl-2-pentanone (MIBK)

The LCS passed for these constituents and the %RPDs between the MS and MSD were under 12% for all failing constituents. The %RPDs for all remaining constituents met method requirements of 30%.

**7.2.3.3.1 VOA Batch 00067870**

Post-column sample S16R000394 was analyzed on 9/29/16 as part of a rerun including samples S16R000374 and S16R000375. The MS/MSD was prepared from sample S16R000375. Although the CCV, continuing calibration blank, and LCS standards passed all acceptance requirements, internal standards and surrogates failed extremely low (or not at all) on S16R000375, S16R000375MS, and S16R000375MSD. Sample S16R000394 was not affected. It is believed that the loss of internal standards was due to a stuck valve or other malfunction in the purge and trap instrument since all samples and QC were spiked at the same time with the same internal standard mix.

**Initial Calibration 09/27/16**

All calibrated constituents passed the linearity requirement of 20% RSD or minimum  $r^2 = 0.99$ . All calibrated constituents passed the ICV criteria of 70% to 130%.

**Daily Tuning Check**

The Daily BFB tuning check passed for all criteria. All samples were injected within 12 hr of the tuning standard.

**RPP-RPT-59874****Daily Calibration Verification Check**

All target constituents passed %Diff/%Drift requirements for the daily calibration verification.

**Internal Standard Recoveries**

Internal standards passed acceptance criteria for the sample and all associated QC samples except the MS and MSD.

**Surrogate Recoveries**

Surrogate recoveries passed acceptance criteria for the samples and associated QC except the MS and MSD.

**Prep Blank**

The prep blank met acceptance requirements for all target constituents reported from this run. Acceptance criteria specifies that the concentration in the blank be either less than the EQL (MDL x 10) or less than 5% of the sample contribution. Toluene was detected at 0.27 µg/L in the blank and 10.7 µg/L in the sample. The EQL was 0.522 µg/L for the blank and 20.9 µg/L for the sample. Concentration in the blank was less than both EQLs. Five percent of the toluene sample concentration comes out to 0.535 µg/L (0.05\*10.7), therefore, the toluene blank concentration meets both criteria.

**Blank Spike Recoveries (LCS)**

The LCS (blank spike) passed required criteria for all target constituents.

**Matrix Spike Recoveries**

The MS/MSD was performed on sample S16R000375 (PRE Sample). The matrix spike failed all recoveries due to loss of internal standard as discussed in the summary section. Matrix effects for vinyl chloride and trichlorofluoromethane were taken from the MS/MSD run on 9/22/16, and matrix effects for the remaining constituents were taken from the MS/MSD run from 10/04/16 (See OmniLIMS batch 00067733).

All constituents passed MS/MSD recoveries except for the following:

- 1,1,2-Trichloroethane
- 1,1-Dichloroethene
- Carbon disulfide
- Ethyl acetate
- n-Butanol

## RPP-RPT-59874

- 2-Butanone (MEK)
- 4-Methyl-2-pentanone (MIBK)

The LCS passed for these constituents and the %RPD between MS and MSD were under 12% for all failing constituents. The %RPDs for all remaining constituents met the method requirements of 30%.

#### 7.2.4 IC-Anions/Organic Acids

- For the pre-column aqueous composite (S16R000370 & S16R000371), the average PO<sub>4</sub>/P ratio was approximately 1.07 times higher than expected, 3.29 vs 3.06.
- For the post-column aqueous composite (S16R000384), the PO<sub>4</sub>/P ratio was approximately 1.11 times higher than expected, 3.40 vs 3.06.
- For the pre-column aqueous composite (S16R000370 & S16R000371), the SO<sub>4</sub>/S ratio was approximately 0.87 times lower than expected, 2.62 vs 3.00.
- For the post-column aqueous composite (S16R000384), the SO<sub>4</sub>/S ratio was approximately 0.90 times lower than expected, 2.69 vs 3.00.

#### 7.2.5 Mass and Charge Balance

A mass balance is the sum of all chemical species in a sample. The calculation shown below is used to convert an analyte concentration to weight percent:

$$\text{Conc}_{\text{analyte}} \times \frac{\text{FW}_{\text{compound}}}{\text{FW}_{\text{analyte}}} \times \frac{V_{\text{Liq}}}{(10^4 \times m_{\text{archive}})} \quad (7-1)$$

Where:

Conc <sub>analyte</sub>	=	concentration provided from the chemical analysis (µg/mL)
FW <sub>compound</sub>	=	formula weight of the assumed compound (mg/mmol)
FW <sub>analyte</sub>	=	formula weight of the analyte (mg/mmol)
V <sub>Liq</sub>	=	V <sub>Total</sub> - V <sub>NaOH</sub> (mL)
m <sub>archive</sub>	=	weight of test sample (g)

Note: 10<sup>4</sup> is a conversion factor that combines the µg to g and weight percent conversions

Ideally, the mass balance for all constituents in a sample should be 100%. However, for aqueous samples, mass balance values from 95-102% are generally considered excellent, while values from 90-110% are acceptable. For solid samples, mass balance values from 90-110% are generally considered good, while values from 80-120% are acceptable. The charge balance calculation converts each analyte from mmol/g to mEq/g, sums the positive and negative equivalents, and then calculates the +/- ratio. Ideally, this ratio should be 1.00. However, for aqueous samples, charge balance values from 0.95 to 1.02 are generally considered excellent, while values from 0.90 to 1.05 are acceptable. For solid samples, charge balance values from 0.90 to 1.10 are generally considered excellent, while values from 0.80 to 1.20 are acceptable. If the values are not within the acceptable range, the data should be reviewed for errors.

## RPP-RPT-59874

### 7.2.5.1 Pre-Column Aqueous Composite Mass and Charge Balance

The average mass balance for the pre-column composite (S16R000370 and S16R000371) was 98.8%. The charge balance was 1.09. Both the mass and charge balance data results would be considered excellent.

### 7.2.5.2 Post-Column Aqueous Composite Mass and Charge Balance

The mass balance for the pre-column composite (S16R000384) was 100.0%. The charge balance was 1.01. Both the mass and charge balance data results would be considered excellent.

### 7.2.5.3 Pre-Column Solid Composite

The mass balance for the post-column solid composite (S16R000399) was 94.9%. The charge balance was 1.13. Both the mass and charge balance data results meet acceptance criteria.

## 8 PROCESS KNOWLEDGE

### 8.1 Organic Process Knowledge and Calculations

Regarding the request for process knowledge on methanol, 2-nitropropane, and 2-ethoxyethanol, it is of limited value for the purpose of determining whether the solidified waste will meet all land disposal restrictions (LDRs) prior to disposal. In most cases, the 40 CFR 268.40 and 268.48 treatment standards for these constituents will not be applicable. For 2-nitropropane, it has a RCRA treatment standard technology code for combustion (CMBST) under the waste description “F005 solvent waste containing 2-nitropropane as the only listed F001-5 solvent.” This treatment standard is not applicable because 2-nitropropane will not be the only F-listed constituent of the waste. Also, there is no UTS for 2-nitropropane. For 2-ethoxyethanol, it has a treatment standard of CMBST under the waste description “F005 solvent waste containing 2-ethoxyethanol as the only listed F001-5 solvent.” This treatment standard is not applicable because 2-ethoxyethanol will not be the only F-listed constituent of the waste. Also, there is no UTS for 2-ethoxyethanol. For methanol, the treatment standards appear in a few places, including:

- a) Methanol has a NWW treatment standard of “0.75 mg/L TCLP” under the waste description “F003 and/or F005 solvent wastes that contain any combination of one or more of the following three solvents as the only listed F001-5 solvents: carbon disulfide, cyclohexanone, and/or methanol.” This treatment standard is not applicable because methanol does not occur alone or in combination with these other two solvents as the only F-listed constituents of the waste.

## RPP-RPT-59874

- b) Methanol has a NWW treatment standard of “Not Applicable” under the “F001, F002, F003, F004, & F005” treatment standard. In this case the applicable treatment standard is - “Not Applicable.”
- c) Methanol has a NWW UTS of “0.75 mg/L TCLP.” This is the only treatment standard applicable among these three chemical constituents. This is where process knowledge is needed to help PFNW predict whether the solidified waste will meet the UTS prior to disposal.

The SVOAs for the test bed sample were all below the limit of detection. However, the detection limits were an order of magnitude above the “Action levels” of 0.050 ppm. Process knowledge was used to rule out the occurrence of these constituents.

For 2,4,5-trichlorophenol, hexachlorobutadiene, hexachloroethane, nitrobenzene, pyridine, cyclohexanone, or cresols (all), it cannot be categorically said that none of these are present in tank waste (because there’s a remote possibility that they might be decomposition products of some other organic), but we can say with some confidence that they were never used as process chemicals that might have entered the waste stream. They are not listed in WHC-EP-0172, “Inventory of Chemicals Used at Hanford Production Plants and Support Operations.”

As for methanol ( $\text{CH}_3\text{OH}$ ; shorthand MeOH), it has not been analyzed in tank waste liquid (supernate) samples. It has been analyzed, however, in tank waste head space (vapor) samples. The average MeOH concentration found in the headspace in single shell tanks is 2.7 ppmv (parts per million by volume, or  $2.7 \text{ mL/m}^3$ ) (Tank Waste Information Network System (TWINS), Queried 11/17/16 and 02/06/17, [Vapor, Headspace Sample Analysis, Analysis Results], <http://twins.labworks.org>). In the double shell tanks that contributed to the test bed composite sample, the average concentration of MeOH in the headspace is 0.6 ppmv with a maximum of 2.1 ppmv (Tank Waste Information Network System (TWINS), Queried 11/17/16 and 02/06/17, [Vapor, Headspace Sample Analysis, Analysis Results], <http://twins.labworks.org>). If we assume that 2 ppmv is an upper bound for the concentration of MeOH in the vapor, we can calculate a reasonable (order-of-magnitude) estimate of the concentration of MeOH in the liquid phase of the test bed sample.

Raoult’s Law relates vapor phase and liquid phase concentrations of a volatile solvent through Equation 8-1:

$$P_s = P^0_s X_s \quad (\text{Eq 8-1})$$

where  $P_s$  is the vapor pressure of component S in the system at equilibrium,  $P^0_s$  is the vapor pressure of pure component S, and  $X_s$  is the mole fraction of component S in the solution. In our case, component S is MeOH, and Equation 8-1 can be rearranged to solve for the concentration of MeOH in the aqueous solution (tank waste supernatant liquid) using Equation 8-2:

$$X_{\text{MeOH}} = P^0_{\text{MeOH}} / P_{\text{MeOH}} \quad (\text{Eq 8-2})$$

## RPP-RPT-59874

Assuming:

- Tank temperature = 100 °F (38 °C)
- Concentration of MeOH in the tank headspace is 2 ppmv
- $P_{\text{MeOH}}^0$  at 38 °C is 240 torr (Antoine's equation, "The Antoine Equation for Vapor-Pressure Data" (Thomson 1946))
- MeOH vapor behaves like an ideal gas
- The tank headspace is at atmospheric pressure, 760 torr
- The system (tank supernatant liquid / tank headspace vapor) is at equilibrium
- The effect of dissolved salts on the MeOH vapor pressure is small compared to other factors (such as tank ventilation)
- 1 L H<sub>2</sub>O at 38 °C contains 55.2 mol H<sub>2</sub>O
- Molecular weight of MeOH = 32.0 g/mol

Calculate:

- $P_{\text{MeOH}} = (2.00 \times 10^{-6})(760) = 1.52 \times 10^{-3}$  torr (pressure directly proportional to volume for ideal gas mixture)
- $X_{\text{MeOH}} = (240) / (1.52 \times 10^{-3}) = 6.33 \times 10^{-6}$  mol MeOH per mol aqueous phase (Eq 8-2)
- MeOH concentration in aqueous phase =

$$(6.33 \times 10^{-6} \text{ mol/mol H}_2\text{O})(32.0 \text{ g/mol}) / (55.2 \text{ mol H}_2\text{O/L}) = 0.011 \text{ g/L} = 11 \text{ mg/L}$$

Therefore, a headspace concentration of 2 ppmv MeOH corresponds a MeOH concentration of 11 mg/L in the aqueous phase.

As for PCBs, the TWINS compiled data on the supernate grab samples used in the test bed composite ranged from 0.0013 to 0.126 ug/mL (TWINS, Queried 11/14/16, [Sample Analysis, Tank Results RPP 241, Tank Results (Hide QA Records)], <http://twins.labworks.org>).

## 8.2 Radionuclide Process Knowledge

Chlorine-36 (Cl-36) has not been measured or estimated in Hanford Tank Waste inventory, based on a query of the TWINS database which contains the Hanford BBI (TWINS, Queried 02/06/17, [Sample Analysis, Tank Results RPP 241, Tank Results (Hide QA Records)], <http://twins.labworks.org>). The parent isotopes of Cl-36 are Argon-36, Chlorine-35, Potassium-39, and Calcium-40. No recorded measurement or estimate of inventory for these parent isotopes exists in the Hanford BBI (TWINS, Queried 02/06/17, [Sample Analysis, Tank Results RPP 241, Tank Results (Hide QA Records)], <http://twins.labworks.org>). Furthermore, Argon-36, a primary decay product (stable isotope), has not been measured or estimated in the Hanford BBI. However, a source and concentration of Cl-36 can be conservatively estimated based on calculated radionuclides inventories reported in RPP-13489, "Activity of Fuel Batches Processed Through Hanford Separations Plants," for spent fuel processed through Hanford separations

## RPP-RPT-59874

plants from 1944 through 1989. The approach used to estimate Cl-36 utilizes the maximum Cl-36 to Tc-99 ratio reported. Table 8-1 contains transcribed concentrations of Cl-36 and Tc-99 from RPP-13489 (Table H-1) and calculated ratios to determine the bounding case.

**Table 8-1. Bounding Cl-36 to Tc-99 Ratio from Fuel Processing Estimates.**

Radioisotope	T-Plant	B-Plant	REDOX	PUREX
Cl-36 (Ci)*	2.02E-01	1.13E-01	1.55E+00	6.81E+00
Tc-99 (Ci)*	7.63E+02	4.26E+02	6.49E+03	2.66E+04
<b>Cl-36:Tc-99 Ratio</b>	<b>2.647E-04</b>	<b>2.653E-04</b>	<b>2.388E-04</b>	<b>2.560E-04</b>

\* RPP-13489, page H-4, Table H-1

Tc-99 concentrations utilized in estimating the Cl-36 content in the composite were obtained from BBIs which represented the specific tank and original sampling date (IDMS, Queried 02/06/17, [Search All Metadata, Document Number], <http://idmsweb.rl.gov>). Decay corrections for Tc-99 were performed to represent the current concentration, however this difference was negligible due to its long half-life (2.11E+05 yr). By multiplying the Tc-99 concentration for the respective tank sample set with the Cl-36:Tc-99 bounding ratio from B-plant operations, Table 8-2 was generated, providing Cl-36 concentration estimates for each tank sample set (i.e., AN-101, AN-106, AY-101, AP-105, AP-106, and AP-107). A total, bounding pre-column composite concentration for Cl-36 was estimated at  $1.15 \times 10^{-6}$   $\mu\text{Ci/mL}$ .

Nickel-59 (Ni-59) concentrations were obtained from BBIs which represented the specific tank and original sampling date (IDMS, Queried 02/06/17, [Search All Metadata, Document Number], <http://idmsweb.rl.gov>). Decay corrections for Ni-59 were performed to represent the current concentration, however this difference was negligible due to its long half-life (7.6E+04 yr). Table 8-2 contains the Ni-59 concentration for each tank sample set and a total pre-column composite, which was estimated at  $3.44 \times 10^{-4}$   $\mu\text{Ci/mL}$ .

## RPP-RPT-59874

**Table 8-2. Estimated Tc-99, Cl-36 and Ni-59 Pre-column Composite Concentrations.**

Liquid Tank Sample Sets	Number of Samples	Total Sample Mass (g)	Density* (g/mL)	Estimated Volume (mL)	Tc-99 (µCi/mL)	Cl-36** (µCi/mL)	Ni-59 (µCi/mL)
AN-101	14	2331	1.18	1959	0.046 <sup>A</sup>	2.14E-06	3.86E-04 <sup>A</sup>
AN-101 CC8	3	67	1.18	56	0.046 <sup>B</sup>	6.11E-08	3.86E-04 <sup>B</sup>
AN-101/C-101	5	615	1.16	517	0.035 <sup>C</sup>	4.32E-07	3.86E-04 <sup>C</sup>
AN-101/C-101 CCC1	9	1793	1.16	1507	0.035 <sup>C</sup>	1.26E-06	3.86E-04 <sup>C</sup>
AN-101/C-104	1	81	1.18	68	0.036 <sup>D</sup>	5.74E-08	4.85E-04 <sup>D</sup>
AN-106/C107 RET	1	60	1.15	50	0.027 <sup>E</sup>	3.20E-08	1.63E-04 <sup>E</sup>
AN-106/C107 RET2	12	1846	1.13	1551	0.014 <sup>F</sup>	5.23E-07	4.39E-05 <sup>F</sup>
AN-106/C107 CCC1	8	1904	1.11	1600	0.018 <sup>G</sup>	6.80E-07	4.39E-05 <sup>G</sup>
AY-101	12	2025	1.06	1702	0.010 <sup>H</sup>	4.13E-07	5.35E-04 <sup>H</sup>
AP-105	7	803	1.40	675	0.152 <sup>I</sup>	2.42E-06	2.42E-04 <sup>I</sup>
AP-106	6	1231	1.21	1034	0.071 <sup>J</sup>	1.73E-06	8.25E-04 <sup>J</sup>
AP-107	4	602	1.21	506	0.086 <sup>K</sup>	1.03E-06	3.86E-04 <sup>K</sup>
<b>Composite Total</b>	<b>83</b>	<b>13359<sup>T</sup></b>	<b>---</b>	<b>11568<sup>T</sup></b>	<b>---</b>	<b>---</b>	<b>---</b>
<b>Composite Average</b>	<b>---</b>	<b>---</b>	<b>1.16</b>	<b>---</b>	<b>0.04</b>	<b>1.15E-06</b>	<b>3.44E-04</b>

A. RPP-RPT-44814, “Derivation of Best-Basis Inventory for Tank 241-AN-101 as of July 16, 2014,” using supernatant results

B. RPP-RPT-44814, “Derivation of Best-Basis Inventory for Tank 241-AN-101 as of July 1, 2013,” using supernatant results

C. RPP-RPT-44814, “Derivation of Best-Basis Inventory for Tank 241-AN-101 as of April 1, 2013,” using supernatant results

D. RPP-RPT-44814, “Derivation of Best-Basis Inventory for Tank 241-AN-101 as of July 1, 2010,” using supernatant results

E. RPP-RPT-43992, “Derivation of Best-Basis Inventory for Tank 241-AN-106 as of January 1, 2012,” using supernatant results

F. RPP-RPT-43992, “Derivation of Best-Basis Inventory for Tank 241-AN-106 as of November 7, 2012,” using supernatant results

G. RPP-RPT-43992, “Derivation of Best-Basis Inventory for Tank 241-AN-106 as of June 11, 2014,” using supernatant results

H. RPP-RPT-43979, “Derivation of Best-Basis Inventory for Tank 241-AY-101 as of July 1, 2013,” using upper supernatant results

I. RPP-RPT-43498, “Derivation of Best-Basis Inventory for Tank 241-AP-105 as of July 1, 2011,” using supernatant results

J. RPP-RPT-43493, “Derivation of Best-Basis Inventory for Tank 241-AP-106 as of October 1, 2014,” using supernatant results

K. RPP-RPT-48103, “Derivation of Best-Basis Inventory for Tank 241-AP-107 as of October 1, 2010,” using supernatant results

\* Average density calculated from the Tc-99 supernate and Ni-59 supernate split used for analysis

\*\* Cl-36 concentration was calculated by multiplying the Tc-99 concentration for each tank by the bounding Cl-36:Tc-99 ratio of  $2.653 \times 10^{-4}$

T The pre-column composite mass and volume does not take into consideration the addition of the 918 g of 19.4 M NaOH; however, it does take into consideration the sample material removed for analysis and the mass of solids formed

## RPP-RPT-59874

## 9 DISCUSSION

In support of LAWPS technology readiness, a treatability study was performed at the 222-S Laboratory whereby 12 L of archived tank supernate (with a sodium molarity of 4.7 M) was processed through SRF ion-exchange resin columns, packaged into twelve 1-L bottles and prepared for shipping off-site. Waste characterization was performed on the filtered composite solids, filtered aqueous pre-column composite, and aqueous post-column composite. A total of 109 constituents were reported for the purpose of meeting off-site shipping, PFNW receiving, and PFNW solid waste disposal requirements per the regulatory framework (LDRs, 10 CFR 61, 40 CFR 268, 49 CFR 172, 49 CFR 173, and WAC 173-303).

Of the constituents analyzed, 28 were radiological (10 of which are regulated), 46 were organic (39 of which are regulated), and 35 were inorganic (13 of which are regulated). A Cs-137 decontamination factor in excess of 250,000 was achieved. Potassium was the only other analyte in which its concentration was obviously changed due to the ion-exchange process. The post-column composite constituents identified as having a value above the respective regulatory limit included Sr-89/90, mercury, cadmium, chromium, silver, and nickel. The constituents that were measured to have a value of less than the EQL, where the EQL was higher than the regulatory limit, included lead and thallium. Methanol, 2-nitropropane, and 2-ethoxyethanol were not directly measured since WHL had no procedural method for doing so. However, a methanol concentration estimate was calculated to be greater than the regulatory limit when using Raoult's Law to relate the results of an upper-bound methanol vapor phase to a liquid phase concentration. Further investigation into the potential for 2-nitropropane and 2-ethoxyethanol in tank waste was not pursued since there is no UTS for these constituents, and RCRA regulations are only applicable if the constituent is the only F001-5 solvent present.

The organic acids accounted for ~52% of the TOC, which was reported at 1.33 g/L. The remaining organics are presumed to be chelate or chelate byproducts of HEDTA, EDTA, citrate, N-nitrosoiminodiacetate (NIDA), nitrilotriacetate (NTA), and succinate. This is based on the prior analysis of organic constituents in tanks 241-A-102, -AN-107, -AW-101, -BY-104/5/6, -C-105, -S-102, and -U-102/5/6/8 (PNNL-11738, "Organic Analysis Progress Report FY 1997").

Although some of the constituents measured from the post-column composite did not meet acceptable regulatory levels for disposal, this does not mean that the solid, cesium-depleted waste form will have the same fate. Disposal requirements for the solid waste form are dependent on the results of a TCLP. By taking into consideration the weight percent ratio of cesium-depleted solution in the Perma-Fix treated solid waste form and the 1:20 weight percent ratio of the solid waste form to solvent used in the TCLP leach, the maximum concentration of any constituent in the TCLP leach solution could be predicted.

Minor differences will be observed between the modified data tables in the body of the text and the OmniLims DSR tables in APPENDIX D. These differences are in response to QA personnel and scientist input on the use of data qualifier flags. No modifications were made to the OmniLims database.

**RPP-RPT-59874****10 EXCEPTIONS TO TEST PLAN**

Specific test plan details were modified from the original test plan, LAB-PLN-16-00003, Rev. 0 “Test Plan for the Preparation of a Supernate Archive Composite from Hanford Tanks 241-AN-101, 241-AN-106, 241-AP-105, 241-AP-106, 241-AP-107, and 241-AY-101 for Shipment to Perma Fix,”, in the revised test plan, LAB-PLN-16-00003, Rev 1.

**10.1 Precipitate Formation after Filtration**

During the removal of composite solids by filtration, the filtrate was sequentially collected into 1 L bottles. Between the time of solids removal and composite processing onto the sRF columns, a precipitate formed. These solids were observed at the bottom of each filtered composite bottle. Analysis identified the solid as natrophosphate. Future composite preparation should more closely consider individual sample chemistry to prevent super-saturation of the composite.

**10.2 Resin Bed Volume**

The sRF resin bed volume ( $\text{Na}^+$  form) used for the second set of lead/lag columns used was increased from 40 mL specified in the test plan to 60 mL. This resulted from uncertainty on the timing of breakthrough due to longer WHL turnaround times for Cs-137 analysis, voiding the ability to measure Cs-137 breakthrough in real-time. Prior estimates of the sRF decontamination factor from literature sources led us to ensure that no Cs-137 would be in the final product. The initial set of columns were replaced with those having the larger resin bed volume.

**10.3 Cesium Breakthrough Curve**

A wide span of cesium decontaminant factors have been reported in literature for Hanford tank waste and simulants. A conservative decontaminant factor value was used by WRPS in calculating the amount of resin needed for obtaining 50% cesium breakthrough. This overestimated the amount of resin needed. Second, the anticipated cesium concentrations originally took into consideration only the Cs-137 isotope. Upon BBI analysis of AP-107 and AY-101, it was determined that the Cs-137 contributed only 22-25% of the total cesium. This underestimated the amount of resin needed. To ensure achieving the primary goal of producing a cesium-depleted product, the amount of resin was deliberately increased. The surprisingly high decontamination factor observed in this work led to no significant breakthrough on the lead or lag column.

## RPP-RPT-59874

### **10.4 Additional Constituents Requested for Analysis**

The request for a nickel tracer on all radiochemistry subsamples was made on 9/26/16. The request for additional SVOA and VOA (total cresols, 2-ethoxyethanol, 2,4-dinitrotoluene, methanol, isobutyl alcohol, 2-nitropropane, trichlorofluoromethane, chloroform, and hexachloroethane) was made on 10/19/16.

### **10.5 Product Consumption for Analysis**

Regulatory limits for PFNW, WIR, and/or WAC were not properly conveyed to the 222-S Laboratory. This issue was present at the very beginning of the study, with regulatory limits being incorrect and misrepresented. Upon the investigation of regulatory limits by WRPS on 11/02/16, the regulations became clear; however, the unnecessary consumption of an additional 664 g (558 mL) of cesium-depleted product to obtain lower SVOA detection limits had already occurred. (In comparison, the initial organic, inorganic, and radiological analysis consumed a total of 371 g (312 mL) of cesium-depleted product.)

### **10.6 Dry Resin Mass Analysis Not Performed**

The dry resin mass analysis was called for in the test plan, however, it was not measured. This was due to time and budget. Also, this was not essential to the production of a cesium-depleted composite.

## **11 ACKNOWLEDGEMENTS**

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**RPP-RPT-59874**

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## 12 LESSONS LEARNED

**Table 12-1. Lessons Learned – Successes.**

Successes	Description	Impact
222-S Management and Lab Support	The 222-S management was engaged with the steps necessary to accomplish the objectives	Requirements were quickly and clearly communicated with the 222-S staff
	There was a clear structure to the points of contact	Laboratory challenges were effectively resolved in a timely manner
	The material acquisition process was greatly expedited	Materials were procured within 3-14 business days of request; The project was completed within 101 days of startup
	Tasks associated with the project were given priority	The increased visibility enhanced the reputation of the 222-S Laboratory as a capable and responsive facility
Collaborative Partnerships with Experienced Personnel	The project was a collaboration of personnel from DOE, PNNL, SRNL, ORP, PFNW, WHL, One System and 222-S	Reinforced collaborative relationships Showcased the knowledge at the 222-S Laboratory
	Personnel freely contributed their knowledge, expertise and materials (i.e., written procedures, sRF resin, and valve manifold)	Allowed for the quick turnaround on treatment process design and setup
Overcoming Obstacles	On 9/8, the archive composite's Na molarity was determined to be low (~3.3 M)	On 9/12, 600 mL of 50% NaOH was added to the archive composite to increase the Na molarity (4.7 M)
	Prior to placing a material request on 8/17, it was realized that the positive pressure, variable stroke rate metering pump to be ordered was not NRTL approved	Time restraints required an alternative pump be identified. Laboratory tests with an in-house peristaltic pump determined that the peristaltic pump would suffice
	On 8/31, it was determined that three of the anticipated archive jars (with material totaling 774g) had previously been depleted or were not in the previous archive activities	Within 1 day, contingency samples were suggested and staged for use; Permission to use contingency samples was provided by Tank Farm engineers within one day
	Manipulators went down on 9/21 and 9/22	Work remained on track. One manipulator was repaired within 6 hours and one was repaired within 24 hours; the third manipulator was not repaired for the duration of the project, however, it had little effect on the project
	At 12:00 AM on 9/21, the column manifold over-pressurized during cesium-removal, resulting in the loss of an estimated 30 mL of filtered composite	Work was halted for 1.5 hours to troubleshoot the manifold's over-pressurization, which was a result of an air gap or particulate plugging
	Longer turnaround times were needed for Cs-137 analysis	On 9/20, ion exchange columns with larger resin beds were exchanged after approximately half of the composite was processed to ensure there would be no breakthrough into the product collected

**Table 12-2. Lessons Learned – Potential Improvements.**

Potential Improvements	Description	Impact	Solution
Moving Targets	Key constituents & their regulatory limits were not clearly/accurately communicated upfront	The Test Plan needed to be revised for the incorporation of organic constituents accidentally left off of the constituent table and for those constituents requested on Oct 19th	Solidify the required deliverables upfront
		Project delayed 21 days for analysis of additional constituents requested on Oct 19th	
		Unnecessarily used 664 g of cesium-depleted product for additional analysis to achieve unneeded detection limit	
	PNFW ship date slipped	79 days after the conclusion of the project, it was identified that the 90 day clock for continued storage of the cesium-depleted material under the treatability study exclusion was going to be exceeded; Therefore, the existing 222-S sample archive procedure was modified so that the material could remain archived in accordance with WAC 173-303-071(3)(s)(v) until a shipping date was set.	Identify deliverables that have the potential to slip
Unclear Roles and Responsibilities of Organizations Involved	It was unclear on who was responsible for providing critical project information	Time was wasted waiting for answers and then again when the answers had to be searched out	Clarify organizations' responsibilities and accountabilities upfront; Within One System led projects, the 222-S Lab should have representation in chemistry-related project planning and execution due to their scientific expertise and low cost
Request for information was not adequately addressed or went unanswered			
Aging 222-S Facility	An HVAC failure was identified in the 11A hot cell facility, restricting work and normal operations prior to project startup	Ventilation was restored on 8/18/16, 13 days prior to project start at the 222-S Laboratory	Priority in retaining the unique capabilities of the 11A facility should be supported monetarily for the success of future projects.
	At 12:30 AM on 9/21/16 in hot cell 11A1B North, during column feed processing, 1 manipulator lost all functionality and 1 manipulator lost 75% functionality.	Feed processing was able to continue with one functioning manipulator. One of the down manipulators was repaired within 6 hours, and the other manipulator was repaired within 24 hours.	
	At 12:00 AM on 9/22/16 in hot cell 11A6, where the analytical balance was located, 1 manipulator lost all functionality.	Work was able to continue with one functioning manipulator. The down manipulator remained out of service for the remainder of the project.	

## RPP-RPT-59874

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**RPP-RPT-59874**

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RPP-RPT-59874

**APPENDIX A.**

**TANK ARCHIVE SUPERNATE SAMPLES USED AND TREATABILITY RECORDS**

(9 pages, including cover sheet)

## RPP-RPT-59874

## Appendix A

**Table A-1. Tank Archive Supernate Samples Used in Composite. (2 pages)**

Tank	Jar	Net weight Difference (%)	Available Mass (g)	Mass Used (g)	Matrix
AN-101	20171.0	-0.6	10.4	4.2	CL
	20158.0	0.0	60.8	59.4	CL
	20658.0	-0.1	97.7	95.5	CL
	20640.0	-0.5	29.3	19.2	CL
	20614.0	-0.1	24.7	23.9	CL
	20196.0	0.0	151.6	150.1	CL
	20678.0	0.0	111.6	99.2	Comp
	20657.0	0.0	156.1	0.0	Comp
	1AN-14-01	0.0	320.6	152.7	GL
	1AN-14-01A	0.0	325.3	318.3	GL
	1AN-14-02	0.0	227.5	322.6	GL
	1AN-14-03	0.0	318.1	224.9	GL
	1AN-14-03A	0.0	230.3	315.6	GL
	1AN-14-04	0.0	318.6	228.7	GL
	1AN-14-04A	-0.2	29.5	317.0	GL
AN-101 CC8	20828.0	-0.1	36.9	28.0	GL
	20825.0	-0.8	12.5	35.4	GL
	20827.0	0.0	46.2	3.1	GL
AN-101/241-C-101	20856.0	0.0	68.1	44.3	GL
	1AN-13-01	0.0	282.0	65.8	GL
	1AN-13-01A	0.0	149.9	279.8	GL
	1AN-13-01DUP	-0.1	81.5	147.4	GL
	20855.0	0.0	256.8	78.2	L
AN-101/241-C-101 CCC1	1AN-13-02	0.0	278.7	254.1	GL
	1AN-13-03A	0.0	107.9	276.0	GL
	1AN-13-03	0.0	131.1	105.6	GL
	1AN-13-04	0.0	257.7	130.0	GL
	1AN-13-04A	0.0	118.4	255.1	GL
	1AN-13-05	0.0	259.9	116.4	GL
	1AN-13-05A	0.0	124.0	257.3	GL
	1AN-13-06	0.0	280.0	122.1	GL
	1AN-13-06A	0.0	83.5	276.8	GL
AN-101/241-C-104	20510.0	0.0	119.8	80.7	GL
AN-106	20652.0	-0.1	62.1	117.3	GL
	20523.0	0.0	98.2	60.0	GL
	6AN-12-01	0.0	108.4	96.2	GL
	6AN-12-01DUP	0.0	252.4	105.7	GL
	6AN-12-03A	0.0	69.9	246.4	GL
	6AN-12-03	-0.1	53.9	68.5	GL
	6AN-12-04	-0.1	30.1	50.7	GL
	6AN-12-04DUP	0.0	107.6	28.1	GL
	6AN-12-05	0.0	260.9	104.3	GL
	6AN-12-05A	0.0	265.2	254.7	GL
	6AN-12-05B	#N/A	128.8	259.2	GL
	6AN-12-06A	0.0	257.5	260.0	GL
	6AN-12-06B	0.0	156.6	254.7	GL
	6AN-14-01	0.0	308.8	154.3	GL
	6AN-14-01A	0.0	306.1	307.6	GL
	6AN-14-01B	0.0	306.7	304.1	GL

## RPP-RPT-59874

## Appendix A

**Table A-1. Tank Archive Supernate Samples Used in Composite. (2 pages)**

Tank	Jar	Net weight Difference (%)	Available Mass (g)	Mass Used (g)	Matrix
AN-106/241-C-107 RET	6AN-14-02	0.0	213.1	305.1	GL
	6AN-14-03	0.0	208.8	211.7	GL
	6AN-14-04	0.0	210.6	207.3	GL
	6AN-14-04DUP	0.0	206.7	209.3	GL
	6AN-14-05	-0.4	125.6	204.4	GL
	6AN-11-01DUP	-0.1	318.5	0.0	GL
	6AN-11-05	-0.2	208.7	0.0	GL
	6AN-11-06	-0.1	319.8	0.0	GL
AY-101	1AY-13-01	-0.2	187.5	81.1	GL
	1AY-13-01A	-0.1	328.0	273.0	GL
	1AY-13-01DUP	-0.2	186.5	160.5	GL
	1AY-13-01B	-0.2	187.1	274.6	GL
	1AY-13-02	-0.2	194.4	145.0	GL
	1AY-13-02A	-0.2	189.3	282.3	GL
	1AY-13-04	-0.4	127.3	142.3	GL
	1AY-13-05	-0.2	196.0	142.4	GL
	1AY-13-06	0.0	158.0	150.1	GL
	1AY-13-07	0.0	122.6	144.4	GL
	1AY-13-08	0.0	128.8	80.7	GL
	1AY-13-09	0.0	126.7	148.9	GL
AP-105	5AP-11-01	-0.1	60.6	152.9	GL
	5AP-11-03	0.0	123.3	117.9	GL
	5AP-11-03DUP	0.0	116.8	123.6	GL
	5AP-11-04	0.0	327.3	122.1	GL
	5AP-11-05	0.0	83.5	56.3	GL
	5AP-11-06	0.0	211.3	118.9	GL
	5AP-11-07	0.0	218.7	111.5	GL
AP-106	6AP-14-01	0.0	221.1	325.1	Liq
	6AP-14-02DUP	0.0	179.9	80.7	Liq
	6AP-14-03	0.0	136.4	210.1	Liq
	6AP-14-04	0.0	104.3	217.2	Liq
	6AP-14-05	-0.5	285.0	219.5	Liq
	6AP-14-02	0.0	149.4	178.3	Liq
AP-107	7AP-10-01		14433.7	136.211	GL
	7AP-10-03A			107.697	GL
	7AP-10-04A			204.905	GL
	7AP-10-02A			153.357	GL
			<b>Total:</b>	13358.519	

\* Measure of weight loss by evaporation during storage (current net weight minus original net weight divided by original net weight).

CL = centrifuged liquid

Comp = composite

Liq = liquid

GL = grab liquid

RPP-RPT-59874

## Appendix A

**Table A-2. RCRA Treatability Study Record.**

1 Sample(s) must be labeled "Hazardous Waste" and major risk.

2Types of actions: Waste received, Test started, Waste used in Test, Test completed.

3 When test is completed, enter in the Comments section the final disposition of the unused sample portion and the sample residues.

A-6002-653 (06/05)

## RPP-RPT-59874

## Appendix A

**Table A-3. Test Bed Filtrate Bottle Records.**

<b>Product Bottle</b>	<b>Empty (g)</b>	<b>Full (g)</b>	<b>Product (g)</b>	<b>Date</b>	<b>Notes</b>
S16R000331	104.297	1208.73	1104.43	10/5/16	Material removed for additional SVOA analysis.
	104.297	545.08	440.78	11/2/16	Mass after additional SVOA analysis.
S16R000332	104.936	1259.37	1154.43	10/5/16	
S16R000333	104.592	1260.61	1156.02	10/5/16	
S16R000334	105.078	1260.41	1155.33	10/5/16	
S16R000335	104.446	1196.40	1091.95	10/5/16	
S16R000336	104.758	1197.89	1093.13	10/5/16	
S16R000337	104.780	1228.65	1123.87	10/5/16	
S16R000338	104.890	1205.64	1100.75	10/5/16	
S16R000339	104.851	1256.19	1151.34	10/5/16	
S16R000340	104.607	1197.38	1092.77	10/5/16	
S16R000341	105.028	1193.72	1088.69	10/5/16	
S16R000413	105.758	1311.21	1205.45	10/6/16	

**Total:** 12854.53 g

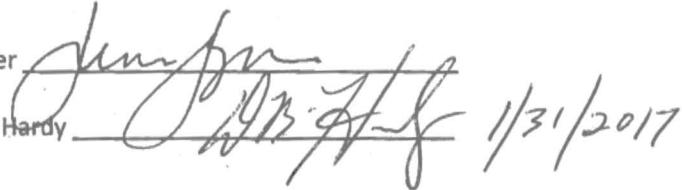
10.80 L

2.85 gal

SVOA = Semi-volatile organic analysis

## RPP-RPT-59874

## Appendix A

RAM ID Number: WRPS-17-001, Rev 0**Title:** Radioactive Test Bed Treatability Study Compliance Strategy at 222-S Laboratory**Key Words:** RCRA, Treatability Study, 222-S**Author/Date:** Eric Van Mason, January 31, 2017**Management Concurrence/Date:** Jessica Joyner**Facility Management Concurrence/Date:** Don Hardy**PURPOSE**

The purpose of this RAM is to document an environmental compliance strategy for the Test Bed Project work at 222-S and establish a basis for 222-S to continue storing treatability study material under the treatability study sample exclusion in WAC 173-303-071(3)(s).

**PROJECT SCOPE**

The Test Bed Project is planned as a series of waste treatment activities using Hanford tank waste samples with the ultimate goal of evaluating the feasibility of off-site disposal. Two stages of treatment are planned to occur before disposal off-site.

The waste used by this Project is decanted supernate waste from six tanks that was already present in 222-S analytical sample archive. The first waste treatment has occurred at the 222-S Laboratory, consisting of solids filtration and ion exchange to remove cesium. Concurrent with these activities, USDOE developed and approved a Waste Incidental to Reprocessing (WIR) determination under DOE O 435.1 which reclassified the treated tank sample from High Level Waste to Low Level Waste (Attachment 1).

Following pretreatment at the 222-S Laboratory (completed), the waste is currently planned for further treatment at Perma-Fix Northwest Richland, Inc. (PFNW) where it will be solidified into a grout matrix that will meet Land Disposal Restrictions and disposal facility acceptance criteria. USDOE plans for final disposal of the treated and solidified waste at the Waste Control Specialists Federal Waste Disposal Facility (WCS FWF), in Andrews, Texas.

**REQUIREMENTS**

Prior to conducting any work, the activities at 222-S were identified as a Resource Conservation and Recovery Act Treatability Study using the 222-S determination process documented in Attachment 2 using Washington State regulations. Designation of the treatment activities as a Treatability Study means there is basis to qualify for the regulatory 'exclusions' in WAC 173-303-071(3)(r) and (s)<sup>1</sup>. These regulatory exclusions allow for generators of a waste sample to be used in a Treatability Study, and laboratories conducting a Treatability study, to manage certain materials as not fully regulated under WAC 173-303, as long as the conditions of the exclusion are complied with.

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<sup>1</sup>WAC 173-303-071(3)(r) and (s) are substantively similar to 40 CFR 261.4(e) and (f).

## RPP-RPT-59874

## Appendix A

The following is a summary of key requirements relevant to use of Hanford tank waste in Treatability Studies using sample inventory already located at 222-S:

- Ecology must be notified in writing 45 days before conducting treatability studies. [WAC 173-303-071(3)(s)(i)] (*One-time notification covering the Test Bed treatability study was provided in 1988 (Reference 1)*)
- 222-S may not initiate treatment on more than 250 kg of “as received” tank waste in any single day. [WAC 173-303-(3)(s)(iii)]
- 222-S may not store more than 1,000 kg of “as received” tank waste that is used in treatability studies [WAC 173-303-071(3)(s)(iv)]
- 222-S may not store treatability study waste/materials under the exclusion for more than 90 days after the treatability study is completed, or, 1 year from the date the treatability study was initiated, whichever comes first. [WAC 173-303-071(3)(s)(v)]
- 222-S may archive up to 500 kg of “treated material” resulting from the treatability study for future evaluation up to 5 years from the date the tank waste was initially “received”. [WAC 173-303-071(3)(s)(v)]
- Any treatment “residue”, unused sample, or other wastes generated during performance of the treatability study must be managed under the full scope of WAC 173-303 if they designate as Hazardous or Mixed waste. [WAC 173-303-071(3)(s)(x)]
- While being held at the laboratory, each container must be labeled with the words “hazardous waste” and the major risk. [WAC 173-303-071(3)(s)(xiii)]
- Reporting Requirements Summary: All relevant dates, quantities, shipping information, material disposition, etc. must be kept as a record for 3 years and provided in an annual report as specified in WAC 173-303-071(3)(r) and (s).

### **222-S TREATABILITY STUDY STRATEGY**

Over the broader scope of the Test Bed Project, two separate treatability studies are occurring. The 222-S treatability study involves the filtration and ion exchange. Further treatment by solidification at PFNW is a separate treatability study. 222-S is both the “generator” and “laboratory” for the waste sample used in the first treatability study. 222-S will be the “generator” of the sample (222-S treated material) used in the second study. It is assumed no mass limits will be exceeded as approximately 3 gallons or 14 kg of tank waste is used.

## RPP-RPT-59874

## Appendix A

The recordkeeping responsibilities relevant to the role 222-S has are being followed. This is done primarily by using the RCRA Treatability Study Record (form A-6002-653), and by responding to calls for information from MSA for the annual Hanford Site treatability study report.

Various material streams were managed while conducting the 222-S treatability study. These are categorized and management methods summarized below:

- **Tank waste sample or excess sample:** The treatability study began when tank waste from analytical sample inventory was composited (9/7/16). All sample was used in the treatment process.
- **Treatment Residues:** Treatment residues include IX column eluants, any rinses, IX media, and filtered solids. These materials are managed as fully regulated mixed waste using existing 222-S waste management procedures. A small portion of some of these residues were removed from the treatability study process for analysis and used under the analytical sample exclusion.
- **Waste:** Waste was generated during the treatability study. This includes materials such as glassware, equipment, consumables, etc. Wastes are being managed according to existing 222-S waste management procedures.
- **Treated Material:** This material is the decontaminated liquid remaining after filtration and ion exchange was performed. Small portions of the treated material was removed from the treatability study process for analysis under the analytical sample exclusion.

The flow chart provided in Attachment 3 depicts how these streams were managed.

## 222-S POST TREATABILITY STUDY ARCHIVE STRATEGY

The 222-S study was completed on 11/7/16, triggering a 90 day clock for continued storage under the treatability study exclusion which ends 2/5/17. At this time, further treatment at PFNW is delayed as USDOE reevaluates plans for future treatment or evaluation of the 222-S treated material. To continue storing the treated material under the exclusion, it must be archived in accordance with WAC 173-303-071(3)(s)(v).

### Archive Administration

The existing 222-S sample archive procedure, ATS-310, Section 1.15 has been modified to include in its scope the ability to archive treated material from a treatability study for future evaluation for up to 5 years from the date a treatability study sample is received. Archiving of treated waste from this study is allowed until 9/7/2021. The archive procedure requires a tickler be established to track the 5 year time limit. The archive procedure also requires an annual evaluation of whether archived items meet criteria for continued archive. If the archived treated material is no longer needed "for future evaluation", it will be disposed with concurrence from the owner of the treated material. 222-S maintains an archive database that will note that this material is associated with the Test Bed treatability study.

### Storage Conditions

The treated material is packaged within a 30 gallon transportation drum. The drum is labeled with the words "hazardous waste" and a major risk ("corrosive") per WAC 173-303-071(3)(s)(xiii). The drum

**RPP-RPT-59874****Appendix A**

will be stored as material excluded from full regulation under WAC 173-303 in the room 2B treatment storage and disposal (TSD) area. The 2B TSD area is behind a locked gate at all times and can only be accessed by authorized personnel. The drum will be tracked in the TSD inventory with a notation that it is Test Bed “excluded treatability study material” and the drum will be labeled to this effect.

**Alternatives**

If at any time the archived Test Bed material cannot meet the criteria for continued archiving, it should be submitted for addition to the 219-S tank system using existing 222-S waste procedures.

**REFERENCES**

1. Letter from R.D. Izatt, T.D. Chikalla, R.E. Lerch to T. Eaton, RE: Notification of Intent to Perform Treatability Tests Exclusive of Resource Conservation and Recovery Act (RCRA) Subtitle (53 Federal Register 27290-27302), November 9, 1988.
2. DOE/ORP Tank Waste Test Bed, Rev. 1, Waste Incidental to Reprocessing Evaluation – Test Samples of Treated, Low-Activity Waste from Hanford Tanks for Off-site Disposal, October 2016.
3. Washington Administrative Code 173-303, Dangerous Waste Regulations.

RPP-RPT-59874

**APPENDIX B.**

**SAMPLE BREAKDOWN DIAGRAM**

(9 pages, including cover sheet)

## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20162592

- Contains Cs-137
- Processed for Cs-137 removal

<u>Jar ID</u>	<u>Customer ID</u>
20171	1AN-12-04A
20158	1AN-12-05A
20658	1AN-12-04B
20640	1AN-12-05B
20614	1AN-12-12
20196	1AN-12-12A
20678	Composite 1
1AN-14-01	1AN-14-01
1AN-14-01A	1AN-14-01A
1AN-14-02	1AN-14-02
1AN-14-03	1AN-14-03
1AN-14-03A	1AN-14-03A
1AN-14-04	1AN-14-04
1AN-14-04A	1AN-14-04A
20828	Composite 2
20825	Composite 3
20827	Composite 6
20856	1AN-13-07A
1AN-13-01	1AN-13-01
1AN-13-01A	1AN-13-01A
1AN-13-01DUP	1AN-13-01DUP
20855	Composite 4
1AN-13-02	1AN-13-02
1AN-13-03A	1AN-13-03A
1AN-13-03	1AN-13-03
1AN-13-04	1AN-13-04
1AN-13-04A	1AN-13-04A

<u>Jar ID</u>	<u>Customer ID</u>
1AN-13-05	1AN-13-05
1AN-13-05A	1AN-13-05A
1AN-13-06	1AN-13-06
1AN-13-06A	1AN-13-06A
20510	1AN-13-11
20652	6AN-12-02
20523	Composite 5
6AN-12-01	6AN-12-01
6AN-12-01DUP	6AN-12-01DUP
6AN-12-03A	6AN-12-03A
6AN-12-03	6AN-12-03
6AN-12-04	6AN-12-04
6AN-12-04DUP	6AN-12-04DUP
6AN-12-05	6AN-12-05
6AN-12-05A	6AN-12-05A
6AN-12-05B	6AN-12-05B
6AN-12-06	6AN-12-06
6AN-12-06A	6AN-12-06A
6AN-12-06B	6AN-12-06B
6AN-14-01	6AN-14-01
6AN-14-01A	6AN-14-01A
6AN-14-01B	6AN-14-01B
6AN-14-02	6AN-14-02
6AN-14-03	6AN-14-03
6AN-14-04	6AN-14-04
6AN-14-04DUP	6AN-14-04DUP
6AN-14-05	6AN-14-05
1AY-13-01	1AY-13-01

<u>Jar ID</u>	<u>Customer ID</u>
1AY-13-01A	1AY-13-01A
1AY-13-01DUP	1AY-13-01DUP
1AY-13-01B	1AY-13-01B
1AY-13-02	1AY-13-02
1AY-13-02A	1AY-13-02A
1AY-13-04	1AY-13-04
1AY-13-05	1AY-13-05
1AY-13-06	1AY-13-06
1AY-13-07	1AY-13-07
1AY-13-08	1AY-13-08
1AY-13-09	1AY-13-09
5AP-11-03	5AP-11-03
5AP-11-03DUP	5AP-11-03DUP
5AP-11-04	5AP-11-04
5AP-11-05	5AP-11-05
5AP-11-06	5AP-11-06
5AP-11-07	5AP-11-07
6AP-14-01	6AP-14-01
6AP-14-02DUP	6AP-14-02DUP
6AP-14-03	6AP-14-03
6AP-14-04	6AP-14-04
6AP-14-05	6AP-14-05
6AP-14-02	6AP-14-02
7AP-10-01	7AP-10-01
7AP-10-03A	7AP-10-03A
7AP-10-04A	7AP-10-04A
7AP-10-02A	7AP-10-02A

## Test Bed Composite-Unfilt



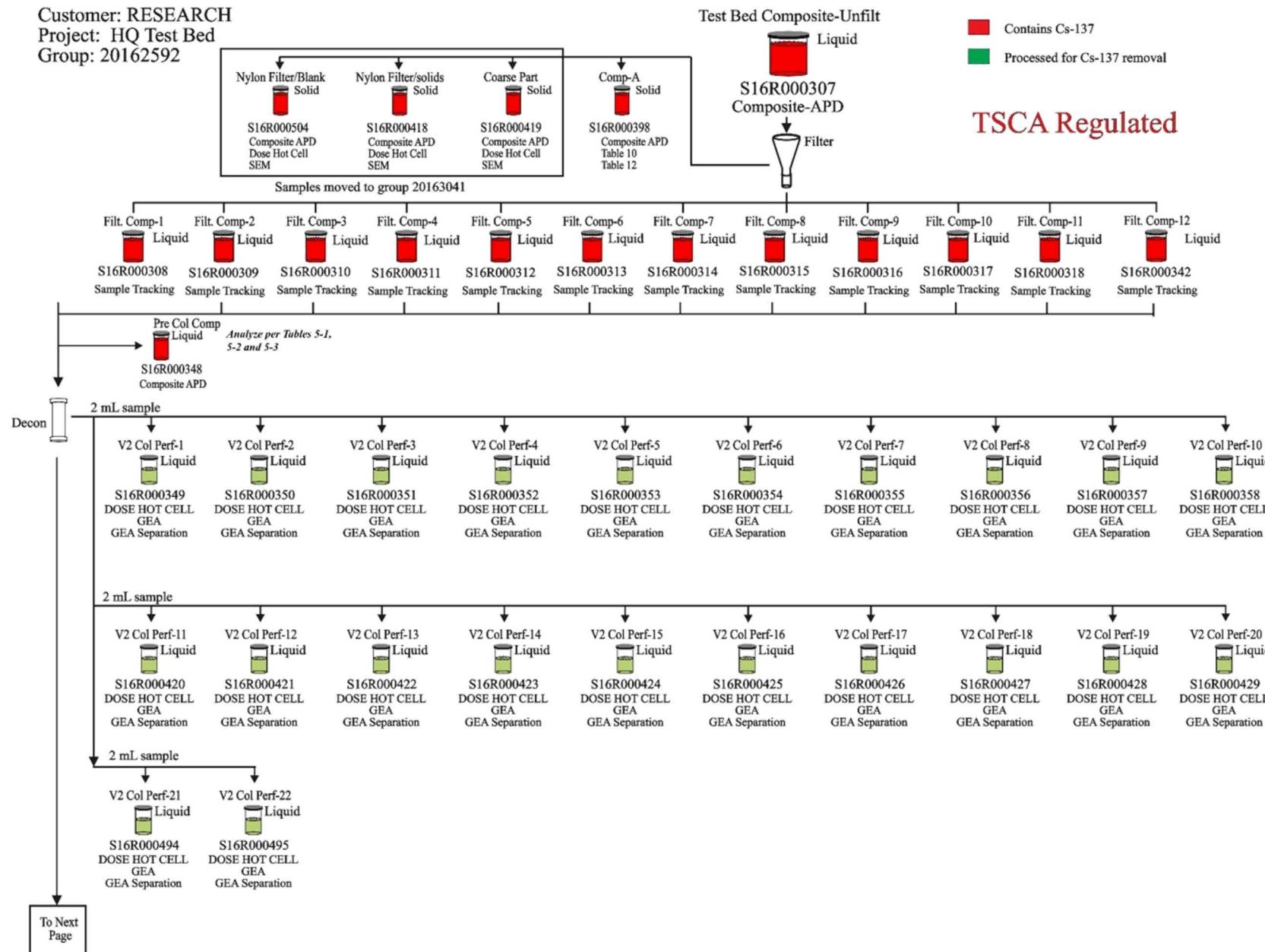
S16R000307  
 Composite-APD

Composite 1: 1AN-12-01 (120.6g), 1AN-12-02A (120.1g), 1AN-12-03 (120.6g),  
 Composite 2: 1AY-13-01 (21.2g), 1AY-13-02 (20.4), 1AY-13-04 (21.1g), 1AY-13-05 (21.4g), 1AY-13-06 (21.0g).  
 Composite 3: 1AY-13-07 (27.4g), 1AY-13-08 (28.8).  
 Composite 4: 20mL ea of 1AN-13-01, 02, 03, 04, 05, 06.  
 Composite 5: 6AN-11-5A (146.7g), 6AN-11-6A (124.6g).  
 Composite 6: 1AY-13-09 (29g), 1AY-13-10 (29.4g)

## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20162592



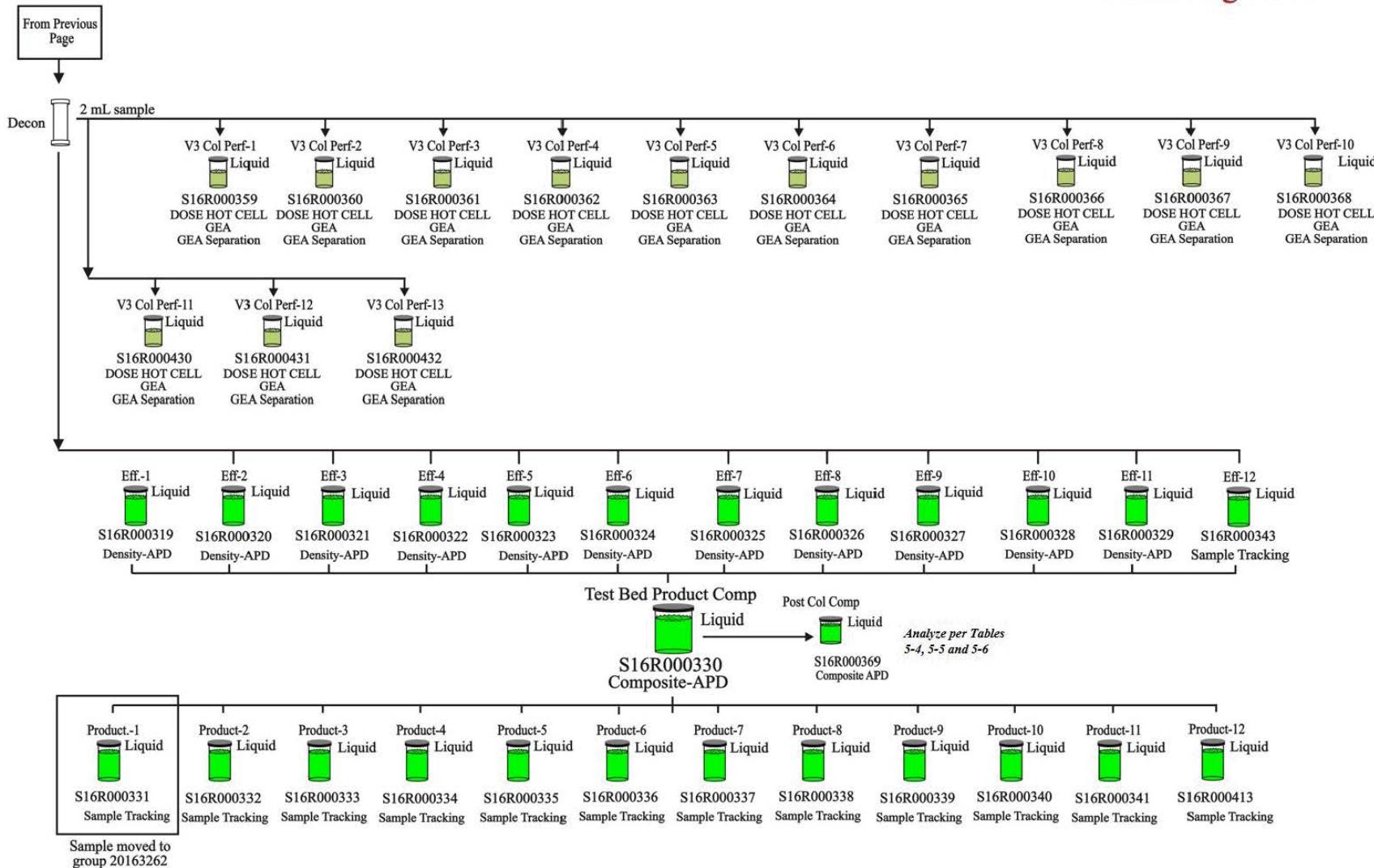
## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20162592

## Test Bed Product Composite

TSCA Regulated

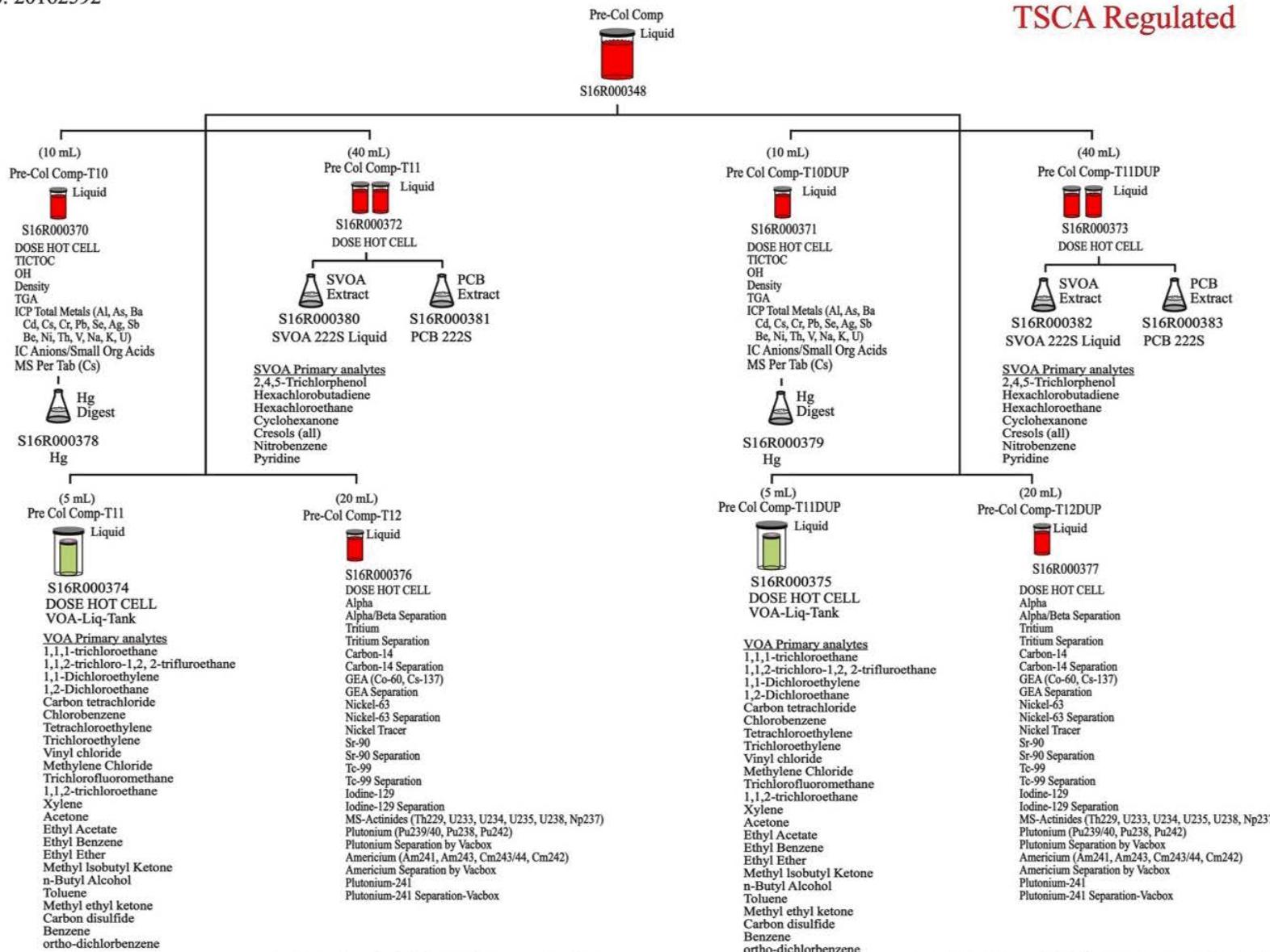


## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20162592

## Pre-Col Comp Analyses



## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20162967

## Post-Col Comp Analyses



S16R000369

## TSCA Regulated

(10 mL)  
 Post-Col Comp-T10  
  
 Liquid  
 S16R000384  
 DOSE HOT CELL  
 TICTOC  
 OH  
 Density  
 TGA  
 ICP Total Metals (Al, As, Ba  
 Cd, Cs, Cr, Pb, Se, Ag, Sb  
 Be, Ni, Th, V, Na, K, U)  
 IC Anions/Small Org Acids  
 MS Per Tab (Cs)  
  
 Hg Digest  
 S16R000386  
 Hg

(40 mL)  
 Post Col Comp-T11  
  
 Liquid  
 S16R000388  
 DOSE HOT CELL  
  
 SVOA Extract  
 S16R000390  
 SVOA 222S Liquid  
  
 PCB Extract  
 S16R000392  
 PCB 222S

(5 mL)  
 Post Col Comp-T11  
  
 Liquid  
 S16R000394  
 DOSE HOT CELL  
 VOA-Liq-Tank  
  
 VOA Primary analytes  
 1,1,1-trichloroethane  
 1,1,2-trichloro-1,2, 2-trifluoroethane  
 1,1-Dichloroethylene  
 1,2-Dichloroethane  
 Carbon tetrachloride  
 Chlorobenzene  
 Tetrachloroethylene  
 Trichloroethylene  
 Vinyl chloride  
 Methylene Chloride  
 Trichlorofluoromethane  
 1,1,2-trichloroethane  
 Xylene  
 Acetone  
 Ethyl Acetate  
 Ethyl Benzene  
 Ethyl Ether  
 Methyl Isobutyl Ketone  
 n-Butyl Alcohol  
 Toluene  
 Methyl ethyl ketone  
 Carbon disulfide  
 Benzene  
 ortho-dichlorobenzene  
 Chloroform  
 Hexachloroethane  
 2-Nitropropane

(20 mL)  
 Post-Col Comp-T12  
  
 Liquid  
 S16R000396  
 DOSE HOT CELL  
 Alpha  
 Alpha/Beta Separation  
 Tritium  
 Tritium Separation  
 Carbon-14  
 Carbon-14 Separation  
 GEA (Co-60, Cs-137)  
 GEA Separation  
 Nickel-63  
 Nickel-63 Separation  
 Nickel Tracer  
 Sr-90  
 Sr-90 Separation  
 Tc-99  
 Tc-99 Separation  
 Iodine-129  
 Iodine-129 Separation  
 MS-Actinides (Th229, U233, U234, U235, U238, Np237)  
 Plutonium (Pu239/40, Pu238, Pu242)  
 Plutonium Separation by Vacbox  
 Americium (Am241, Am243, Cm243/44, Cm242)  
 Americium Separation by Vacbox  
 Plutonium-241  
 Plutonium-241 Separation-Vacbox  
 Total Activity  
 Total Activity Separation

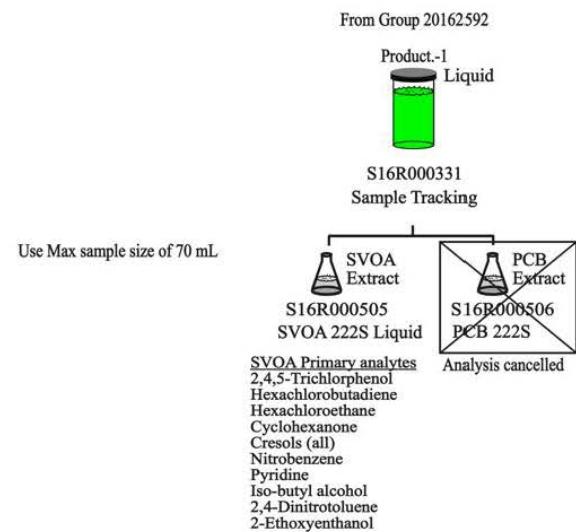
Nb-94, Sn-116, Ra-226 will be based on process knowledge.

RPP-RPT-59874  
Appendix B

Customer: RESEARCH  
Project: HQ Test Bed  
Group: 20163262

Post-Col Comp Re-analyses

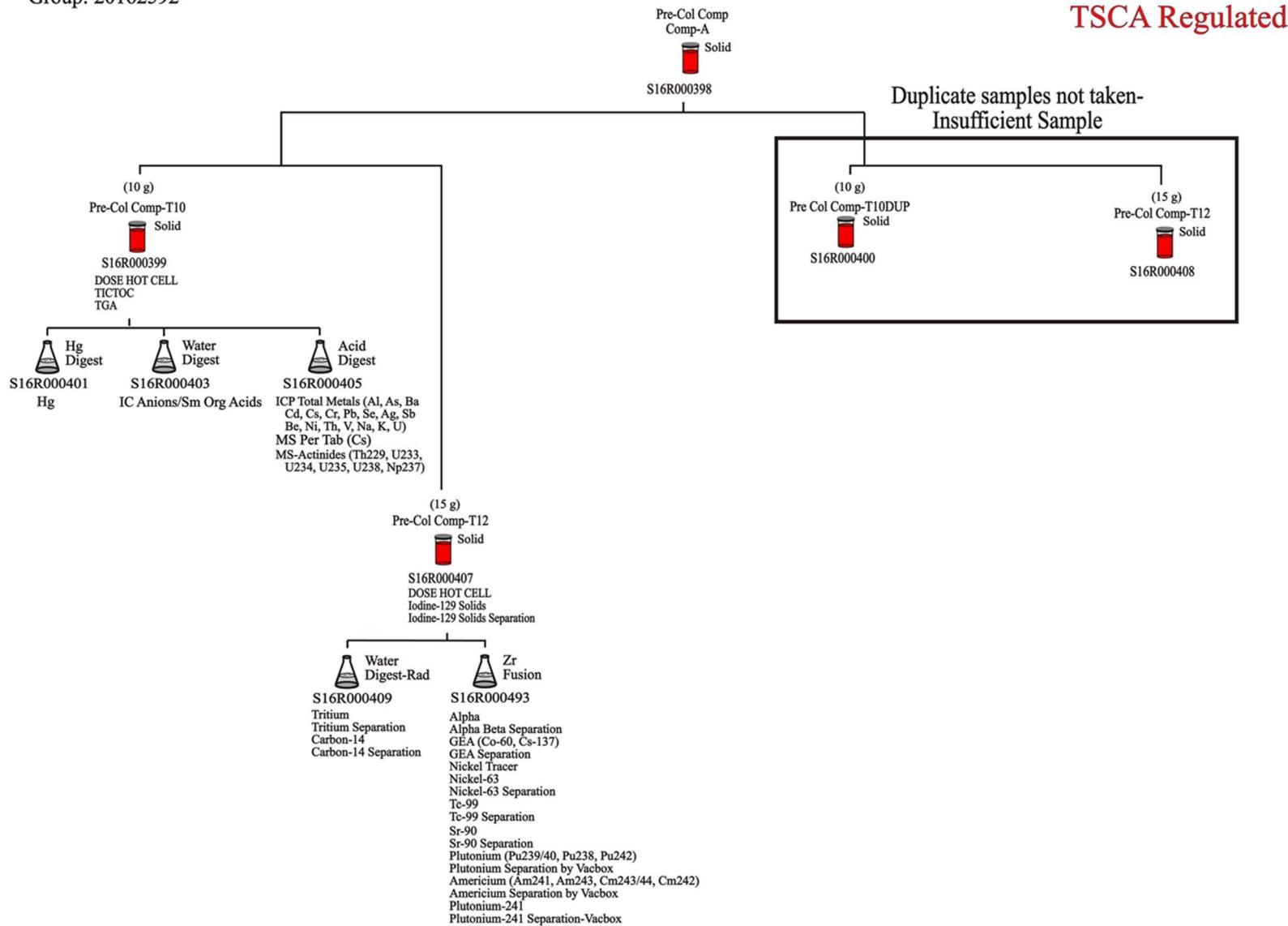
TSCA Regulated



PPR-RPT-59874  
Appendix B

Customer: RESEARCH  
Project: HQ Test Bed  
Group: 20162592

## Pre-Col Comp Analyses



Nb-94, Sn-126, Ra-226 will be based on process knowledge.

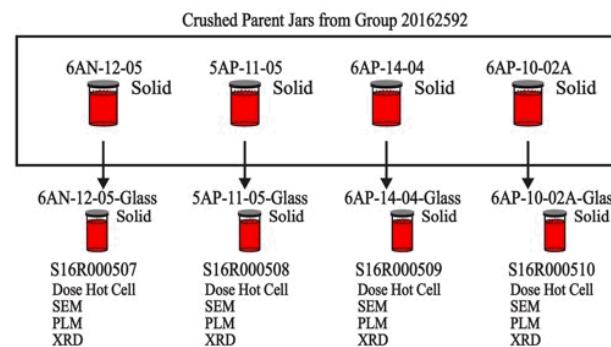
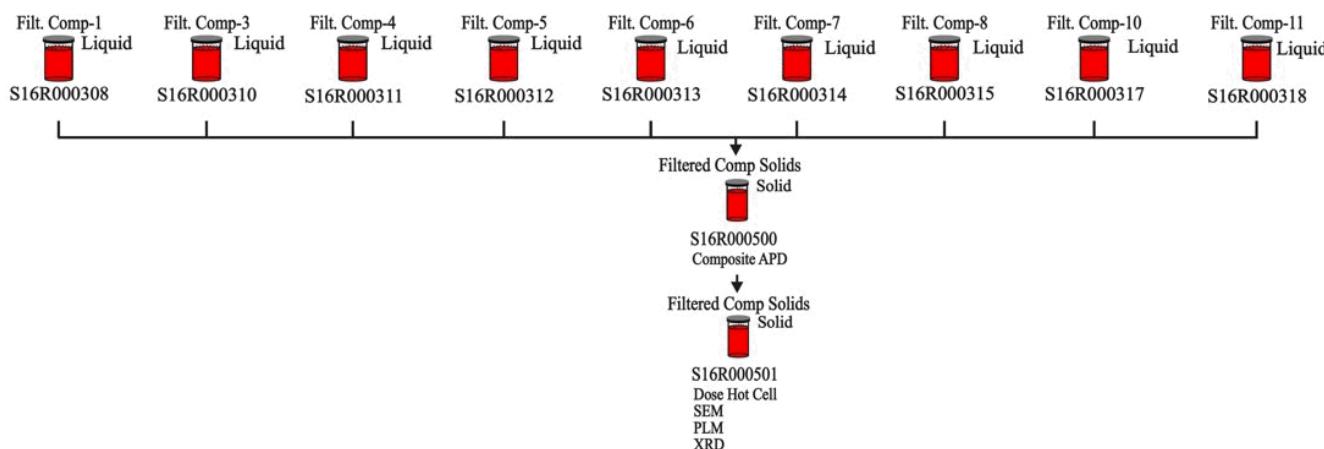
## RPP-RPT-59874

## Appendix B

Customer: RESEARCH  
 Project: HQ Test Bed  
 Group: 20163041

## Pre-Col Comp Solids Analyses

TSCA Regulated



APD = analytical process development group  
 GEA = gamma energy analysis  
 IC = ion chromatography  
 ICP = inductively coupled plasma spectroscopy  
 MS = mass spectroscopy  
 TGA = thermogravimetric analysis  
 TIC/TOC = total inorganic carbon/total organic carbon  
 TSCA = Toxic Substances Control Act of 1976  
 VOA = volatile organic analysis

RPP-RPT-59874

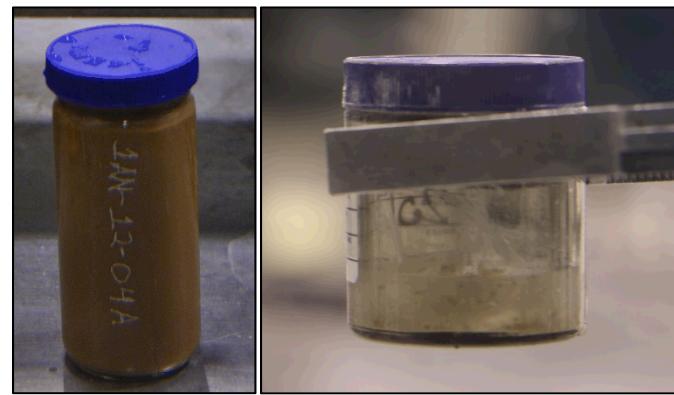
**APPENDIX C.**

**TANK WASTE ARCHIVE PHOTOS**

(29 pages, including cover sheet)

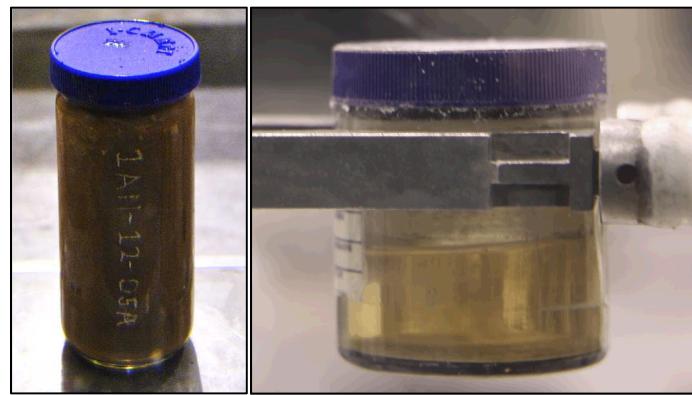
## RPP-RPT-59874

## Appendix C



As received.

As found.

**Figure C-1. Tank 241-AN-101; Jar ID 20171; Customer Jar ID 1AN-12-04A.**

As received.

As found.

**Figure C-2. Tank 241-AN-101; Jar ID 20158; Customer Jar ID 1AN-12-05A.**

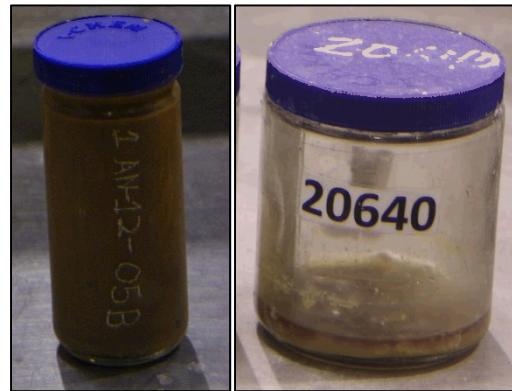
As received.

As found.

**Figure C- 3. Tank 241-AN-101; Jar ID 20658; Customer Jar ID 1AN-12-04B.**

## RPP-RPT-59874

## Appendix C



As received.

As found.

**Figure C-4. Tank 241-AN-101; Jar ID 20640; Customer Jar ID 1AN-12-05B.**

As received.

As found.

**Figure C-5. Tank 241-AN-101; Jar ID 20614 (A and B).**

As received.

As found.

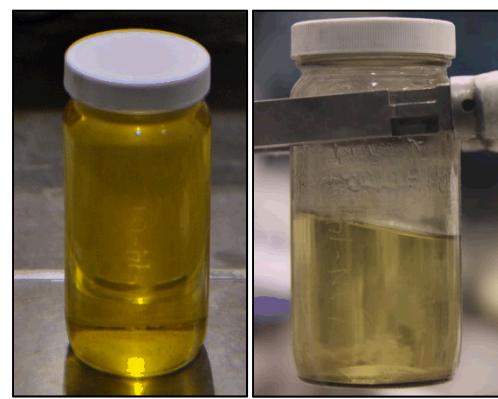
**Figure C-6. Tank 241-AN-101; Jar ID 20196 (A & B); Customer Jar ID 1AN-12-12A.**

RPP-RPT-59874  
Appendix C

As found.

**Figure C-7. Tank 241-AN-101; Jar ID 20678.**

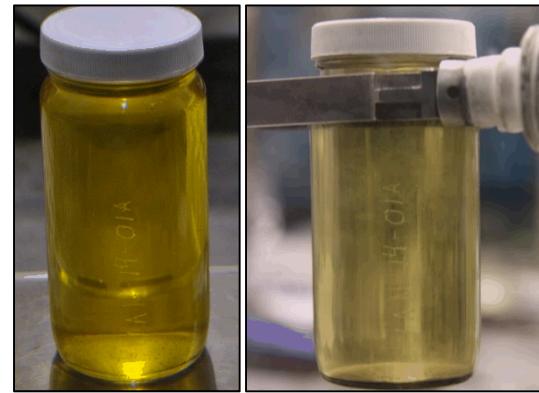
As found

**Figure C- 8. Tank 241-AN-101; Jar ID 20657.**

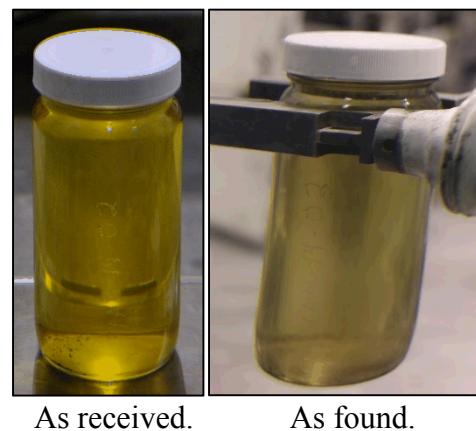
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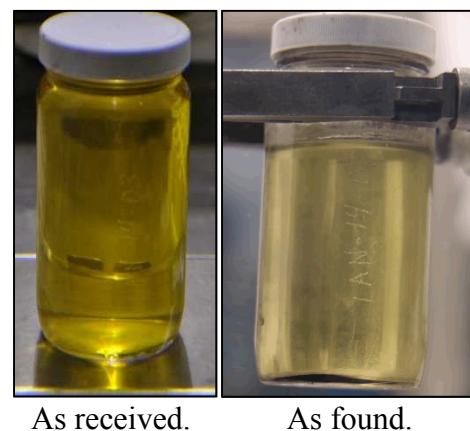
**Figure C-9. Tank 241-AN-101; Jar ID 1AN-14-01; Customer Jar ID 1AN-14-01.**

RPP-RPT-59874  
Appendix C

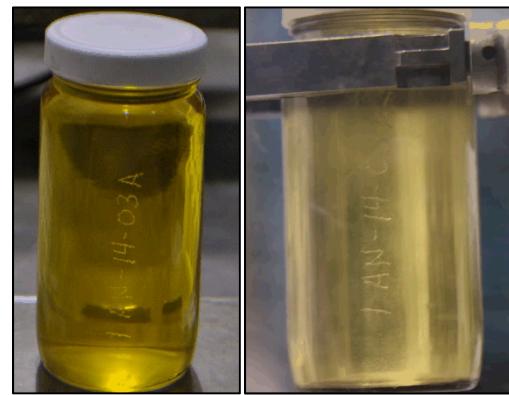
**Figure C-10. Tank 241-AN-101; Jar ID 1AN-14-01A; Customer Jar ID 1AN-14-01A.**



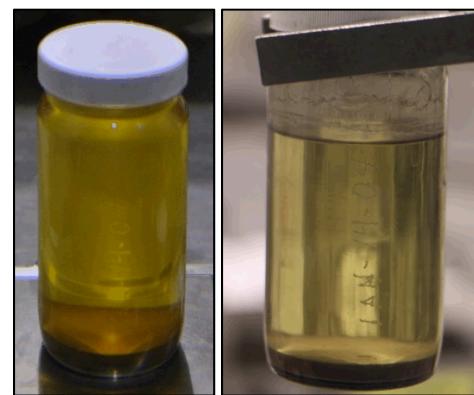
**Figure C-11. Tank 241-AN-101; Jar ID 1AN-14-02; Customer Jar ID 1AN-14-02.**



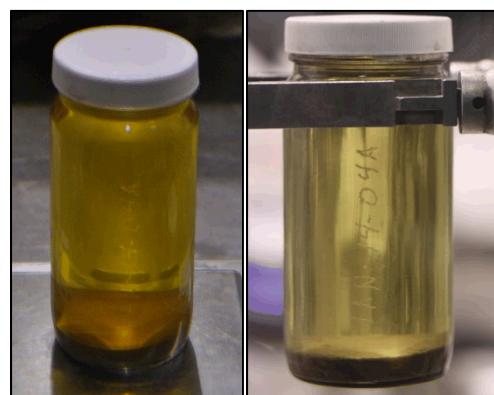
**Figure C-12. Tank 241-AN-101; Jar ID 1AN-14-03; Customer Jar ID 1AN-14-03.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-13. Tank 241-AN-101; Jar ID 1AN-14-03A; Customer Jar ID 1AN-14-03A.**

As received. As found.

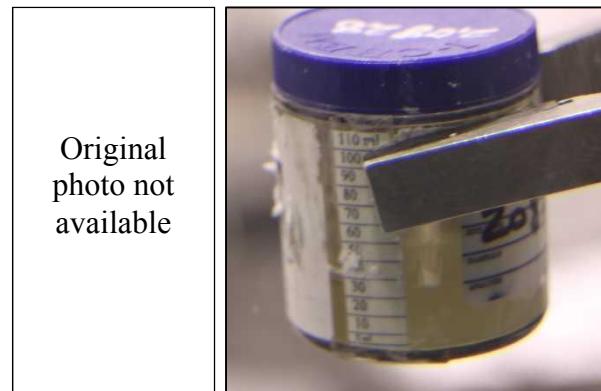
**Figure C-14. Tank 241-AN-101; Jar ID 1AN-14-04; Customer Jar ID 1AN-14-04.**

As received. As found.

**Figure C-15. Tank 241-AN-101; Jar ID 1AN-14-04A; Customer Jar ID 1AN-14-04A.**

## RPP-RPT-59874

## Appendix C



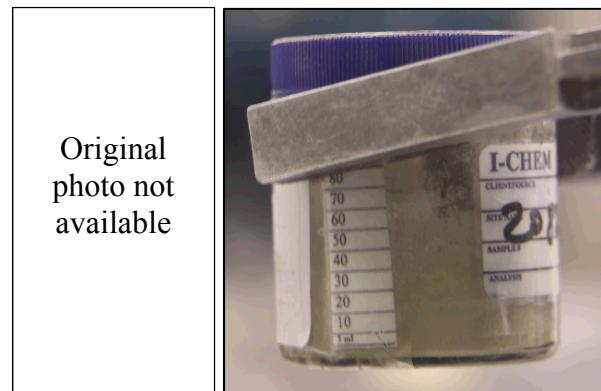
As found.

**Figure C-16. Tank 241-AN-101 CC8; Jar ID 20828, Comp.**



As found.

**Figure C-17. Tank 241-AN-101 CC8; Jar ID 20825, Comp.**



As found.

**Figure C-18. Tank 241-AN-101 CC8; Jar ID 20827, Comp.**

RPP-RPT-59874  
Appendix C

As found.

**Figure C-19.** Tank 241-AN-101/C-101; Jar ID 20856.

As received.

As found.

**Figure C-20.** Tank 241-AN-101/C-101; Jar ID 1AN-13-01; Customer Jar ID 1AN-13-01.

As received.

As found.

**Figure C-21.** Tank 241-AN-101/C-101; Jar ID 1AN-13-01A;  
Customer Jar ID 1AN-13-01A.

## RPP-RPT-59874

## Appendix C



**Figure C-22. Tank 241-AN-101/C-101; Jar ID 1AN-13-01DUP;  
Customer Jar ID 1AN-13-01DUP.**



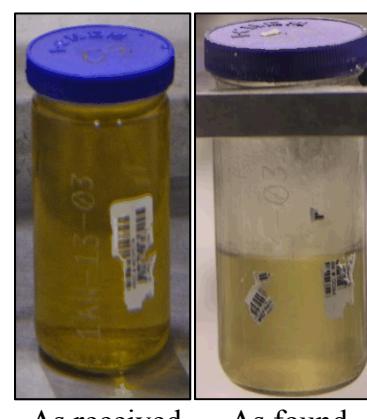
**Figure C-23. Tank 241-AN-101/C-101; Jar ID 20855;  
Customer Jar ID AN-101/C-101 Comp.**



**Figure C-24. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-02;  
Customer Jar ID 1AN-13-02.**

RPP-RPT-59874  
Appendix C

**Figure C-25. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-03A;  
Customer Jar ID 1AN-13-03A.**



**Figure C-26. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-03;  
Customer Jar ID 1AN-13-03.**



**Figure C-27. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-04;  
Customer Jar ID 1AN-13-04.**

## RPP-RPT-59874

## Appendix C



As received. As found.

**Figure C-28. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-04A; Customer Jar ID 1AN-13-04A.**



As received. As found.

**Figure C-29. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-05; Customer Jar ID 1AN-13-05.**

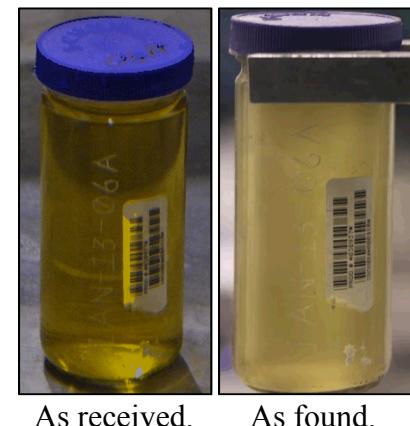


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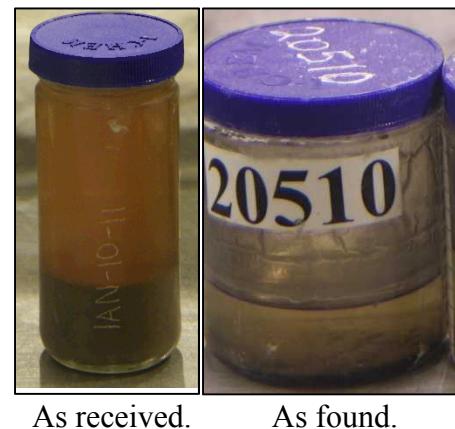
**Figure C-30. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-05A; Customer Jar ID 1AN-13-05A.**

RPP-RPT-59874  
Appendix C

**Figure C-31. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-06;  
Customer Jar ID 1AN-13-06.**



**Figure C-32. Tank 241-AN-101/C-101 CCC1; Jar ID 1AN-13-06A;  
Customer Jar ID 1AN-13-06A.**



**Figure C-33. Tank 241-AN-101/C-104; Jar ID 20510; Customer Jar ID 1AN-10-11.**

## RPP-RPT-59874

## Appendix C



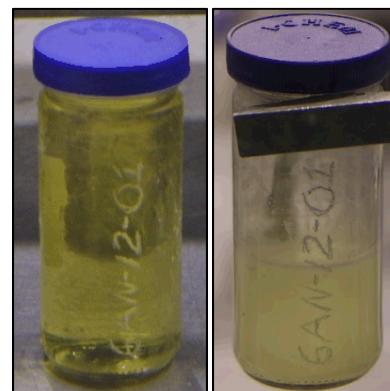
As found.

**Figure C-34. Tank 241-AN-106; Jar ID 20652.**



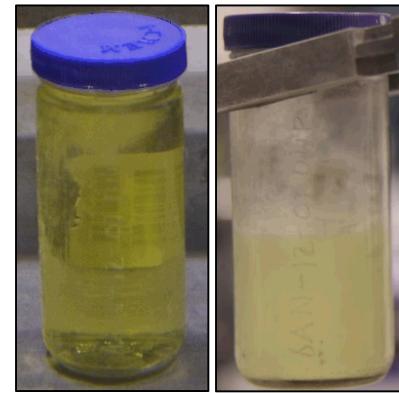
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**Figure C-35. Tank 241-AN-106; Jar ID 20523; Customer Jar ID 6AN-11-Comp.**

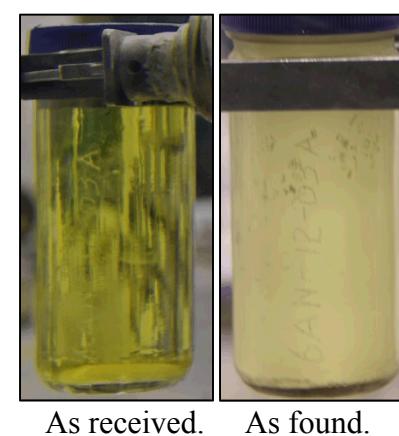


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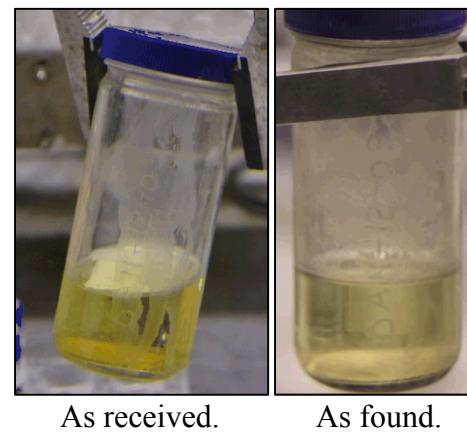
**Figure C-36. Tank 241-AN-106; Jar ID 6AN-12-01; Customer Jar ID 6AN-12-01.**

RPP-RPT-59874  
Appendix C

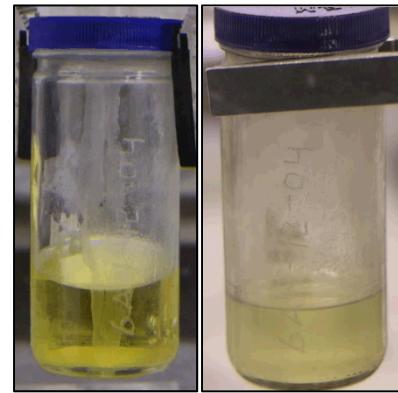
**Figure C-37. Tank 241-AN-106; Jar ID 6AN-12-01DUP; Customer Jar ID 6AN-12-01DUP.**



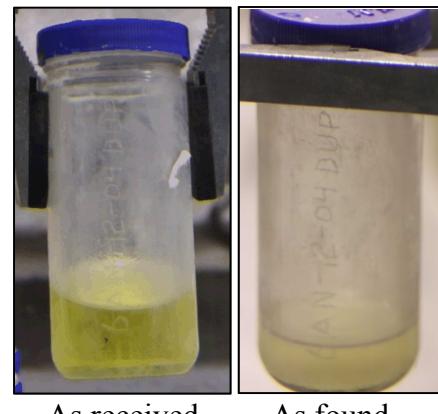
**Figure C-38. Tank 241-AN-106; Jar ID 6AN-12-03A; Customer Jar ID 6AN-12-03A.**



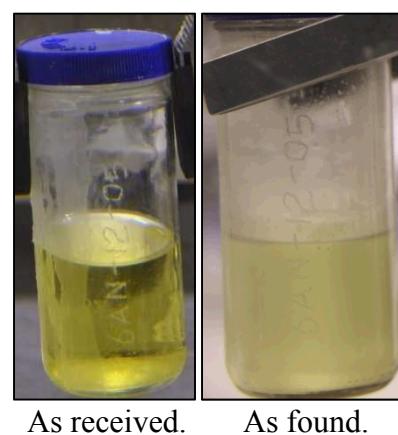
**Figure C-39. Tank 241-AN-106; Jar ID 6AN-12-03; Customer Jar ID 6AN-12-03.**

RPP-RPT-59874  
Appendix C

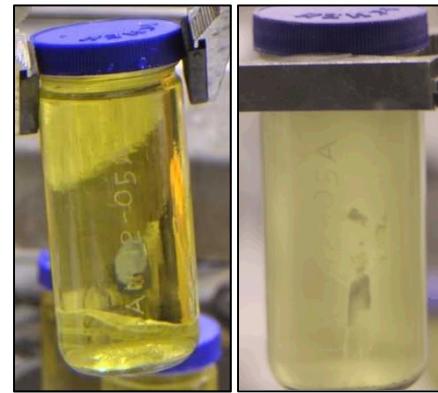
**Figure C-40. Tank 241-AN-106; Jar ID 6AN-12-04; Customer Jar ID 6AN-12-04.**



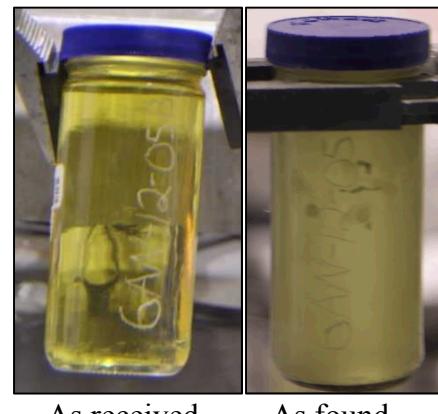
**Figure C-41. Tank 241-AN-106; Jar ID 6AN-12-04DUP;  
Customer Jar ID 6AN-12-04DUP.**



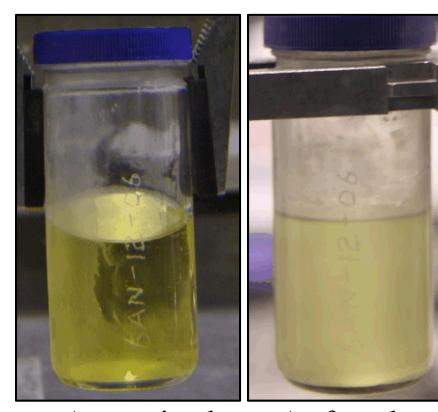
**Figure C-42. Tank 241-AN-106; Jar ID 6AN-12-05; Customer Jar ID 6AN-12-05.**

RPP-RPT-59874  
Appendix C

**Figure C-43. Tank 241-AN-106; Jar ID 6AN-12-05A; Customer Jar ID 6AN-12-05A.**



**Figure C-44. Tank 241-AN-106; Jar ID 6AN-12-05B; Customer Jar ID 6AN-12-05B.**



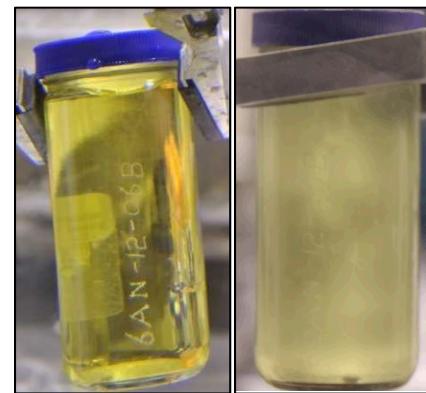
**Figure C-45. Tank 241-AN-106; Jar ID 6AN-12-06; Customer Jar ID 6AN-12-06.**

## RPP-RPT-59874

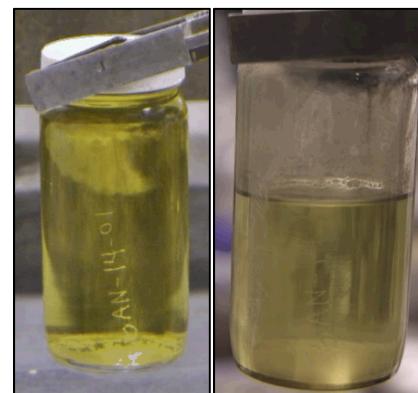
## Appendix C



As received. As found.

**Figure C-46. Tank 241-AN-106; Jar ID 6AN-12-06A; Customer Jar ID 6AN-12-06A3.**

As received. As found.

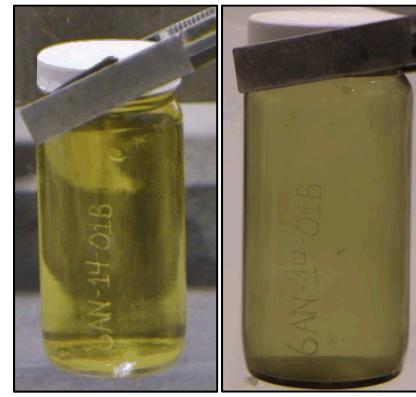
**Figure C-47. Tank 241-AN-106; Jar ID 6AN-12-06B; Customer Jar ID 6AN-12-06B.**

As received. As found.

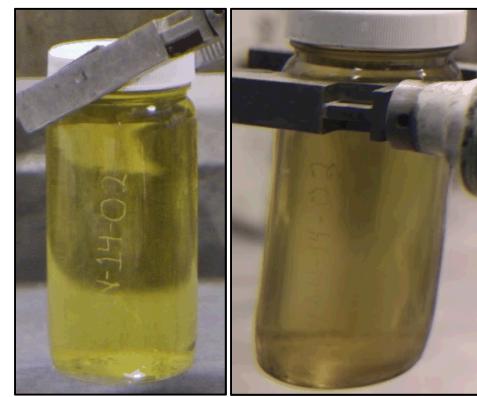
**Figure C-48. Tank 241-AN-106; Jar ID 6AN-14-01; Customer Jar ID 6AN-14-01.**

RPP-RPT-59874  
Appendix C

As received. As found.

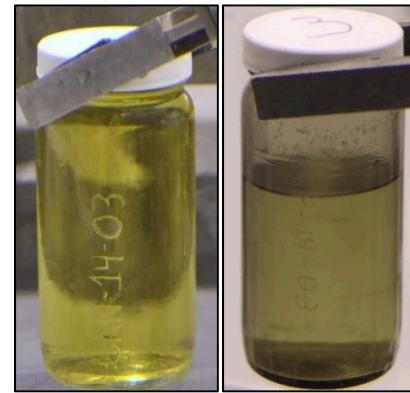
**Figure C-49. Tank 241-AN-106; Jar ID 6AN-14-01A; Customer Jar ID 6AN-14-01A.**

As received. As found.

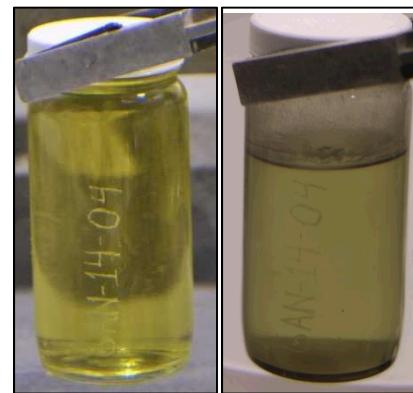
**Figure C-50. Tank 241-AN-106; Jar ID 6AN-14-01B; Customer Jar ID 6AN-14-01B.**

As received. As found.

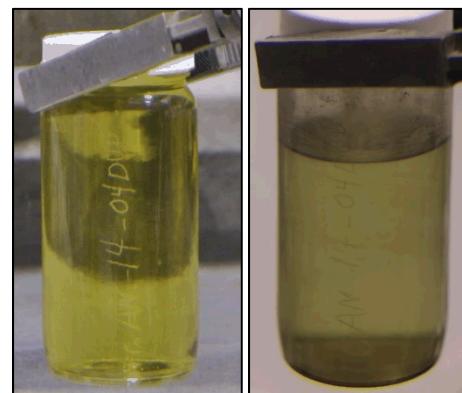
**Figure C-51. Tank 241-AN-106; Jar ID 6AN-14-02; Customer Jar ID 6AN-14-02.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-52. Tank 241-AN-106; Jar ID 6AN-14-03; Customer Jar ID 6AN-14-03.**

As received. As found.

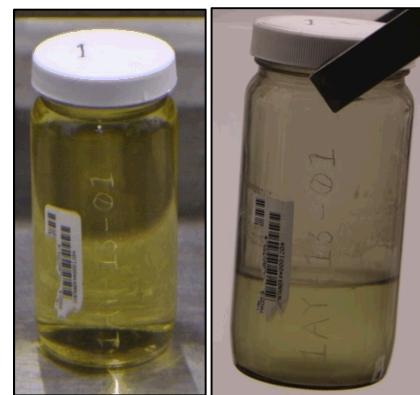
**Figure C-53. Tank 241-AN-106; Jar ID 6AN-14-04; Customer Jar ID 6AN-14-04.**

As received. As found.

**Figure C-54. Tank 241-AN-106; Jar ID 6AN-14-04DUP; Customer Jar ID 6AN-14-04DUP.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-55. Tank 241-AN-106; Jar ID 6AN-14-05; Customer Jar ID 6AN-14-05.**

As received. As found.

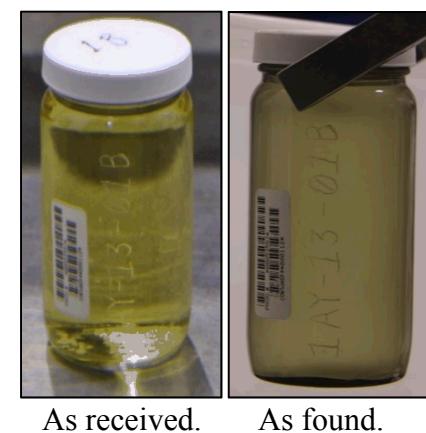
**Figure C-56. Tank 241-AY-101; Jar ID 1AY-13-01; Customer Jar ID 1AY-13-01.**

As received. As found.

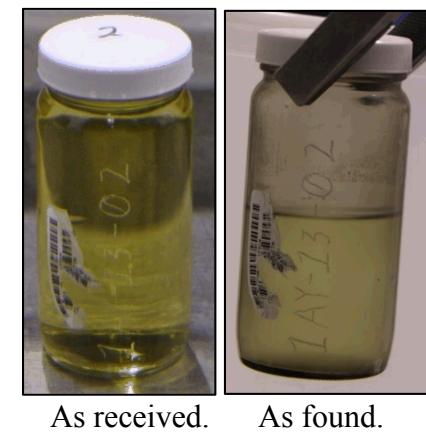
**Figure C-57. Tank 241-AY-101; Jar ID 1AY-13-01A; Customer Jar ID 1AY-13-01A.**

RPP-RPT-59874  
Appendix C

**Figure C-58. Tank 241-AY-101; Jar ID 1AY-13-01DUP; Customer Jar ID 1AY-13-01DUP.**



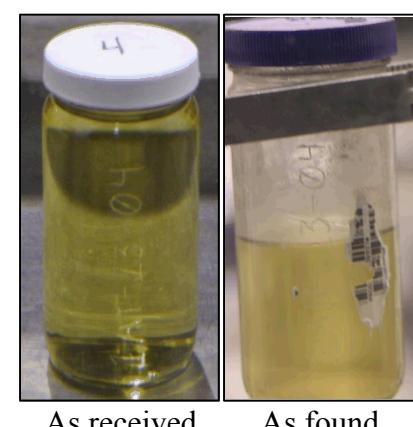
**Figure C-59. Tank 241-AY-101; Jar ID 1AY-13-01B; Customer Jar ID 1AY-13-01B.**



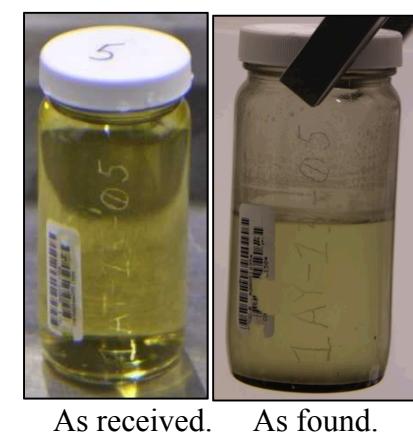
**Figure C-60. Tank 241-AY-101; Jar ID 1AY-13-02; Customer Jar ID 1AY-13-02.**

RPP-RPT-59874  
Appendix C

**Figure C-61. Tank 241-AY-101; Jar ID 1AY-13-02A; Customer Jar ID 1AY-13-02A.**



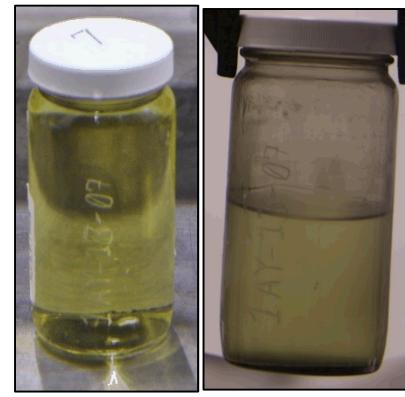
**Figure C-62. Tank 241-AY-101; Jar ID 1AY-13-04; Customer Jar ID 1AY-13-04.**



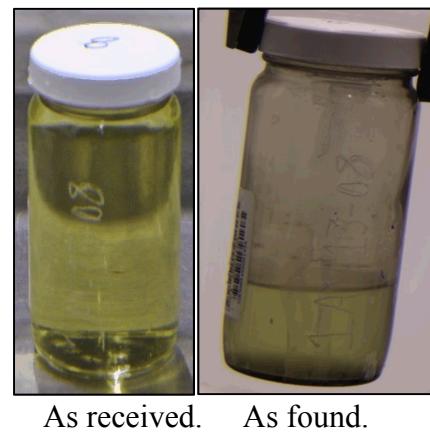
**Figure C-63. Tank 241-AY-101; Jar ID 1AY-13-05; Customer Jar ID 1AY-13-05.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-64. Tank 241-AY-101; Jar ID 1AY-13-06; Customer Jar ID 1AY-13-06.**

As received. As found.

**Figure C-65. Tank 241-AY-101; Jar ID 1AY-13-07; Customer Jar ID 1AY-13-07.**

As received. As found.

**Figure C-66. Tank 241-AY-101; Jar ID 1AY-13-08; Customer Jar ID 1AY-13-08.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-67. Tank 241-AY-101; Jar ID 1AY-13-09; Customer Jar ID 1AY-13-09.**

As received. As found.

**Figure C-68. Tank 241-AP-105; Jar ID 5AP-11-01; Customer Jar ID 5AP-11-01.**

As received. As found.

**Figure C-69. Tank 241-AP-105; Jar ID 5AP-11-03; Customer Jar ID 5AP-11-03.**

## RPP-RPT-59874

## Appendix C



As received. As found.

**Figure C-70. Tank 241-AP-105; Jar ID 5AP-11-03DUP; Customer Jar ID 5AP-11-03DUP.**

As received. As found.

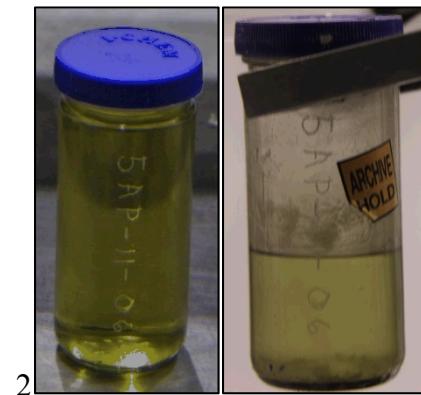
**Figure C-71. Tank 241-AP-105; Jar ID 5AP-11-04; Customer Jar ID 5AP-11-04.**

As received. As found.

**Figure C-72. Tank 241-AP-105; Jar ID 5AP-11-05; Customer Jar ID 5AP-11-05.**

## RPP-RPT-59874

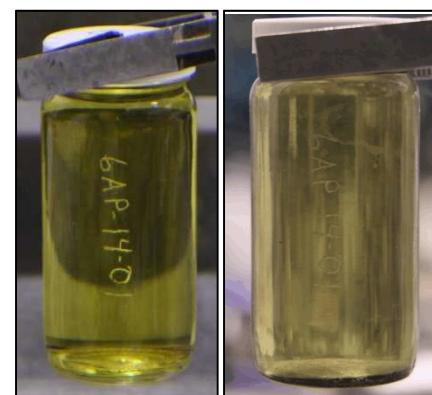
## Appendix C



As received. As found.

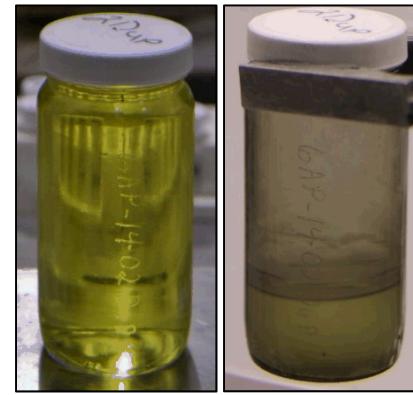
**Figure C-73. Tank 241-AP-105; Jar ID 5AP-11-06; Customer Jar ID 5AP-11-06.**

As received. As found.

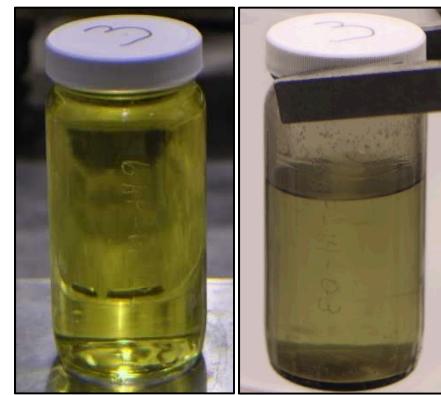
**Figure C-74. Tank 241-AP-105; Jar ID 5AP-11-07; Customer Jar ID 5AP-11-07.**

As received. As found.

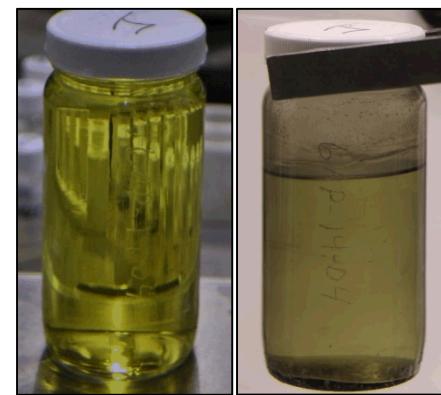
**Figure C-75. Tank 241-AP-106; Jar ID 6AP-14-01; Customer Jar ID 6AP-14-01.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-76. Tank 241-AP-106; Jar ID 6AP-14-02DUP; Customer Jar ID 6AP-14-02DUP.**

As received. As found.

**Figure C-77. Tank 241-AP-106; Jar ID 6AP-14-03; Customer Jar ID 6AP-14-03.**

As received. As found.

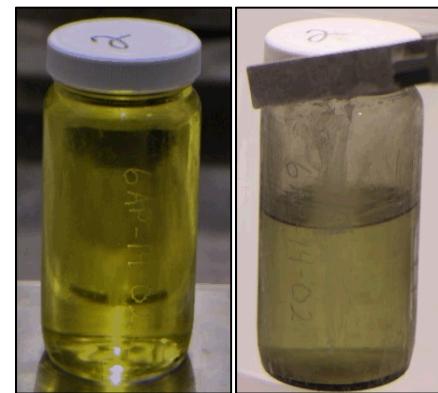
**Figure C-78. AP-106; Jar ID 6AP-14-04; Customer Jar ID 6AP-14-04.**

## RPP-RPT-59874

## Appendix C



As received. As found.

**Figure C-79. Tank 241-AP-106; Jar ID 6AP-14-05; Customer Jar ID 6AP-14-05.**

As received. As found.

**Figure C-80. Tank 241-AP-106; Jar ID 6AP-14-02; Customer Jar ID 6AP-14-02.**

As received. As found.

**Figure C-81. Tank 241-AP-107; Jar ID 7AP-10-01; Customer Jar ID 7AP-10-01.**

RPP-RPT-59874  
Appendix C

As received. As found.

**Figure C-82. Tank 241-AP-107; Jar ID 7AP-10-03A; Customer Jar ID 7AP-10-03A.**

As received. As found.

**Figure C-83. Tank 241-AP-107; Jar ID 7AP-10-04A; Customer Jar ID 7AP-10-04A.**

As received. As found.

**Figure C-84. Tank 241-AP-107; Jar ID 7AP-10-02A; Customer Jar ID 7AP-10-02A.**

RPP-RPT-59874

**APPENDIX D.**

**DATA SUMMARY REPORTS**

(19 pages, including cover sheet)

**RPP-RPT-59874**  
**Appendix D**

**Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000370	Inorg.		71-50-1	Acetate	µg/mL	92.9	<0.0100	567	566	567	0.229	96.7	5.00	n/a	--
S16R000371	Inorg.		71-50-1	Acetate	µg/mL	92.9	<0.0100	569	n/a	n/a	n/a	n/a	5.00	n/a	--
S16R000370	Inorg.		7429-90-5	Aluminum	µg/mL	98.6	<0.0140	4.05E+03	4.12E+03	4.09E+3	1.69	91.1	1.40	n/a	--
S16R000371	Inorg.		7429-90-5	Aluminum	µg/mL	98.6	<0.0140	4.12E+03	n/a	n/a	n/a	n/a	1.40	n/a	--
S16R000370	Inorg.		7440-36-0	Antimony	µg/mL	95.2	<0.0180	<0.180	<0.180	n/a	n/a	85.4	0.180	n/a	U
S16R000371	Inorg.		7440-36-0	Antimony	µg/mL	95.2	<0.0180	<0.180	n/a	n/a	n/a	n/a	0.180	n/a	U
S16R000370	Inorg.		7440-38-2	Arsenic	µg/mL	99.2	<0.0150	<1.50	<1.50	n/a	n/a	93.2	1.50	n/a	U
S16R000371	Inorg.		7440-38-2	Arsenic	µg/mL	99.2	<0.0150	<1.50	n/a	n/a	n/a	n/a	1.50	n/a	U
S16R000370	Inorg.		7440-39-3	Barium	µg/mL	98.8	<1.00E-03	0.348	0.361	0.355	3.55	89.9	0.100	n/a	J
S16R000371	Inorg.		7440-39-3	Barium	µg/mL	98.8	<1.00E-03	0.385	n/a	n/a	n/a	n/a	0.100	n/a	J
S16R000370	Inorg.		7440-41-7	Beryllium	µg/mL	99.1	<1.00E-03	0.343	0.345	0.344	0.550	93.8	0.100	n/a	J
S16R000371	Inorg.		7440-41-7	Beryllium	µg/mL	99.1	<1.00E-03	0.354	n/a	n/a	n/a	n/a	0.100	n/a	J
S16R000370	Inorg.		7440-69-9	Bismuth	µg/mL	98.0	<0.0190	<1.90	<1.90	n/a	n/a	91.0	1.90	n/a	U
S16R000371	Inorg.		7440-69-9	Bismuth	µg/mL	98.0	<0.0190	<1.90	n/a	n/a	n/a	n/a	1.90	n/a	U
S16R000370	Inorg.		7440-42-8	Boron	µg/mL	99.2	<2.00E-03	10.1	10.3	10.2	2.35	93.8	0.200	n/a	e
S16R000371	Inorg.		7440-42-8	Boron	µg/mL	99.2	<2.00E-03	10.6	n/a	n/a	n/a	n/a	0.200	n/a	--
S16R000370	Inorg.		24959-67-9	Bromide	µg/mL	94.4	<6.00E-03	40.8	40.1	40.4	1.69	92.0	3.00	n/a	J
S16R000371	Inorg.		24959-67-9	Bromide	µg/mL	94.4	<6.00E-03	40.8	n/a	n/a	n/a	n/a	3.00	n/a	J
S16R000370	Inorg.		7440-43-9	Cadmium	µg/mL	101	<1.00E-03	0.703	0.696	0.700	1.09	89.9	0.100	n/a	J
S16R000371	Inorg.		7440-43-9	Cadmium	µg/mL	101	<1.00E-03	0.726	n/a	n/a	n/a	n/a	0.100	n/a	J
S16R000370	Inorg.		7440-70-2	Calcium	µg/mL	99.6	<0.120	<12.0	<12.0	n/a	n/a	87.2	12.0	n/a	U
S16R000371	Inorg.		7440-70-2	Calcium	µg/mL	99.6	<0.120	<12.0	n/a	n/a	n/a	n/a	12.0	n/a	U
S16R000370	Inorg.		7440-45-1	Cerium	µg/mL	97.5	0.0562	<2.50	3.18	n/a	n/a	96.2	2.50	n/a	U
S16R000371	Inorg.		7440-45-1	Cerium	µg/mL	97.5	0.0562	3.45	n/a	n/a	n/a	n/a	2.50	n/a	BJ
S16R000370	Inorg.		7440-46-2	Cesium	µg/mL	106	<3.00E-06	2.08	2.07	2.08	0.530	93.3	7.50E-03	n/a	--
S16R000371	Inorg.		7440-46-2	Cesium	µg/mL	106	<3.00E-06	2.18	n/a	n/a	n/a	n/a	7.50E-03	n/a	--
S16R000370	Inorg.		16887-00-6	Chloride	µg/mL	92.5	<4.00E-03	1.25E+03	1.25E+03	1.25E+3	0.209	97.2	2.00	n/a	--
S16R000371	Inorg.		16887-00-6	Chloride	µg/mL	92.5	<4.00E-03	1.25E+03	n/a	n/a	n/a	n/a	2.00	n/a	--
S16R000370	Inorg.		7440-47-3	Chromium	µg/mL	99.3	<2.00E-03	235	239	237	1.61	90.3	0.200	n/a	--
S16R000371	Inorg.		7440-47-3	Chromium	µg/mL	99.3	<2.00E-03	237	n/a	n/a	n/a	n/a	0.200	n/a	--
S16R000370	Inorg.		7440-48-4	Cobalt	µg/mL	99.8	<1.00E-03	1.36	1.39	1.37	1.66	90.1	0.100	n/a	--
S16R000371	Inorg.		7440-48-4	Cobalt	µg/mL	99.8	<1.00E-03	1.42	n/a	n/a	n/a	n/a	0.100	n/a	--
S16R000370	Inorg.		7440-50-8	Copper	µg/mL	99.2	<2.00E-03	0.501	0.371	0.436	29.8	96.9	0.200	n/a	J
S16R000371	Inorg.		7440-50-8	Copper	µg/mL	99.2	<2.00E-03	0.510	n/a	n/a	n/a	n/a	0.200	n/a	J
S16R000370	Inorg.		Density	Density	g/mL	99.86	n/a	1.202	1.202	1.202	0.0	n/a	1.000E-03	n/a	--
S16R000371	Inorg.		Density	Density	g/mL	99.86	n/a	1.199	n/a	n/a	n/a	n/a	1.000E-03	n/a	--
S16R000370	Inorg.		7440-53-1	Europium	µg/mL	99.5	<1.00E-03	<0.100	<0.100	n/a	n/a	97.0	0.100	n/a	U
S16R000371	Inorg.		7440-53-1	Europium	µg/mL	99.5	<1.00E-03	<0.100	n/a	n/a	n/a	n/a	0.100	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000370	Inorg.		16984-48-8	Fluoride	µg/mL	93.4	<1.00E-03	1.85E+03	1.85E+03	1.85E+3	0.357	98.2	5.00	n/a	--
S16R000371	Inorg.		16984-48-8	Fluoride	µg/mL	93.4	<1.00E-03	1.88E+03	n/a	n/a	n/a	n/a	5.00	n/a	--
S16R000370	Inorg.		12311-97-6	Formate	µg/mL	93.7	0.0520	657	655	656	0.328	97.6	3.50	n/a	--
S16R000371	Inorg.		12311-97-6	Formate	µg/mL	93.7	0.0520	659	n/a	n/a	n/a	n/a	3.50	n/a	--
S16R000370	Inorg.		666-14-8	Glycolate	µg/mL	93.5	<5.00E-03	164	164	164	0.0323	97.6	2.50	n/a	--
S16R000371	Inorg.		666-14-8	Glycolate	µg/mL	93.5	<5.00E-03	167	n/a	n/a	n/a	n/a	2.50	n/a	--
S16R000370	Inorg.		HYDROXIDE	Hydroxide	µg/mL	104	<9.22	2.53E+04	2.44E+04	2.49E+4	3.68	98.2	553	n/a	--
S16R000371	Inorg.		HYDROXIDE	Hydroxide	µg/mL	104	<9.22	2.45E+04	n/a	n/a	n/a	n/a	553	n/a	--
S16R000370	Inorg.		7439-89-6	Iron	µg/mL	100	<0.0200	92.3	94.4	93.3	2.25	91.4	2.00	n/a	--
S16R000371	Inorg.		7439-89-6	Iron	µg/mL	100	<0.0200	93.9	n/a	n/a	n/a	n/a	2.00	n/a	--
S16R000370	Inorg.		7439-91-0	Lanthanum	µg/mL	98.8	<1.00E-03	<0.100	<0.100	n/a	n/a	95.3	0.100	n/a	U
S16R000371	Inorg.		7439-91-0	Lanthanum	µg/mL	98.8	<1.00E-03	<0.100	n/a	n/a	n/a	n/a	0.100	n/a	U
S16R000370	Inorg.		7439-92-1	Lead	µg/mL	102	<0.0130	2.29	2.67	2.48	15.4	91.9	1.30	n/a	J
S16R000371	Inorg.		7439-92-1	Lead	µg/mL	102	<0.0130	2.37	n/a	n/a	n/a	n/a	1.30	n/a	J
S16R000370	Inorg.		7439-93-2	Lithium	µg/mL	100	<1.00E-03	<0.100	<0.100	n/a	n/a	98.2	0.100	n/a	U
S16R000371	Inorg.		7439-93-2	Lithium	µg/mL	100	<1.00E-03	<0.100	n/a	n/a	n/a	n/a	0.100	n/a	U
S16R000370	Inorg.		7439-95-4	Magnesium	µg/mL	99.0	<9.00E-03	<0.900	<0.900	n/a	n/a	95.3	0.900	n/a	U
S16R000371	Inorg.		7439-95-4	Magnesium	µg/mL	99.0	<9.00E-03	<0.900	n/a	n/a	n/a	n/a	0.900	n/a	U
S16R000370	Inorg.		7439-96-5	Manganese	µg/mL	100	<1.00E-03	<0.100	<0.100	n/a	n/a	92.4	0.100	n/a	U
S16R000371	Inorg.		7439-96-5	Manganese	µg/mL	100	<1.00E-03	<0.100	n/a	n/a	n/a	n/a	0.100	n/a	U
S16R000378	Inorg.	HG	7439-97-6	Mercury	µg/mL	96.6	1.20E-05	0.206	n/a	n/a	n/a	n/a	4.40E-04	n/a	J
S16R000379	Inorg.	HG	7439-97-6	Mercury	µg/mL	96.6	1.20E-05	0.208	0.213	0.210	2.01	102	4.40E-04	n/a	J
S16R000370	Inorg.		7439-98-7	Molybdenum	µg/mL	96.0	<2.00E-03	17.2	17.6	17.4	2.45	92.0	0.200	n/a	--
S16R000371	Inorg.		7439-98-7	Molybdenum	µg/mL	96.0	<2.00E-03	17.6	n/a	n/a	n/a	n/a	0.200	n/a	--
S16R000370	Inorg.		7440-00-8	Neodymium	µg/mL	96.7	<0.0150	<1.50	<1.50	n/a	n/a	97.2	1.50	n/a	U
S16R000371	Inorg.		7440-00-8	Neodymium	µg/mL	96.7	<0.0150	<1.50	n/a	n/a	n/a	n/a	1.50	n/a	U
S16R000370	Inorg.		7440-02-0	Nickel	µg/mL	98.5	<2.00E-03	34.1	34.7	34.4	1.85	87.2	0.200	n/a	--
S16R000371	Inorg.		7440-02-0	Nickel	µg/mL	98.5	<2.00E-03	34.3	n/a	n/a	n/a	n/a	0.200	n/a	--
S16R000370	Inorg.		7440-30-1	Niobium	µg/mL	95.9	<6.00E-03	<0.600	<0.600	n/a	n/a	94.2	0.600	n/a	U
S16R000371	Inorg.		7440-30-1	Niobium	µg/mL	95.9	<6.00E-03	<0.600	n/a	n/a	n/a	n/a	0.600	n/a	U
S16R000370	Inorg.		14797-55-8	Nitrate	µg/mL	93.6	<0.0210	5.25E+04	5.28E+04	5.26E+4	0.591	97.4	105	n/a	--
S16R000371	Inorg.		14797-55-8	Nitrate	µg/mL	93.6	<0.0210	5.31E+04	n/a	n/a	n/a	n/a	105	n/a	--
S16R000370	Inorg.		14797-65-0	Nitrite	µg/mL	94.4	<9.00E-03	2.95E+04	2.96E+04	2.95E+4	0.488	98.1	45.0	n/a	--
S16R000371	Inorg.		14797-65-0	Nitrite	µg/mL	94.4	<9.00E-03	2.97E+04	n/a	n/a	n/a	n/a	45.0	n/a	--
S16R000370	Inorg.		338-70-5	Oxalate	µg/mL	93.7	<9.00E-03	954	949	952	0.533	93.6	4.50	n/a	--
S16R000371	Inorg.		338-70-5	Oxalate	µg/mL	93.7	<9.00E-03	944	n/a	n/a	n/a	n/a	4.50	n/a	--
S16R000370	Inorg.		7440-05-3	Palladium	µg/mL	99.8	<0.0120	2.11	1.53	1.82	31.8	90.1	1.20	n/a	J
S16R000371	Inorg.		7440-05-3	Palladium	µg/mL	99.8	<0.0120	1.88	n/a	n/a	n/a	n/a	1.20	n/a	J

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000370	Inorg.		%WATER	Percent water	%	101	n/a	74.0	80.3	77.2	8.15	n/a	0.0100	n/a	--
S16R000371	Inorg.		%WATER	Percent water	%	101	n/a	75.5	n/a	n/a	n/a	n/a	0.0100	n/a	--
S16R000370	Inorg.		14265-44-2	Phosphate	µg/mL	93.8	<0.0140	2.91E+03	2.94E+03	2.93E+3	1.07	95.8	7.00	n/a	--
S16R000371	Inorg.		14265-44-2	Phosphate	µg/mL	93.8	<0.0140	2.66E+03	n/a	n/a	n/a	n/a	7.00	n/a	--
S16R000370	Inorg.		7723-14-0	Phosphorus	µg/mL	96.2	<0.0130	726	853	790	16.1	98.3	1.30	n/a	--
S16R000371	Inorg.		7723-14-0	Phosphorus	µg/mL	96.2	<0.0130	907	n/a	n/a	n/a	n/a	1.30	n/a	--
S16R000370	Inorg.		7440-09-7	Potassium	µg/mL	97.9	<0.0220	848	886	867	4.30	94.3	2.20	n/a	--
S16R000371	Inorg.		7440-09-7	Potassium	µg/mL	97.9	<0.0220	871	n/a	n/a	n/a	n/a	2.20	n/a	--
S16R000370	Inorg.		7440-10-0	Praseodymium	µg/mL	100	<0.0260	<2.60	<2.60	n/a	n/a	99.7	2.60	n/a	U
S16R000371	Inorg.		7440-10-0	Praseodymium	µg/mL	100	<0.0260	<2.60	n/a	n/a	n/a	n/a	2.60	n/a	U
S16R000370	Inorg.		7440-16-6	Rhodium	µg/mL	97.5	<0.0120	2.15	2.43	2.29	12.4	94.9	1.20	n/a	J
S16R000371	Inorg.		7440-16-6	Rhodium	µg/mL	97.5	<0.0120	2.29	n/a	n/a	n/a	n/a	1.20	n/a	J
S16R000370	Inorg.		7440-17-7	Rubidium	µg/mL	98.3	<0.0570	<5.70	<5.70	n/a	n/a	94.3	5.70	n/a	U
S16R000371	Inorg.		7440-17-7	Rubidium	µg/mL	98.3	<0.0570	<5.70	n/a	n/a	n/a	n/a	5.70	n/a	U
S16R000370	Inorg.		7440-18-8	Ruthenium	µg/mL	104	<5.00E-03	4.01	4.04	4.03	0.668	87.7	0.500	n/a	J
S16R000371	Inorg.		7440-18-8	Ruthenium	µg/mL	104	<5.00E-03	4.16	n/a	n/a	n/a	n/a	0.500	n/a	J
S16R000370	Inorg.		7440-19-9	Samarium	µg/mL	96.5	<0.0170	1.86	<1.70	n/a	n/a	93.0	1.70	n/a	J
S16R000371	Inorg.		7440-19-9	Samarium	µg/mL	96.5	<0.0170	1.78	n/a	n/a	n/a	n/a	1.70	n/a	J
S16R000370	Inorg.		7782-49-2	Selenium	µg/mL	98.1	<0.0300	<3.00	<3.00	n/a	n/a	95.3	3.00	n/a	U
S16R000371	Inorg.		7782-49-2	Selenium	µg/mL	98.1	<0.0300	<3.00	n/a	n/a	n/a	n/a	3.00	n/a	U
S16R000370	Inorg.		7440-21-3	Silicon	µg/mL	103	<0.0120	36.5	46.8	41.7	24.8	103	1.20	n/a	c
S16R000371	Inorg.		7440-21-3	Silicon	µg/mL	103	<0.0120	36.6	n/a	n/a	n/a	n/a	1.20	n/a	--
S16R000370	Inorg.		7440-22-4	Silver	µg/mL	105	<3.00E-03	13.8	14.2	14.0	2.52	93.5	3.00	n/a	J
S16R000371	Inorg.		7440-22-4	Silver	µg/mL	105	<3.00E-03	16.5	n/a	n/a	n/a	n/a	3.00	n/a	J
S16R000370	Inorg.		7440-23-5	Sodium	µg/mL	105	<0.0920	9.70E+04	9.93E+04	9.82E+4	2.30	106	92.0	n/a	--
S16R000371	Inorg.		7440-23-5	Sodium	µg/mL	105	<0.0920	1.16E+05	n/a	n/a	n/a	n/a	92.0	n/a	--
S16R000370	Inorg.		7440-24-6	Strontium	µg/mL	98.9	<2.00E-03	<0.200	<0.200	n/a	n/a	88.0	0.200	n/a	U
S16R000371	Inorg.		7440-24-6	Strontium	µg/mL	98.9	<2.00E-03	<0.200	n/a	n/a	n/a	n/a	0.200	n/a	U
S16R000370	Inorg.		14808-79-8	Sulfate	µg/mL	94.6	<9.00E-03	4.18E+03	4.17E+03	4.17E+3	0.310	97.2	4.50	n/a	--
S16R000371	Inorg.		14808-79-8	Sulfate	µg/mL	94.6	<9.00E-03	4.16E+03	n/a	n/a	n/a	n/a	4.50	n/a	--
S16R000370	Inorg.		7704-34-9	Sulfur	µg/mL	97.5	<0.0280	1.57E+03	1.60E+03	1.59E+3	1.51	89.2	2.80	n/a	--
S16R000371	Inorg.		7704-34-9	Sulfur	µg/mL	97.5	<0.0280	1.59E+03	n/a	n/a	n/a	n/a	2.80	n/a	--
S16R000370	Inorg.		7440-25-7	Tantalum	µg/mL	98.4	<5.00E-03	<0.500	<0.500	n/a	n/a	97.1	0.500	n/a	U
S16R000371	Inorg.		7440-25-7	Tantalum	µg/mL	98.4	<5.00E-03	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	U
S16R000370	Inorg.		13494-80-9	Tellurium	µg/mL	99.4	<9.00E-03	<0.900	<0.900	n/a	n/a	93.0	0.900	n/a	U
S16R000371	Inorg.		13494-80-9	Tellurium	µg/mL	99.4	<9.00E-03	<0.900	n/a	n/a	n/a	n/a	0.900	n/a	U
S16R000370	Inorg.		7440-28-0	Thallium	µg/mL	102	<0.0150	<1.50	<1.50	n/a	n/a	87.4	1.50	n/a	U
S16R000371	Inorg.		7440-28-0	Thallium	µg/mL	102	<0.0150	<1.50	n/a	n/a	n/a	n/a	1.50	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag	
S16R000370	Inorg.		7772-98-7	Thiosulfate	µg/mL	96.1	<6.00E-03	<3.00	<3.00	n/a	n/a	97.3	3.00	n/a	U	
S16R000371	Inorg.		7772-98-7	Thiosulfate	µg/mL	96.1	<6.00E-03	<3.00	n/a	n/a	n/a	n/a	3.00	n/a	U	
S16R000370	Inorg.		7440-29-1	Thorium	µg/mL	97.2	<0.0130	<1.30	<1.30	n/a	n/a	89.7	1.30	n/a	U	
S16R000371	Inorg.		7440-29-1	Thorium	µg/mL	97.2	<0.0130	<1.30	n/a	n/a	n/a	n/a	1.30	n/a	U	
S16R000370	Inorg.		7440-31-5	Tin	µg/mL	99.5	<8.00E-03	9.32	9.54	9.43	2.32	88.0	0.800	n/a	--	
S16R000371	Inorg.		7440-31-5	Tin	µg/mL	99.5	<8.00E-03	9.65	n/a	n/a	n/a	n/a	0.800	n/a	--	
S16R000370	Inorg.		7440-32-6	Titanium	µg/mL	93.1	<2.00E-03	<0.200	<0.200	n/a	n/a	91.9	0.200	n/a	U	
S16R000371	Inorg.		7440-32-6	Titanium	µg/mL	93.1	<2.00E-03	<0.200	n/a	n/a	n/a	n/a	0.200	n/a	U	
S16R000370	Inorg.		TIC	TIC	µg/mL	95.8	<7.00	5.09E+3	5.13E+3	5.11E+3	0.783	97.1	35.0	n/a	--	
S16R000371	Inorg.		TIC	TIC	µg/mL	95.8	<7.00	5.13E+03	n/a	n/a	n/a	n/a	35.0	n/a	--	
S16R000370	Inorg.		TOC	TOC	µg/mL	92.1	<20.0	1.39E+3	1.38E+3	1.38E+3	0.722	92.6	100	n/a	--	
S16R000371	Inorg.		TOC	TOC	µg/mL	92.1	<20.0	1.40E+03	n/a	n/a	n/a	n/a	100	n/a	--	
S16R000370	Inorg.		7440-33-7	Tungsten	µg/mL	98.5	<0.0160	22.2	22.9	22.6	3.00	95.1	1.60	n/a	--	
S16R000371	Inorg.		7440-33-7	Tungsten	µg/mL	98.5	<0.0160	22.8	n/a	n/a	n/a	n/a	1.60	n/a	--	
S16R000370	Inorg.		7440-61-1	Uranium	µg/mL	96.3	<0.0290	21.1	22.4	21.7	6.30	97.7	2.90	n/a	J	
S16R000371	Inorg.		7440-61-1	Uranium	µg/mL	96.3	<0.0290	23.5	n/a	n/a	n/a	n/a	2.90	n/a	J	
S16R000370	Inorg.		7440-62-2	Vanadium	µg/mL	97.1	<1.00E-03	0.177	0.225	0.201	23.8	92.4	0.100	n/a	J	
S16R000371	Inorg.		7440-62-2	Vanadium	µg/mL	97.1	<1.00E-03	0.233	n/a	n/a	n/a	n/a	0.100	n/a	J	
S16R000370	Inorg.		7440-65-5	Yttrium	µg/mL	98.3	<2.00E-03	<0.200	<0.200	n/a	n/a	94.4	0.200	n/a	U	
S16R000371	Inorg.		7440-65-5	Yttrium	µg/mL	98.3	<2.00E-03	<0.200	n/a	n/a	n/a	n/a	0.200	n/a	U	
S16R000370	Inorg.		7440-66-6	Zinc	µg/mL	99.6	<0.0320	<3.20	<3.20	n/a	n/a	91.5	3.20	n/a	U	
S16R000371	Inorg.		7440-66-6	Zinc	µg/mL	99.6	<0.0320	<3.20	n/a	n/a	n/a	n/a	3.20	n/a	U	
S16R000370	Inorg.		7440-67-7	Zirconium	µg/mL	94.5	<1.00E-03	<0.100	0.194	n/a	n/a	92.1	0.100	n/a	U	
S16R000371	Inorg.		7440-67-7	Zirconium	µg/mL	94.5	<1.00E-03	0.132	n/a	n/a	n/a	n/a	0.100	n/a	J	
S16R000374	VOA		71-55-6	1,1,1-Trichloroethane	µg/L	103	<0.0433	<1.73	n/a	n/a	n/a	n/a	1.73	n/a	U	
S16R000375	VOA		71-55-6	1,1,1-Trichloroethane	µg/L	103	<0.0433	<1.73	n/a	n/a	n/a	n/a	104	1.73	n/a	U
S16R000374	VOA		76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	µg/L	85.4	<0.0396	<1.58	n/a	n/a	n/a	n/a	1.58	n/a	U	
S16R000375	VOA		76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	µg/L	85.4	<0.0396	<1.58	n/a	n/a	n/a	n/a	1.58	n/a	U	
S16R000374	VOA		79-00-5	1,1,2-Trichloroethane	µg/L	105	<0.0230	<0.920	n/a	n/a	n/a	n/a	0.920	n/a	U	
S16R000375	VOA		79-00-5	1,1,2-Trichloroethane	µg/L	105	<0.0230	<0.920	n/a	n/a	n/a	n/a	48.0	0.920	n/a	Ub
S16R000374	VOA		75-35-4	1,1-Dichloroethene	µg/L	85.8	<0.172	<6.87	n/a	n/a	n/a	n/a	6.87	n/a	U	
S16R000375	VOA		75-35-4	1,1-Dichloroethene	µg/L	85.8	<0.172	<6.87	n/a	n/a	n/a	n/a	149	6.87	n/a	Ub
S16R000374	VOA		95-50-1	1,2-Dichlorobenzene	µg/L	100	<0.0455	<1.82	n/a	n/a	n/a	n/a	1.82	n/a	U	
S16R000375	VOA		95-50-1	1,2-Dichlorobenzene	µg/L	100	<0.0455	<1.82	n/a	n/a	n/a	n/a	99.7	1.82	n/a	U
S16R000374	VOA		107-06-2	1,2-Dichloroethane	µg/L	110	<0.0243	<0.972	n/a	n/a	n/a	n/a	0.972	n/a	U	
S16R000375	VOA		107-06-2	1,2-Dichloroethane	µg/L	110	<0.0243	<0.972	n/a	n/a	n/a	n/a	110	0.972	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000374	VOA		71-36-3	1-Butanol	µg/L	98.6	<1.15	456	n/a	n/a	n/a	n/a	46.0	n/a	Jb
S16R000375	VOA		71-36-3	1-Butanol	µg/L	98.6	<1.15	1.05E+03	n/a	n/a	n/a	128	46.0	n/a	B
S16R000374	VOA		78-93-3	2-Butanone	µg/L	110	<0.458	<18.3	n/a	n/a	n/a	n/a	18.3	n/a	Ub
S16R000375	VOA		78-93-3	2-Butanone	µg/L	110	<0.458	<18.3	n/a	n/a	n/a	134	18.3	n/a	Ub
S16R000374	VOA		79-46-9	2-Nitropropane	µg/L	102	<0.265	<10.6	n/a	n/a	n/a	n/a	10.6	n/a	U
S16R000375	VOA		79-46-9	2-Nitropropane	µg/L	102	<0.265	<10.6	n/a	n/a	n/a	n/a	10.6	n/a	U
S16R000374	VOA		108-10-1	4-Methyl-2-Pentanone	µg/L	108	<0.453	<18.1	n/a	n/a	n/a	n/a	18.1	n/a	Ub
S16R000375	VOA		108-10-1	4-Methyl-2-Pentanone	µg/L	108	<0.453	<18.1	n/a	n/a	n/a	133	18.1	n/a	Ub
S16R000374	VOA		67-64-1	Acetone	µg/L	105	<0.573	152	n/a	n/a	n/a	n/a	22.9	n/a	J
S16R000375	VOA		67-64-1	Acetone	µg/L	105	<0.573	362	n/a	n/a	n/a	111	22.9	n/a	--
S16R000374	VOA		71-43-2	Benzene	µg/L	102	<0.0163	<0.652	n/a	n/a	n/a	n/a	0.652	n/a	U
S16R000375	VOA		71-43-2	Benzene	µg/L	102	<0.0163	<0.652	n/a	n/a	n/a	97.9	0.652	n/a	U
S16R000374	VOA		75-15-0	Carbon disulfide	µg/L	80.0	<0.0211	<0.844	n/a	n/a	n/a	n/a	0.844	n/a	U
S16R000375	VOA		75-15-0	Carbon disulfide	µg/L	80.0	<0.0211	<0.844	n/a	n/a	n/a	59.0	0.844	n/a	Ub
S16R000374	VOA		56-23-5	Carbon tetrachloride	µg/L	98.8	<0.0479	<1.92	n/a	n/a	n/a	n/a	1.92	n/a	U
S16R000375	VOA		56-23-5	Carbon tetrachloride	µg/L	98.8	<0.0479	<1.92	n/a	n/a	n/a	98.3	1.92	n/a	U
S16R000374	VOA		108-90-7	Chlorobenzene	µg/L	102	<0.0320	<1.28	n/a	n/a	n/a	n/a	1.28	n/a	U
S16R000375	VOA		108-90-7	Chlorobenzene	µg/L	102	<0.0320	<1.28	n/a	n/a	n/a	96.1	1.28	n/a	U
S16R000374	VOA		67-66-3	Chloroform	µg/L	108	<0.0414	<1.66	n/a	n/a	n/a	n/a	1.66	n/a	U
S16R000375	VOA		67-66-3	Chloroform	µg/L	108	<0.0414	<1.66	n/a	n/a	n/a	109	1.66	n/a	U
S16R000374	VOA		60-29-7	Diethyl ether	µg/L	99.0	<0.0423	<1.69	n/a	n/a	n/a	n/a	1.69	n/a	U
S16R000375	VOA		60-29-7	Diethyl ether	µg/L	99.0	<0.0423	<1.69	n/a	n/a	n/a	106	1.69	n/a	U
S16R000374	VOA		141-78-6	Ethyl acetate	µg/L	112	<0.183	<7.34	n/a	n/a	n/a	n/a	7.34	n/a	U
S16R000375	VOA		141-78-6	Ethyl acetate	µg/L	112	<0.183	<7.34	n/a	n/a	n/a	0.0	7.34	n/a	Ub
S16R000374	VOA		100-41-4	Ethylbenzene	µg/L	95.5	<0.0334	<1.34	n/a	n/a	n/a	n/a	1.34	n/a	U
S16R000375	VOA		100-41-4	Ethylbenzene	µg/L	95.5	<0.0334	<1.34	n/a	n/a	n/a	89.7	1.34	n/a	U
S16R000374	VOA		67-72-1	Hexachloroethane	µg/L	103	<0.111	<4.44	n/a	n/a	n/a	n/a	4.44	n/a	U
S16R000375	VOA		67-72-1	Hexachloroethane	µg/L	103	<0.111	<4.44	n/a	n/a	n/a	87.5	4.44	n/a	U
S16R000374	VOA		75-09-2	Methylene Chloride	µg/L	101	<0.0396	<1.58	n/a	n/a	n/a	n/a	1.58	n/a	U
S16R000375	VOA		75-09-2	Methylene Chloride	µg/L	101	<0.0396	<1.58	n/a	n/a	n/a	99.0	1.58	n/a	U
S16R000374	VOA		95-47-6	o-Xylene	µg/L	101	<0.0325	<1.30	n/a	n/a	n/a	n/a	1.30	n/a	U
S16R000375	VOA		95-47-6	o-Xylene	µg/L	101	<0.0325	<1.30	n/a	n/a	n/a	95.1	1.30	n/a	U
S16R000374	VOA		127-18-4	Tetrachloroethene	µg/L	97.4	<0.0583	<2.33	n/a	n/a	n/a	n/a	2.33	n/a	U
S16R000375	VOA		127-18-4	Tetrachloroethene	µg/L	97.4	<0.0583	<2.33	n/a	n/a	n/a	92.5	2.33	n/a	U
S16R000374	VOA		108-88-3	Toluene	µg/L	96.1	<0.0522	4.09	n/a	n/a	n/a	n/a	2.09	n/a	J
S16R000375	VOA		108-88-3	Toluene	µg/L	96.1	<0.0522	2.89	n/a	n/a	n/a	94.1	2.09	n/a	J
S16R000374	VOA		79-01-6	Trichloroethene	µg/L	105	<0.0378	<1.51	n/a	n/a	n/a	n/a	1.51	n/a	U
S16R000375	VOA		79-01-6	Trichloroethene	µg/L	105	<0.0378	<1.51	n/a	n/a	n/a	182	1.51	n/a	Ub

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000374	VOA		75-69-4	Trichlorofluoromethane	µg/L	92.8	<0.0458	<1.83	n/a	n/a	n/a	95.0	1.83	n/a	U
S16R000375	VOA		75-69-4	Trichlorofluoromethane	µg/L	92.8	<0.0458	<1.83	n/a	n/a	n/a	n/a	1.83	n/a	U
S16R000374	VOA		75-01-4	Vinyl chloride	µg/L	86.1	<0.0486	<1.94	n/a	n/a	n/a	87.9	1.94	n/a	U
S16R000375	VOA		75-01-4	Vinyl chloride	µg/L	86.1	<0.0486	<1.94	n/a	n/a	n/a	n/a	1.94	n/a	U
S16R000374	VOA		106-42-3	Xylene (m & p)	µg/L	95.6	<0.0703	<2.81	n/a	n/a	n/a	n/a	2.81	n/a	U
S16R000375	VOA		106-42-3	Xylene (m & p)	µg/L	95.6	<0.0703	<2.81	n/a	n/a	n/a	87.9	2.81	n/a	U
S16R000380	SVOA	O	95-95-4	2,4,5-Trichlorophenol	µg/L	81.7	<686	<686	<686	n/a	n/a	n/a	686	n/a	U
S16R000382	SVOA	O	95-95-4	2,4,5-Trichlorophenol	µg/L	81.7	<686	<686	n/a	n/a	n/a	n/a	686	n/a	U
S16R000380	SVOA	O	121-14-2	2,4-Dinitrotoluene	µg/L	82.5	<364	<364	<364	n/a	n/a	n/a	364	n/a	U
S16R000382	SVOA	O	121-14-2	2,4-Dinitrotoluene	µg/L	82.5	<364	<364	n/a	n/a	n/a	n/a	364	n/a	U
S16R000380	SVOA	O	110-80-5	2-Ethoxyethanol	µg/L	78.7	<932	<932	<932	n/a	n/a	n/a	932	n/a	U
S16R000382	SVOA	O	110-80-5	2-Ethoxyethanol	µg/L	78.7	<932	<932	n/a	n/a	n/a	n/a	932	n/a	U
S16R000380	SVOA	O	95-48-7	2-Methylphenol	µg/L	80.9	<631	<631	<631	n/a	n/a	n/a	631	n/a	U
S16R000382	SVOA	O	95-48-7	2-Methylphenol	µg/L	80.9	<631	<631	n/a	n/a	n/a	n/a	631	n/a	U
S16R000380	SVOA	O	108-39-4M	Cresol (m & p)	µg/L	81.9	<562	<562	<562	n/a	n/a	n/a	562	n/a	U
S16R000382	SVOA	O	108-39-4M	Cresol (m & p)	µg/L	81.9	<562	<562	n/a	n/a	n/a	n/a	562	n/a	U
S16R000380	SVOA	O	108-94-1	Cyclohexanone	µg/L	84.1	<651	<651	<651	n/a	n/a	n/a	651	n/a	U
S16R000382	SVOA	O	108-94-1	Cyclohexanone	µg/L	84.1	<651	<651	n/a	n/a	n/a	n/a	651	n/a	U
S16R000380	SVOA	O	87-68-3	Hexachlorobutadiene	µg/L	14.6	<751	<751	<751	n/a	n/a	n/a	751	n/a	U
S16R000382	SVOA	O	87-68-3	Hexachlorobutadiene	µg/L	14.6	<751	<751	n/a	n/a	n/a	n/a	751	n/a	U
S16R000380	SVOA	O	67-72-1	Hexachloroethane	µg/L	12.2	<700	<700	<700	n/a	n/a	n/a	700	n/a	U
S16R000382	SVOA	O	67-72-1	Hexachloroethane	µg/L	12.2	<700	<700	n/a	n/a	n/a	n/a	700	n/a	U
S16R000380	SVOA	O	78-83-1	Isobutyl alcohol	µg/L	39.6	<1.08E+03	<1.08E+03	<1.08E+03	n/a	n/a	n/a	1.08E+03	n/a	U
S16R000382	SVOA	O	78-83-1	Isobutyl alcohol	µg/L	39.6	<1.08E+03	<1.08E+03	n/a	n/a	n/a	n/a	1.08E+03	n/a	U
S16R000380	SVOA	O	98-95-3	Nitrobenzene	µg/L	74.8	<609	<609	<609	n/a	n/a	n/a	609	n/a	U
S16R000382	SVOA	O	98-95-3	Nitrobenzene	µg/L	74.8	<609	<609	n/a	n/a	n/a	n/a	609	n/a	U
S16R000380	SVOA	O	110-86-1	Pyridine	µg/L	73.1	<731	<731	<731	n/a	n/a	n/a	731	n/a	U
S16R000382	SVOA	O	110-86-1	Pyridine	µg/L	73.1	<731	<731	n/a	n/a	n/a	n/a	731	n/a	U
S16R000381	PCB	O	12674-11-2	Aroclor 1016	µg/L	n/a	<94.5	<94.5	<94.5	n/a	n/a	n/a	94.5	n/a	U
S16R000383	PCB	O	12674-11-2	Aroclor 1016	µg/L	n/a	<94.5	<94.5	n/a	n/a	n/a	n/a	94.5	n/a	U
S16R000381	PCB	O	11104-28-2	Aroclor 1221	µg/L	n/a	<17.8	<17.8	<17.8	n/a	n/a	n/a	17.8	n/a	U
S16R000383	PCB	O	11104-28-2	Aroclor 1221	µg/L	n/a	<17.8	<17.8	n/a	n/a	n/a	n/a	17.8	n/a	U
S16R000381	PCB	O	11141-16-5	Aroclor 1232	µg/L	n/a	<20.5	<20.5	<20.5	n/a	n/a	n/a	20.5	n/a	U
S16R000383	PCB	O	11141-16-5	Aroclor 1232	µg/L	n/a	<20.5	<20.5	n/a	n/a	n/a	n/a	20.5	n/a	U
S16R000381	PCB	O	53469-21-9	Aroclor 1242	µg/L	n/a	<31.5	<31.5	<31.5	n/a	n/a	n/a	31.5	n/a	U
S16R000383	PCB	O	53469-21-9	Aroclor 1242	µg/L	n/a	<31.5	<31.5	n/a	n/a	n/a	n/a	31.5	n/a	U
S16R000381	PCB	O	12672-29-6	Aroclor 1248	µg/L	n/a	<17.8	<17.8	<17.8	n/a	n/a	n/a	17.8	n/a	U
S16R000383	PCB	O	12672-29-6	Aroclor 1248	µg/L	n/a	<17.8	<17.8	n/a	n/a	n/a	n/a	17.8	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000381	PCB	O	11097-69-1	Aroclor 1254	µg/L	83.6	<6.50	<6.50	<6.50	n/a	n/a	78.6	6.50	n/a	U
S16R000383	PCB	O	11097-69-1	Aroclor 1254	µg/L	83.6	<6.50	<6.50	n/a	n/a	n/a	n/a	6.50	n/a	U
S16R000381	PCB	O	11096-82-5	Aroclor 1260	µg/L	n/a	<71.5	<71.5	<71.5	n/a	n/a	n/a	71.5	n/a	U
S16R000383	PCB	O	11096-82-5	Aroclor 1260	µg/L	n/a	<71.5	<71.5	n/a	n/a	n/a	n/a	71.5	n/a	U
S16R000376	Rad		14331-83-0	Actinium-228	µCi/mL	n/a	<9.57E-04	<5.92E-03	<4.30E-03	n/a	n/a	n/a	5.92E-03	n/a	U
S16R000377	Rad		14331-83-0	Actinium-228	µCi/mL	n/a	<9.57E-04	<6.46E-03	n/a	n/a	n/a	n/a	6.46E-03	n/a	U
S16R000376	Rad		14596-10-2	Americium-241	µCi/mL	97.3	<4.67E-07	1.62E-05	1.96E-05	1.79E-	18.9	n/a	2.81E-07	4.79	--
S16R000377	Rad		14596-10-2	Americium-241	µCi/mL	97.3	<4.67E-07	1.86E-05	n/a	n/a	n/a	n/a	1.97E-07	4.65	--
S16R000376	Rad		14596-10-2	Americium-241	µCi/mL	n/a	<4.50E-04	<0.219	<0.0108	n/a	n/a	n/a	0.219	n/a	U
S16R000377	Rad		14596-10-2	Americium-241	µCi/mL	n/a	<4.50E-04	<0.0559	n/a	n/a	n/a	n/a	0.0559	n/a	U
S16R000376	Rad		14234-35-6	Antimony-125	µCi/mL	110	<5.62E-04	<0.131	<0.0425	n/a	n/a	n/a	0.131	n/a	U
S16R000377	Rad		14234-35-6	Antimony-125	µCi/mL	110	<5.62E-04	<0.0830	n/a	n/a	n/a	n/a	0.0830	n/a	U
S16R000376	Rad		14913-49-6	Bismuth-212	µCi/mL	n/a	<3.54E-03	<0.0259	<0.0167	n/a	n/a	n/a	0.0259	n/a	U
S16R000377	Rad		14913-49-6	Bismuth-212	µCi/mL	n/a	<3.54E-03	<0.0282	n/a	n/a	n/a	n/a	0.0282	n/a	U
S16R000376	Rad		14733-03-0	Bismuth-214	µCi/mL	n/a	<6.33E-04	<0.0427	<0.0274	n/a	n/a	n/a	0.0427	n/a	U
S16R000377	Rad		14733-03-0	Bismuth-214	µCi/mL	n/a	<6.33E-04	<0.0635	n/a	n/a	n/a	n/a	0.0635	n/a	U
S16R000376	Rad		14762-75-5	Carbon-14	µCi/mL	99.0	<7.77E-07	5.90E-04	n/a	n/a	n/a	n/a	7.76E-07	0.887	--
S16R000377	Rad		14762-75-5	Carbon-14	µCi/mL	99.0	<7.77E-07	5.95E-04	5.91E-04	5.93E-	0.675	98.2	7.76E-07	0.884	--
S16R000376	Rad		13967-70-9	Cesium-134	µCi/mL	n/a	<1.48E-03	<0.173	<0.0735	n/a	n/a	n/a	0.173	n/a	U
S16R000377	Rad		13967-70-9	Cesium-134	µCi/mL	n/a	<1.48E-03	<0.0441	n/a	n/a	n/a	n/a	0.0441	n/a	U
S16R000376	Rad		10045-97-3	Cesium-137	µCi/mL	95.7	<2.98E-04	48.1	50.6	49.3	5.12	n/a	0.0240	0.07	--
S16R000377	Rad		10045-97-3	Cesium-137	µCi/mL	95.7	<2.98E-04	49.9	n/a	n/a	n/a	n/a	0.0154	0.06	--
S16R000376	Rad		10198-40-0	Cobalt-60	µCi/mL	97.8	<2.77E-04	<1.69E-03	1.07E-03	n/a	n/a	n/a	1.69E-03	n/a	U
S16R000377	Rad		10198-40-0	Cobalt-60	µCi/mL	97.8	<2.77E-04	<3.69E-03	n/a	n/a	n/a	n/a	3.69E-03	n/a	U
S16R000376	Rad		15510-73-3	Curium-242	µCi/mL	n/a	<1.01E-07	1.55E-07	<2.15E-07	n/a	n/a	n/a	1.05E-07	50.69	J
S16R000377	Rad		15510-73-3	Curium-242	µCi/mL	n/a	<1.01E-07	<1.14E-07	n/a	n/a	n/a	n/a	1.14E-07	n/a	U
S16R000376	Rad		CM-243/244	Curium-243/244	µCi/mL	n/a	<2.89E-07	3.43E-07	<4.30E-07	n/a	n/a	n/a	1.94E-07	34.83	J
S16R000377	Rad		CM-243/244	Curium-243/244	µCi/mL	n/a	<2.89E-07	2.43E-07	n/a	n/a	n/a	n/a	1.16E-07	42.47	J
S16R000370	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	36.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000371	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	36.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000376	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	46.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000377	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	46.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000375	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	14.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000372	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	46.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000373	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	46.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000374	Rad	NONE		Dose Rate	mRem/hr	n/a	n/a	14.0	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000376	Rad	14683-23-9		Europium-152	µCi/mL	n/a	<5.53E-04	<0.0854	<0.0457	n/a	n/a	n/a	0.0854	n/a	U
S16R000377	Rad	14683-23-9		Europium-152	µCi/mL	n/a	<5.53E-04	<0.142	n/a	n/a	n/a	n/a	0.142	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000376	Rad		15585-10-1	Europium-154	µCi/mL	n/a	<7.74E-04	<3.50E-03	<2.77E-03	n/a	n/a	n/a	3.50E-03	n/a	U
S16R000377	Rad		15585-10-1	Europium-154	µCi/mL	n/a	<7.74E-04	<8.73E-03	n/a	n/a	n/a	n/a	8.73E-03	n/a	U
S16R000376	Rad		14391-16-3	Europium-155	µCi/mL	n/a	<5.21E-04	<0.0864	<0.0396	n/a	n/a	n/a	0.0864	n/a	U
S16R000377	Rad		14391-16-3	Europium-155	µCi/mL	n/a	<5.21E-04	<0.163	n/a	n/a	n/a	n/a	0.163	n/a	U
S16R000376	Rad		ALPHA	Gross alpha	µCi/mL	101	<4.60E-04	8.83E-04	n/a	n/a	n/a	n/a	4.60E-04	118.716	J
S16R000377	Rad		ALPHA	Gross alpha	µCi/mL	101	<4.60E-04	1.26E-03	9.30E-04	1.09E-	29.8	98.1	4.60E-04	72.705	J
S16R000376	Rad		15046-84-1	Iodine-129	µCi/mL	92.9	<9.81E-06	1.04E-04	1.18E-04	1.11E-	12.7	n/a	1.62E-05	11.9	--
S16R000377	Rad		15046-84-1	Iodine-129	µCi/mL	92.9	<9.81E-06	1.22E-04	n/a	n/a	n/a	n/a	9.86E-06	10.5	--
S16R000376	Rad		15092-94-1	Lead-212	µCi/mL	n/a	<5.96E-04	<0.0541	<0.0458	n/a	n/a	n/a	0.0541	n/a	U
S16R000377	Rad		15092-94-1	Lead-212	µCi/mL	n/a	<5.96E-04	<0.116	n/a	n/a	n/a	n/a	0.116	n/a	U
S16R000376	Rad		15067-28-4	Lead-214	µCi/mL	n/a	<4.65E-04	<0.0482	<0.0349	n/a	n/a	n/a	0.0482	n/a	U
S16R000377	Rad		15067-28-4	Lead-214	µCi/mL	n/a	<4.65E-04	<0.108	n/a	n/a	n/a	n/a	0.108	n/a	U
S16R000376	Rad		13994-20-2	Neptunium-237	µg/mL	109	<1.12E-07	8.65E-03	7.88E-03	8.27E-	9.40	102	5.60E-05	n/a	--
S16R000377	Rad		13994-20-2	Neptunium-237	µg/mL	109	<1.12E-07	7.85E-03	n/a	n/a	n/a	n/a	5.60E-05	n/a	--
S16R000376	Rad		13981-37-8	Nickel-63	µCi/mL	94.5	<3.54E-06	0.0190	0.0197	0.0194	3.62	n/a	3.06E-06	0.404	--
S16R000377	Rad		13981-37-8	Nickel-63	µCi/mL	94.5	<3.54E-06	0.0195	n/a	n/a	n/a	n/a	3.14E-06	0.405	--
S16R000376	Rad		14681-63-1	Niobium-94	µCi/mL	n/a	<3.41E-04	<2.35E-03	<1.54E-03	n/a	n/a	n/a	2.35E-03	n/a	U
S16R000377	Rad		14681-63-1	Niobium-94	µCi/mL	n/a	<3.41E-04	<3.41E-03	n/a	n/a	n/a	n/a	3.41E-03	n/a	U
S16R000376	Rad		13981-16-3	Plutonium-238	µCi/mL	n/a	6.98E-07	9.96E-05	1.09E-04	1.04E-	8.74	n/a	5.07E-07	2.04	--
S16R000377	Rad		13981-16-3	Plutonium-238	µCi/mL	n/a	6.98E-07	1.07E-04	n/a	n/a	n/a	n/a	4.86E-07	1.91	--
S16R000376	Rad		PU-239/240	Plutonium-239/240	µCi/mL	116	<6.60E-07	1.16E-03	1.28E-03	1.22E-	9.73	n/a	5.53E-07	0.59	--
S16R000377	Rad		PU-239/240	Plutonium-239/240	µCi/mL	116	<6.60E-07	1.21E-03	n/a	n/a	n/a	n/a	4.32E-07	0.56	--
S16R000376	Rad		14119-32-5	Plutonium-241	µCi/mL	101	<5.05E-05	9.67E-04	9.67E-04	9.67E-	0.0	n/a	2.83E-05	5.903	--
S16R000377	Rad		14119-32-5	Plutonium-241	µCi/mL	101	<5.05E-05	8.97E-04	n/a	n/a	n/a	n/a	2.81E-05	6.183	--
S16R000376	Rad		13982-10-0	Plutonium-242	µCi/mL	n/a	<4.09E-07	1.80E-05	6.80E-06	1.24E-	90.1	n/a	2.67E-07	4.79	Y
S16R000377	Rad		13982-10-0	Plutonium-242	µCi/mL	n/a	<4.09E-07	4.26E-06	n/a	n/a	n/a	n/a	1.89E-07	9.52	Y
S16R000376	Rad		13966-00-2	Potassium-40	µCi/mL	n/a	<3.59E-03	<0.0253	<0.0219	n/a	n/a	n/a	0.0253	n/a	U
S16R000377	Rad		13966-00-2	Potassium-40	µCi/mL	n/a	<3.59E-03	<0.0545	n/a	n/a	n/a	n/a	0.0545	n/a	U
S16R000376	Rad		13982-63-3	Radium-226	µCi/mL	n/a	<4.61E-03	<0.405	<0.266	n/a	n/a	n/a	0.405	n/a	U
S16R000377	Rad		13982-63-3	Radium-226	µCi/mL	n/a	<4.61E-03	<1.24	n/a	n/a	n/a	n/a	1.24	n/a	U
S16R000376	Rad		RU/RH-106	Ruthenium/Rhodium-	µCi/mL	n/a	<1.92E-03	<0.212	<0.132	n/a	n/a	n/a	0.212	n/a	U
S16R000377	Rad		RU/RH-106	Ruthenium/Rhodium-	µCi/mL	n/a	<1.92E-03	<0.297	n/a	n/a	n/a	n/a	0.297	n/a	U
S16R000376	Rad		SR-89/90	Strontium-89/90	µCi/mL	105	1.48E-03	0.0801	0.0815	0.0808	1.76	n/a	7.86E-04	6.278	--
S16R000377	Rad		SR-89/90	Strontium-89/90	µCi/mL	105	1.48E-03	0.0817	n/a	n/a	n/a	n/a	7.79E-04	6.165	--
S16R000376	Rad		14133-76-7	Technetium-99	µCi/mL	105	<3.77E-05	0.0496	0.0530	0.0513	6.60	n/a	3.40E-05	0.74	--
S16R000377	Rad		14133-76-7	Technetium-99	µCi/mL	105	<3.77E-05	0.0449	n/a	n/a	n/a	n/a	3.03E-05	0.74	--
S16R000376	Rad		14913-50-9	Thallium-208	µCi/mL	n/a	<2.06E-04	<0.0319	<0.0101	n/a	n/a	n/a	0.0319	n/a	U
S16R000377	Rad		14913-50-9	Thallium-208	µCi/mL	n/a	<2.06E-04	<0.0344	n/a	n/a	n/a	n/a	0.0344	n/a	U

**RPP-RPT-59874**  
**Appendix D**

**Table D-1. Pre-Column Aqueous Composite Data Summary Report. (9 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike %	Det Limit	Count Error	Qual Flag
S16R000376	Rad		14274-82-9	Thorium-228	$\mu\text{Ci}/\text{mL}$	n/a	<0.105	<12.0	<5.94	n/a	n/a	n/a	12.0	n/a	U
S16R000377	Rad		14274-82-9	Thorium-228	$\mu\text{Ci}/\text{mL}$	n/a	<0.105	<27.4	n/a	n/a	n/a	n/a	27.4	n/a	U
S16R000376	Rad		15594-54-4	Thorium-229	$\mu\text{g}/\text{mL}$	n/a	<1.12E-07	<0.0280	<0.0280	n/a	n/a	n/a	0.0280	n/a	IU
S16R000377	Rad		15594-54-4	Thorium-229	$\mu\text{g}/\text{mL}$	n/a	<1.12E-07	<0.0280	n/a	n/a	n/a	n/a	0.0280	n/a	IU
S16R000376	Rad		TH-232	Thorium-232	$\mu\text{g}/\text{mL}$	92.0	<3.21E-07	<0.0802	<0.0802	n/a	n/a	98.0	0.0802	n/a	U
S16R000377	Rad		TH-232	Thorium-232	$\mu\text{g}/\text{mL}$	92.0	<3.21E-07	0.272	n/a	n/a	n/a	n/a	0.0802	n/a	J
S16R000376	Rad		15065-10-8	Thorium-234	$\mu\text{Ci}/\text{mL}$	n/a	<4.35E-03	<1.72	<0.195	n/a	n/a	n/a	1.72	n/a	U
S16R000377	Rad		15065-10-8	Thorium-234	$\mu\text{Ci}/\text{mL}$	n/a	<4.35E-03	<0.496	n/a	n/a	n/a	n/a	0.496	n/a	U
S16R000376	Rad		10028-17-8	Tritium	$\mu\text{Ci}/\text{mL}$	92.4	<1.80E-05	1.36E-03	1.36E-03	1.36E-	0.0	102	1.80E-05	3.314	--
S16R000377	Rad		10028-17-8	Tritium	$\mu\text{Ci}/\text{mL}$	92.4	<1.80E-05	1.53E-03	n/a	n/a	n/a	n/a	1.80E-05	3.093	--
S16R000376	Rad		13968-55-3	Uranium-233	$\mu\text{g}/\text{mL}$	110	<5.00E-07	<2.50E-04	<2.50E-04	n/a	n/a	103	2.50E-04	n/a	U
S16R000377	Rad		13968-55-3	Uranium-233	$\mu\text{g}/\text{mL}$	110	<5.00E-07	3.33E-04	n/a	n/a	n/a	n/a	2.50E-04	n/a	J
S16R000376	Rad		13966-29-5	Uranium-234	$\mu\text{g}/\text{mL}$	n/a	2.34E-07	<0.0135	0.0235	n/a	n/a	n/a	0.0135	n/a	IU
S16R000377	Rad		13966-29-5	Uranium-234	$\mu\text{g}/\text{mL}$	n/a	2.34E-07	0.0324	n/a	n/a	n/a	n/a	0.0135	n/a	BIJ
S16R000376	Rad		15117-96-1	Uranium-235	$\mu\text{g}/\text{mL}$	91.2	1.96E-07	0.655	0.615	0.635	6.36	96.8	0.0135	n/a	B
S16R000377	Rad		15117-96-1	Uranium-235	$\mu\text{g}/\text{mL}$	91.2	1.96E-07	0.696	n/a	n/a	n/a	n/a	0.0135	n/a	B

B = Applied when the preparation blanks are either <EQL or  $\leq 5\%$  of the measured concentration in the sample.

I = Applied when the result was determined via indirect calibration.

J = Applied to results that are considered estimates. Some examples of when a "J" flag may be applied include (but are not limited to):

- Result with concentration  $\geq \text{MDL}$ , but  $<$  the EQL
- Radiochemical result with counting uncertainty  $> 30\%$
- An "unknown" constituent reported for an organic analysis.

U = Applied to analytes that were analyzed for, but were not detected, or were detected below the MDL

Y = Applied to results that require verbal descriptions or qualifying comments. This flag is used by the chemist, project coordinator, or other technical authority to identify data that is questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations.

a = Applied when the LCS has a percent recovery outside the customer or analytical method specified range. The "a" flag is not applied based on LCSD results.

b = Applied when the MS is outside the acceptance criterion of 75-125%.

c = Applied when the RPD between duplicate samples, LCSDs, or MSDs, is  $\geq 20\%$ . The exception is for ICP analysis on fusion digests, where the ICP RPD acceptance criterion for solids is  $\leq 35\%$ .

e = Applied when the percent difference between the serial dilution and sample is  $\geq 10\%$ .

## RPP-RPT-59874

## Appendix D

Table D-2. Post-Column Aqueous Composite Data Summary Report. (5 pages)

Sample Group: 20162967

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000384	Inorg.		71-50-1	Acetate	µg/mL	94.2	<0.0100	562	566	564	0.736	98.6	5.00	n/a	--
S16R000384	Inorg.		7429-90-5	Aluminum	µg/mL	101	<0.0140	3.90E+03	3.87E+03	3.89E+03	0.663	97.2	14.0	n/a	--
S16R000384	Inorg.		7440-36-0	Antimony	µg/mL	98.9	<0.0180	<0.180	<0.180	n/a	n/a	87.7	0.180	n/a	U
S16R000384	Inorg.		7440-38-2	Arsenic	µg/mL	103	<0.0150	<1.50	<1.50	n/a	n/a	94.7	1.50	n/a	U
S16R000384	Inorg.		7440-39-3	Barium	µg/mL	104	<1.00E-03	0.353	0.317	0.335	10.9	90.3	0.100	n/a	J
S16R000384	Inorg.		7440-41-7	Beryllium	µg/mL	101	<1.00E-03	0.330	0.326	0.328	1.06	88.8	0.100	n/a	J
S16R000384	Inorg.		7440-69-9	Bismuth	µg/mL	102	<0.0190	<1.90	<1.90	n/a	n/a	89.0	1.90	n/a	U
S16R000384	Inorg.		7440-42-8	Boron	µg/mL	104	<2.00E-03	10.0	9.93	9.98	1.13	86.8	0.200	n/a	e
S16R000384	Inorg.		24959-67-9	Bromide	µg/mL	96.6	<6.00E-03	41.7	40.7	41.2	2.41	93.9	3.00	n/a	J
S16R000384	Inorg.		7440-43-9	Cadmium	µg/mL	102	<1.00E-03	0.657	0.659	0.658	0.434	88.2	0.100	n/a	J
S16R000384	Inorg.		7440-70-2	Calcium	µg/mL	103	<0.120	<12.0	<12.0	n/a	n/a	89.3	12.0	n/a	U
S16R000384	Inorg.		7440-45-1	Cerium	µg/mL	103	<0.0250	<2.50	<2.50	n/a	n/a	85.8	2.50	n/a	U
S16R000384	Inorg.		7440-46-2	Cesium	µg/mL	106	<3.00E-06	0.0230	0.0238	0.0234	3.51	83.8	6.00E-04	n/a	e
S16R000384	Inorg.		16887-00-6	Chloride	µg/mL	95.2	<4.00E-03	1.24E+03	1.25E+03	1.24E+03	0.799	102	2.00	n/a	--
S16R000384	Inorg.		7440-47-3	Chromium	µg/mL	103	<2.00E-03	231	231	231	0.248	87.1	0.200	n/a	--
S16R000384	Inorg.		7440-48-4	Cobalt	µg/mL	104	<1.00E-03	1.33	1.37	1.35	3.05	89.8	0.100	n/a	--
S16R000384	Inorg.		7440-50-8	Copper	µg/mL	103	<2.00E-03	0.485	0.390	0.437	21.9	87.8	0.200	n/a	J
S16R000384	Inorg.		Density	Density	g/mL	99.86	n/a	1.192	n/a	n/a	n/a	n/a	1.000E-	n/a	--
S16R000384	Inorg.		7440-53-1	Europium	µg/mL	104	<1.00E-03	<0.100	<0.100	n/a	n/a	88.4	0.100	n/a	U
S16R000384	Inorg.		16984-48-8	Fluoride	µg/mL	96.3	<1.00E-03	1.92E+03	2.03E+03	1.97E+03	5.67	98.5	5.00	n/a	--
S16R000384	Inorg.		12311-97-6	Formate	µg/mL	95.3	<7.00E-03	650	656	653	0.820	99.6	3.50	n/a	--
S16R000384	Inorg.		666-14-8	Glycolate	µg/mL	95.4	<5.00E-03	163	170	166	4.39	99.9	2.50	n/a	--
S16R000384	Inorg.		HYDROXIDE	Hydroxide	µg/mL	104	<9.22	2.43E+04	2.31E+04	2.37E+04	5.38	109	553	n/a	--
S16R000384	Inorg.		7439-89-6	Iron	µg/mL	102	<0.0200	89.6	89.9	89.7	0.255	86.7	2.00	n/a	--
S16R000384	Inorg.		7439-91-0	Lanthanum	µg/mL	102	<1.00E-03	<0.100	<0.100	n/a	n/a	86.3	0.100	n/a	U
S16R000384	Inorg.		7439-92-1	Lead	µg/mL	101	<0.0130	<1.30	<1.30	n/a	n/a	88.8	1.30	n/a	U
S16R000384	Inorg.		7439-93-2	Lithium	µg/mL	103	<1.00E-03	<0.100	<0.100	n/a	n/a	86.0	0.100	n/a	U
S16R000384	Inorg.		7439-95-4	Magnesium	µg/mL	101	<9.00E-03	<0.900	<0.900	n/a	n/a	88.1	0.900	n/a	U
S16R000384	Inorg.		7439-96-5	Manganese	µg/mL	103	<1.00E-03	<0.100	<0.100	n/a	n/a	87.8	0.100	n/a	U
S16R000386	Inorg.	HG	7439-97-6	Mercury	µg/mL	96.6	1.20E-05	0.118	n/a	n/a	n/a	n/a	4.40E-04	n/a	J
S16R000384	Inorg.		7439-98-7	Molybdenum	µg/mL	99.4	<2.00E-03	16.6	16.6	16.6	0.0314	88.2	0.200	n/a	--
S16R000384	Inorg.		7440-00-8	Neodymium	µg/mL	102	<0.0150	<1.50	<1.50	n/a	n/a	86.9	1.50	n/a	U
S16R000396	Inorg.		13994-20-2	Neptunium-237	µg/mL	101	<1.12E-07	7.83E-03	7.51E-03	7.67E-03	4.20	99.6	5.60E-05	n/a	--
S16R000384	Inorg.		7440-02-0	Nickel	µg/mL	104	<2.00E-03	34.6	34.7	34.6	0.201	89.7	0.200	n/a	--
S16R000384	Inorg.		7440-30-1	Niobium	µg/mL	102	<6.00E-03	<0.600	<0.600	n/a	n/a	91.2	0.600	n/a	U
S16R000384	Inorg.		14797-55-8	Nitrate	µg/mL	96.7	<0.0210	5.36E+04	5.65E+04	5.50E+04	5.26	96.7	105	n/a	--
S16R000384	Inorg.		14797-65-0	Nitrite	µg/mL	96.2	<9.00E-03	2.99E+04	3.15E+04	3.07E+04	5.42	97.9	45.0	n/a	--
S16R000384	Inorg.		338-70-5	Oxalate	µg/mL	95.3	<9.00E-03	949	956	953	0.803	95.6	4.50	n/a	--

**RPP-RPT-59874**  
**Appendix D**

**Table D-2. Post-Column Aqueous Composite Data Summary Report. (5 pages)**

Sample Group: 20162967

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000384	Inorg.		7440-05-3	Palladium	µg/mL	104	<0.0120	1.79	<1.20	n/a	n/a	84.5	1.20	n/a	J
S16R000384	Inorg.		%WATER	Percent water	%	99.6	n/a	77.7	78.2	78.0	0.577	n/a	0.0100	n/a	--
S16R000384	Inorg.		14265-44-2	Phosphate	µg/mL	96.1	<0.0140	2.90E+03	2.91E+03	2.91E+03	0.158	96.4	7.00	n/a	--
S16R000384	Inorg.		7723-14-0	Phosphorus	µg/mL	99.4	<0.0130	857	858	857	0.144	86.8	1.30	n/a	--
S16R000384	Inorg.		7440-09-7	Potassium	µg/mL	102	<0.0220	745	745	745	0.0824	85.1	2.20	n/a	--
S16R000384	Inorg.		7440-10-0	Praseodymium	µg/mL	104	<0.0260	<2.60	<2.60	n/a	n/a	83.8	2.60	n/a	U
S16R000384	Inorg.		7440-16-6	Rhodium	µg/mL	102	<0.0120	1.38	1.74	1.56	23.4	87.5	1.20	n/a	J
S16R000384	Inorg.		7440-17-7	Rubidium	µg/mL	103	<0.0570	<5.70	<5.70	n/a	n/a	89.5	5.70	n/a	U
S16R000384	Inorg.		7440-18-8	Ruthenium	µg/mL	107	<5.00E-03	4.11	4.21	4.16	2.40	85.2	0.500	n/a	J
S16R000384	Inorg.		7440-19-9	Samarium	µg/mL	103	<0.0170	<1.70	<1.70	n/a	n/a	85.7	1.70	n/a	U
S16R000384	Inorg.		7782-49-2	Selenium	µg/mL	102	<0.0300	<3.00	<3.00	n/a	n/a	94.3	3.00	n/a	U
S16R000384	Inorg.		7440-21-3	Silicon	µg/mL	102	<0.0120	34.7	33.8	34.3	2.67	98.9	1.20	n/a	--
S16R000384	Inorg.		7440-22-4	Silver	µg/mL	102	<3.00E-03	12.8	12.8	12.8	0.0705	88.4	3.00	n/a	--
S16R000384	Inorg.		7440-23-5	Sodium	µg/mL	102	<0.0920	1.00E+05	9.89E+04	9.95E+04	1.09	94.0	92.0	n/a	--
S16R000384	Inorg.		7440-24-6	Strontium	µg/mL	103	<2.00E-03	<0.200	<0.200	n/a	n/a	90.0	0.200	n/a	U
S16R000384	Inorg.		14808-79-8	Sulfate	µg/mL	96.7	<9.00E-03	4.17E+03	4.20E+03	4.19E+03	0.742	100	4.50	n/a	--
S16R000384	Inorg.		7704-34-9	Sulfur	µg/mL	101	<0.0280	1.56E+03	1.55E+03	1.56E+03	0.293	86.3	2.80	n/a	--
S16R000384	Inorg.		7440-25-7	Tantalum	µg/mL	105	<5.00E-03	<0.500	<0.500	n/a	n/a	93.4	0.500	n/a	U
S16R000384	Inorg.		13494-80-9	Tellurium	µg/mL	104	<9.00E-03	<0.900	<0.900	n/a	n/a	92.4	0.900	n/a	U
S16R000384	Inorg.		7440-28-0	Thallium	µg/mL	106	<0.0150	<1.50	<1.50	n/a	n/a	84.7	1.50	n/a	U
S16R000384	Inorg.		7772-98-7	Thiosulfate	µg/mL	98.6	<6.00E-03	<3.00	<3.00	n/a	n/a	99.5	3.00	n/a	U
S16R000384	Inorg.		7440-29-1	Thorium	µg/mL	103	<0.0130	<1.30	<1.30	n/a	n/a	86.4	1.30	n/a	U
S16R000396	Inorg.		15594-54-4	Thorium-229	µg/mL	n/a	<1.12E-07	<0.0280	<0.0280	n/a	n/a	n/a	0.0280	n/a	IU
S16R000396	Inorg.		TH-232	Thorium-232	µg/mL	97.4	<3.21E-07	<0.0802	<0.0802	n/a	n/a	97.6	0.0802	n/a	U
S16R000384	Inorg.		TIC	TIC	µg/mL	95.8	<7.00	5.03E+03	n/a	n/a	n/a	n/a	14.0	n/a	--
S16R000384	Inorg.		7440-31-5	Tin	µg/mL	99.9	<8.00E-03	8.75	8.93	8.84	2.06	86.5	0.800	n/a	--
S16R000384	Inorg.		7440-32-6	Titanium	µg/mL	99.5	<2.00E-03	<0.200	<0.200	n/a	n/a	88.0	0.200	n/a	U
S16R000384	Inorg.		TOC	TOC	µg/mL	92.1	<20.0	1.33E+03	n/a	n/a	n/a	n/a	40.0	n/a	--
S16R000384	Inorg.		7440-33-7	Tungsten	µg/mL	103	<0.0160	21.6	21.8	21.7	1.21	90.0	1.60	n/a	--
S16R000396	Inorg.		13968-55-3	Uranium-233	µg/mL	101	<5.00E-07	<2.50E-04	<2.50E-04	n/a	n/a	100	2.50E-04	n/a	U
S16R000396	Inorg.		13966-29-5	Uranium-234	µg/mL	n/a	1.68E-07	<0.0135	0.0176	n/a	n/a	n/a	0.0135	n/a	IU
S16R000396	Inorg.		15117-96-1	Uranium-235	µg/mL	95.0	7.78E-08	0.640	0.666	0.653	4.00	102	0.0135	n/a	B
S16R000396	Inorg.		U-238	Uranium-238	µg/mL	97.5	<4.18E-07	26.0	25.8	25.9	0.772	98.3	0.104	n/a	--
S16R000384	Inorg.		7440-62-2	Vanadium	µg/mL	102	<1.00E-03	0.160	0.183	0.171	13.5	89.2	0.100	n/a	J
S16R000384	Inorg.		7440-65-5	Yttrium	µg/mL	103	<2.00E-03	<0.200	<0.200	n/a	n/a	88.7	0.200	n/a	U
S16R000384	Inorg.		7440-66-6	Zinc	µg/mL	103	<0.0320	<3.20	<3.20	n/a	n/a	91.8	3.20	n/a	U
S16R000384	Inorg.		7440-67-7	Zirconium	µg/mL	99.6	<1.00E-03	<0.100	0.101	n/a	n/a	87.0	0.100	n/a	U
S16R000394	VOA		71-55-6	1,1,1-Trichloroethane	µg/L	108	<0.0433	<1.73	n/a	n/a	n/a	n/a	1.73	n/a	U

## RPP-RPT-59874

## Appendix D

Table D-2. Post-Column Aqueous Composite Data Summary Report. (5 pages)

Sample Group: 20162967

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000394	VOA		76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	µg/L	99.6	<0.0396	<1.58	n/a	n/a	n/a	n/a	1.58	n/a	U
S16R000394	VOA		79-00-5	1,1,2-Trichloroethane	µg/L	104	<0.0230	<0.920	n/a	n/a	n/a	n/a	0.920	n/a	U
S16R000394	VOA		75-35-4	1,1-Dichloroethene	µg/L	101	<0.172	<6.87	n/a	n/a	n/a	n/a	6.87	n/a	U
S16R000394	VOA		95-50-1	1,2-Dichlorobenzene	µg/L	103	<0.0455	<1.82	n/a	n/a	n/a	n/a	1.82	n/a	U
S16R000394	VOA		107-06-2	1,2-Dichloroethane	µg/L	110	<0.0243	<0.972	n/a	n/a	n/a	n/a	0.972	n/a	U
S16R000394	VOA		71-36-3	1-Butanol	µg/L	111	<1.15	1.34E+03	n/a	n/a	n/a	n/a	46.0	n/a	b
S16R000394	VOA		78-93-3	2-Butanone	µg/L	111	<0.458	<18.3	n/a	n/a	n/a	n/a	18.3	n/a	Ub
S16R000394	VOA		79-46-9	2-Nitropropane	µg/L	103	<0.265	<10.6	n/a	n/a	n/a	n/a	10.6	n/a	U
S16R000394	VOA		108-10-1	4-Methyl-2-Pentanone	µg/L	108	<0.453	<18.1	n/a	n/a	n/a	n/a	18.1	n/a	Ub
S16R000394	VOA		67-64-1	Acetone	µg/L	106	<0.573	209	n/a	n/a	n/a	n/a	22.9	n/a	J
S16R000394	VOA		71-43-2	Benzene	µg/L	104	<0.0163	<0.652	n/a	n/a	n/a	n/a	0.652	n/a	U
S16R000394	VOA		75-15-0	Carbon disulfide	µg/L	96.9	<0.0211	<0.844	n/a	n/a	n/a	n/a	0.844	n/a	U
S16R000394	VOA		56-23-5	Carbon tetrachloride	µg/L	107	<0.0479	<1.92	n/a	n/a	n/a	n/a	1.92	n/a	U
S16R000394	VOA		108-90-7	Chlorobenzene	µg/L	102	<0.0320	<1.28	n/a	n/a	n/a	n/a	1.28	n/a	U
S16R000394	VOA		67-66-3	Chloroform	µg/L	111	<0.0414	<1.66	n/a	n/a	n/a	n/a	1.66	n/a	U
S16R000394	VOA		60-29-7	Diethyl ether	µg/L	107	<0.0423	<1.69	n/a	n/a	n/a	n/a	1.69	n/a	U
S16R000394	VOA		141-78-6	Ethyl acetate	µg/L	110	<0.183	<7.34	n/a	n/a	n/a	n/a	7.34	n/a	U
S16R000394	VOA		100-41-4	Ethylbenzene	µg/L	97.1	<0.0334	<1.34	n/a	n/a	n/a	n/a	1.34	n/a	U
S16R000394	VOA		67-72-1	Hexachloroethane	µg/L	106	<0.111	<4.44	n/a	n/a	n/a	n/a	4.44	n/a	U
S16R000394	VOA		75-09-2	Methylene Chloride	µg/L	106	<0.0396	<1.58	n/a	n/a	n/a	n/a	1.58	n/a	U
S16R000394	VOA		95-47-6	o-Xylene	µg/L	102	<0.0325	<1.30	n/a	n/a	n/a	n/a	1.30	n/a	U
S16R000394	VOA		127-18-4	Tetrachloroethene	µg/L	100	<0.0583	<2.33	n/a	n/a	n/a	n/a	2.33	n/a	U
S16R000394	VOA		108-88-3	Toluene	µg/L	100	0.268	10.7	n/a	n/a	n/a	n/a	2.09	n/a	BJb
S16R000394	VOA		79-01-6	Trichloroethene	µg/L	106	<0.0378	<1.51	n/a	n/a	n/a	n/a	1.51	n/a	U
S16R000394	VOA		75-69-4	Trichlorofluoromethane	µg/L	103	<0.0458	<1.83	n/a	n/a	n/a	n/a	1.83	n/a	U
S16R000394	VOA		75-01-4	Vinyl chloride	µg/L	97.9	<0.0486	<1.94	n/a	n/a	n/a	n/a	1.94	n/a	U
S16R000394	VOA		106-42-3	Xylene (m & p)	µg/L	96.1	<0.0703	<2.81	n/a	n/a	n/a	n/a	2.81	n/a	U
S16R000390	SVOA	O	95-95-4	2,4,5-Trichlorophenol	µg/L	86.6	<686	<686	<686	n/a	n/a	0.0	686	n/a	Ub
S16R000505	SVOA	O	95-95-4	2,4,5-Trichlorophenol	µg/L	72.9	<39.2	<196	<196	n/a	n/a	0.0	196	n/a	Ub
S16R000390	SVOA	O	121-14-2	2,4-Dinitrotoluene	µg/L	86.1	<364	<364	<364	n/a	n/a	86.0	364	n/a	U
S16R000505	SVOA	O	121-14-2	2,4-Dinitrotoluene	µg/L	72.0	<20.8	<104	<104	n/a	n/a	82.0	104	n/a	U
S16R000390	SVOA	O	110-80-5	2-Ethoxyethanol	µg/L	81.4	<932	<932	<932	n/a	n/a	70.4	932	n/a	U
S16R000505	SVOA	O	110-80-5	2-Ethoxyethanol	µg/L	64.8	<53.3	<266	<266	n/a	n/a	71.9	266	n/a	Ua
S16R000390	SVOA	O	95-48-7	2-Methylphenol	µg/L	84.2	<631	<631	<631	n/a	n/a	18.5	631	n/a	Ub
S16R000505	SVOA	O	95-48-7	2-Methylphenol	µg/L	71.9	<36.1	<180	<180	n/a	n/a	0.0	180	n/a	Ub
S16R000390	SVOA	O	108-39-4M	Cresol (m & p)	µg/L	84.3	<562	<562	<562	n/a	n/a	0.0	562	n/a	Ub
S16R000505	SVOA	O	108-39-4M	Cresol (m & p)	µg/L	75.1	<32.1	<161	<161	n/a	n/a	0.0	161	n/a	Ub

## RPP-RPT-59874

## Appendix D

Table D-2. Post-Column Aqueous Composite Data Summary Report. (5 pages)

Sample Group: 20162967

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000390	SVOA	O	108-94-1	Cyclohexanone	µg/L	87.8	<651	<651	<651	n/a	n/a	85.2	651	n/a	U
S16R000505	SVOA	O	108-94-1	Cyclohexanone	µg/L	74.3	<37.2	<186	<186	n/a	n/a	64.3	186	n/a	Ub
S16R000390	SVOA	O	87-68-3	Hexachlorobutadiene	µg/L	16.9	<751	<751	<751	n/a	n/a	0.0	751	n/a	Ub
S16R000505	SVOA	O	87-68-3	Hexachlorobutadiene	µg/L	17.6	<42.9	<215	<215	n/a	n/a	0.0	215	n/a	Ub
S16R000390	SVOA	O	67-72-1	Hexachloroethane	µg/L	11.1	<700	<700	<700	n/a	n/a	11.0	700	n/a	Ub
S16R000505	SVOA	O	67-72-1	Hexachloroethane	µg/L	0.0	<40.0	<200	<200	n/a	n/a	0.0	200	n/a	Ub
S16R000390	SVOA	O	78-83-1	Isobutyl alcohol	µg/L	36.9	<1.08E+03	<1.08E+03	<1.08E+03	n/a	n/a	43.0	1.08E+03	n/a	Ub
S16R000505	SVOA	O	78-83-1	Isobutyl alcohol	µg/L	30.8	<61.6	<308	<308	n/a	n/a	0.0	308	n/a	Ub
S16R000390	SVOA	O	98-95-3	Nitrobenzene	µg/L	79.8	<609	<609	<609	n/a	n/a	185	609	n/a	Ub
S16R000505	SVOA	O	98-95-3	Nitrobenzene	µg/L	70.8	<34.8	<174	<174	n/a	n/a	125	174	n/a	U
S16R000390	SVOA	O	110-86-1	Pyridine	µg/L	74.9	<731	<731	<731	n/a	n/a	12.3	731	n/a	Ub
S16R000505	SVOA	O	110-86-1	Pyridine	µg/L	62.8	<41.8	<209	<209	n/a	n/a	0.0	209	n/a	Uab
S16R000392	PCB	O	12674-11-2	Aroclor 1016	µg/L	n/a	<94.5	<94.5	<94.5	n/a	n/a	n/a	94.5	n/a	U
S16R000392	PCB	O	11104-28-2	Aroclor 1221	µg/L	n/a	<17.8	<17.8	<17.8	n/a	n/a	n/a	17.8	n/a	U
S16R000392	PCB	O	11141-16-5	Aroclor 1232	µg/L	n/a	<20.5	<20.5	<20.5	n/a	n/a	n/a	20.5	n/a	U
S16R000392	PCB	O	53469-21-9	Aroclor 1242	µg/L	n/a	<31.5	<31.5	<31.5	n/a	n/a	n/a	31.5	n/a	U
S16R000392	PCB	O	12672-29-6	Aroclor 1248	µg/L	n/a	<17.8	<17.8	<17.8	n/a	n/a	n/a	17.8	n/a	U
S16R000392	PCB	O	11097-69-1	Aroclor 1254	µg/L	72.1	<6.50	<6.50	<6.50	n/a	n/a	81.5	6.50	n/a	U
S16R000392	PCB	O	11096-82-5	Aroclor 1260	µg/L	n/a	<71.5	<71.5	<71.5	n/a	n/a	n/a	71.5	n/a	U
S16R000396	Rad		14331-83-0	Actinium-228	µCi/mL	n/a	<1.18E-04	<2.11E-04	<1.47E-04	n/a	n/a	n/a	2.11E-04	n/a	U
S16R000396	Rad		14596-10-2	Americium-241	µCi/mL	n/a	<2.21E-04	<4.04E-05	<1.32E-04	n/a	n/a	n/a	4.04E-05	n/a	U
S16R000396	Rad		14596-10-2	Americium-241	µCi/mL	97.3	<4.67E-07	1.68E-05	n/a	n/a	n/a	n/a	2.38E-07	4.95	--
S16R000396	Rad		14234-35-6	Antimony-125	µCi/mL	114	<6.73E-05	<1.09E-04	<3.19E-04	n/a	n/a	n/a	1.09E-04	n/a	U
S16R000396	Rad		14913-49-6	Bismuth-212	µCi/mL	n/a	<3.91E-04	<8.76E-04	<1.32E-03	n/a	n/a	n/a	8.76E-04	n/a	U
S16R000396	Rad		14733-03-0	Bismuth-214	µCi/mL	n/a	<5.81E-05	<1.50E-04	<1.15E-04	n/a	n/a	n/a	1.50E-04	n/a	U
S16R000396	Rad		14762-75-5	Carbon-14	µCi/mL	99.2	<7.96E-07	5.76E-04	5.77E-04	5.76E-04	0.173	98.6	7.96E-07	0.897	--
S16R000396	Rad		13967-70-9	Cesium-134	µCi/mL	n/a	<1.95E-04	<2.31E-04	<1.79E-04	n/a	n/a	n/a	2.31E-04	n/a	U
S16R000396	Rad		10045-97-3	Cesium-137	µCi/mL	97.4	<3.27E-05	1.64E-04	1.82E-04	1.73E-04	10.1	n/a	3.86E-05	8.50	--
S16R000396	Rad		10198-40-0	Cobalt-60	µCi/mL	98.4	<3.70E-05	9.37E-04	9.68E-04	9.52E-04	3.27	n/a	3.36E-05	2.55	--
S16R000396	Rad		15510-73-3	Curium-242	µCi/mL	n/a	<1.01E-07	2.15E-07	n/a	n/a	n/a	n/a	1.17E-07	45.22	J
S16R000396	Rad		CM-243/244	Curium-243/244	µCi/mL	n/a	<2.89E-07	3.95E-07	n/a	n/a	n/a	n/a	1.19E-07	33.54	J
S16R000388	Rad		NONE	Dose Rate	mRem/hr	n/a	n/a	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	--
S16R000384	Rad		NONE	Dose Rate	mRem/hr	n/a	n/a	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	--
S16R000396	Rad		14683-23-9	Europium-152	µCi/mL	n/a	<7.11E-05	<9.66E-05	<1.96E-04	n/a	n/a	n/a	9.66E-05	n/a	U
S16R000396	Rad		15585-10-1	Europium-154	µCi/mL	n/a	<8.51E-05	<1.54E-04	<1.27E-04	n/a	n/a	n/a	1.54E-04	n/a	U
S16R000396	Rad		14391-16-3	Europium-155	µCi/mL	n/a	<7.28E-05	<5.70E-05	<2.03E-04	n/a	n/a	n/a	5.70E-05	n/a	U
S16R000396	Rad		ALPHA	Gross alpha	µCi/mL	101	<4.60E-04	2.54E-04	n/a	n/a	n/a	n/a	6.53E-07	4.479	--
S16R000396	Rad		15046-84-1	Iodine-129	µCi/mL	97.8	<9.43E-06	1.16E-04	1.28E-04	1.22E-04	9.92	n/a	1.45E-05	11.2	--

**RPP-RPT-59874**  
**Appendix D**

**Table D-2. Post-Column Aqueous Composite Data Summary Report. (5 pages)**

Sample Group: 20162967

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000396	Rad		15092-94-1	Lead-212	$\mu\text{Ci/mL}$	n/a	<6.02E-05	<8.28E-05	<1.54E-04	n/a	n/a	n/a	8.28E-05	n/a	U
S16R000396	Rad		15067-28-4	Lead-214	$\mu\text{Ci/mL}$	n/a	<5.44E-05	6.59E-05	<1.46E-04	n/a	n/a	n/a	5.78E-05	29.4	--
S16R000396	Rad		13981-37-8	Nickel-63	$\mu\text{Ci/mL}$	101	<5.03E-06	0.0202	0.0196	0.0199	3.02	n/a	3.53E-06	0.405	--
S16R000396	Rad		14681-63-1	Niobium-94	$\mu\text{Ci/mL}$	n/a	<3.17E-05	<7.51E-05	<6.85E-05	n/a	n/a	n/a	7.51E-05	n/a	U
S16R000396	Rad		13981-16-3	Plutonium-238	$\mu\text{Ci/mL}$	n/a	6.98E-07	9.92E-05	n/a	n/a	n/a	n/a	5.46E-07	2.04	--
S16R000396	Rad		PU-239/240	Plutonium-239/240	$\mu\text{Ci/mL}$	116	<6.60E-07	1.20E-03	n/a	n/a	n/a	n/a	5.44E-07	0.59	--
S16R000396	Rad		14119-32-5	Plutonium-241	$\mu\text{Ci/mL}$	101	<5.05E-05	8.21E-04	n/a	n/a	n/a	n/a	2.76E-05	6.5	--
S16R000396	Rad		13982-10-0	Plutonium-242	$\mu\text{Ci/mL}$	n/a	<4.09E-07	4.24E-06	n/a	n/a	n/a	n/a	3.13E-07	9.94	Y
S16R000396	Rad		13966-00-2	Potassium-40	$\mu\text{Ci/mL}$	n/a	<6.58E-04	<6.28E-04	<7.61E-04	n/a	n/a	n/a	6.28E-04	n/a	U
S16R000396	Rad			RadChem Separation	unitless	n/a	n/a	Complete	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000396	Rad			RadChem Separation	unitless	n/a	n/a	Complete	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000396	Rad		13982-63-3	Radium-226	$\mu\text{Ci/mL}$	n/a	<4.23E-04	<5.28E-04	<1.46E-03	n/a	n/a	n/a	5.28E-04	n/a	U
S16R000396	Rad		RU/RH-106	Ruthenium/Rhodium-	$\mu\text{Ci/mL}$	n/a	<1.96E-04	<3.94E-04	<4.04E-04	n/a	n/a	n/a	3.94E-04	n/a	U
S16R000396	Rad		SR-89/90	Strontium-89/90	$\mu\text{Ci/mL}$	109	<1.23E-05	0.0740	0.0741	0.0740	0.159	n/a	1.07E-05	0.659	--
S16R000396	Rad		14133-76-7	Technetium-99	$\mu\text{Ci/mL}$	102	<5.36E-05	0.0447	0.0462	0.0455	3.24	n/a	3.19E-05	0.76	--
S16R000396	Rad		14913-50-9	Thallium-208	$\mu\text{Ci/mL}$	n/a	<2.92E-05	<6.54E-05	<7.30E-05	n/a	n/a	n/a	6.54E-05	n/a	U
S16R000396	Rad		14274-82-9	Thorium-228	$\mu\text{Ci/mL}$	n/a	<0.0241	<0.0197	<0.0391	n/a	n/a	n/a	0.0197	n/a	U
S16R000396	Rad		15065-10-8	Thorium-234	$\mu\text{Ci/mL}$	n/a	<1.63E-03	<8.10E-04	<6.73E-04	n/a	n/a	n/a	8.10E-04	n/a	U
S16R000396	Rad		15832-50-5	Tin-126	$\mu\text{Ci/mL}$	n/a	<2.76E-05	2.15E-04	2.08E-04	2.12E-04	3.67	n/a	2.57E-05	4.84	--
S16R000396	Rad		NONE	Dose Rate	mRem/hr	n/a	n/a	<0.500	n/a	n/a	n/a	n/a	0.500	n/a	--
S16R000396	Rad			Total Activity	$\mu\text{Ci/mL}$	106	<1.51E-06	0.189	0.192	0.191	1.67	n/a	1.51E-06	0.1	--
S16R000396	Rad		10028-17-8	Tritium	$\mu\text{Ci/mL}$	94.3	<1.99E-05	1.36E-03	1.37E-03	1.36E-03	0.733	97.5	1.67E-05	3.267	--

B = Applied when the preparation blanks are either <EQL or  $\leq 5\%$  of the measured concentration in the sample.

I = Applied when the result was determined via indirect calibration.

J = Applied to results that are considered estimates. Some examples of when a "J" flag may be applied include (but are not limited to):

- Result with concentration  $\geq \text{MDL}$ , but  $<$  the EQL
- Radiochemical result with counting uncertainty  $> 30\%$
- An "unknown" constituent reported for an organic analysis.

U = Applied to analytes that were analyzed for, but were not detected, or were detected below the MDL

Y = Applied to results that require verbal descriptions or qualifying comments. This flag is used by the chemist, project coordinator, or other technical authority to identify data that is questionable or may be inaccurate because of interferences, sampling problems, or instrumentation limitations.

a = Applied when the LCS has a percent recovery outside the customer or analytical method specified range. The "a" flag is not applied based on LCSD results.

b = Applied when the MS is outside the acceptance criterion of 75-125%.

c = Applied when the RPD between duplicate samples, LCSDs, or MSDs, is  $\geq 20\%$ . The exception is for ICP analysis on fusion digests, where the ICP RPD acceptance criterion for solids is  $\leq 35\%$ .

e = Applied when the percent difference between the serial dilution and sample is  $\geq 10\%$ .

**RPP-RPT-59874**  
**Appendix D**

**Table D-3. Pre-Column Solids Composite Data Summary Report. (4 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000399	Inorg.		%WATER	Percent water	%	100	n/a	30.7	28.7	29.7	6.75	n/a	0.0100	n/a	--
S16R000399	Inorg.		TIC	Total inorganic carbon	µg/g	95.2	<7.00	9.77E+03	9.73E+03	9.75E+03	0.410	93.1	59.8	n/a	--
S16R000399	Inorg.		TOC	Total organic carbon	µg/g	92.0	<20.0	1.85E+03	1.36E+03	1.60E+03	30.5	99.6	171	n/a	c
S16R000401	Inorg.	HG	7439-97-6	Mercury	µg/g	106	1.50E-05	96.2	117	106	19.3	264	0.220	n/a	b
S16R000405	Inorg.	A	7440-22-4	Silver	µg/g	90.8	<3.00E-03	23.5	28.9	26.2	20.7	106	5.42	n/a	J
S16R000405	Inorg.	A	7429-90-5	Aluminum	µg/g	90.0	<0.0140	6.47E+04	7.62E+04	7.04E+04	16.3	1280	25.3	n/a	--
S16R000405	Inorg.	A	7440-38-2	Arsenic	µg/g	89.4	<0.0150	<27.1	<27.2	n/a	n/a	91.5	27.1	n/a	U
S16R000405	Inorg.	A	7440-42-8	Boron	µg/g	90.6	3.64E-03	13.2	12.4	12.8	6.55	102	3.61	n/a	BJ
S16R000405	Inorg.	A	7440-39-3	Barium	µg/g	90.7	<1.00E-03	114	137	126	18.0	96.6	1.81	n/a	--
S16R000405	Inorg.	A	7440-41-7	Beryllium	µg/g	91.7	<1.00E-03	4.28	5.18	4.73	19.2	104	1.81	n/a	J
S16R000405	Inorg.	A	7440-69-9	Bismuth	µg/g	88.9	<0.0190	208	257	232	21.5	96.6	34.3	n/a	J
S16R000405	Inorg.	A	7440-70-2	Calcium	µg/g	99.8	<0.120	3.27E+03	3.90E+03	3.58E+03	17.7	138	217	n/a	b
S16R000405	Inorg.	A	7440-43-9	Cadmium	µg/g	93.4	<1.00E-03	30.8	36.0	33.4	15.6	99.4	1.81	n/a	--
S16R000405	Inorg.	A	7440-45-1	Cerium	µg/g	94.1	0.0693	191	154	173	21.5	96.4	45.1	n/a	BJ
S16R000405	Inorg.	A	7440-48-4	Cobalt	µg/g	92.0	<1.00E-03	3.47	4.19	3.83	18.8	96.2	1.81	n/a	J
S16R000405	Inorg.	A	7440-47-3	Chromium	µg/g	92.0	5.18E-03	356	423	389	17.1	110	3.61	n/a	--
S16R000405	Inorg.	A	7440-50-8	Copper	µg/g	91.2	<2.00E-03	53.1	64.8	59.0	19.9	103	3.61	n/a	--
S16R000405	Inorg.	A	7440-53-1	Europium	µg/g	92.0	<1.00E-03	<1.81	<1.81	n/a	n/a	97.5	1.81	n/a	U
S16R000405	Inorg.	A	7439-89-6	Iron	µg/g	93.6	<0.0200	9.56E+03	1.14E+04	1.05E+04	17.9	295	36.1	n/a	--
S16R000405	Inorg.	A	7440-09-7	Potassium	µg/g	89.9	<0.0220	317	400	359	23.2	111	39.7	n/a	J
S16R000405	Inorg.	A	7439-91-0	Lanthanum	µg/g	93.0	<1.00E-03	<1.81	<1.81	n/a	n/a	104	1.81	n/a	U
S16R000405	Inorg.	A	7439-93-2	Lithium	µg/g	95.5	<1.00E-03	2.39	3.84	3.11	46.7	105	1.81	n/a	J
S16R000405	Inorg.	A	7439-95-4	Magnesium	µg/g	93.0	<9.00E-03	798	949	874	17.3	120	16.3	n/a	--
S16R000405	Inorg.	A	7439-96-5	Manganese	µg/g	93.4	<1.00E-03	506	604	555	17.6	113	1.81	n/a	--
S16R000405	Inorg.	A	7439-98-7	Molybdenum	µg/g	93.0	<2.00E-03	7.45	9.55	8.50	24.7	96.7	3.61	n/a	J
S16R000405	Inorg.	A	7440-23-5	Sodium	µg/g	93.4	<0.0920	1.76E+05	1.90E+05	1.83E+05	7.49	1640	166	n/a	--
S16R000405	Inorg.	A	7440-00-8	Neodymium	µg/g	94.8	<0.0150	<27.1	<27.2	n/a	n/a	103	27.1	n/a	U
S16R000405	Inorg.	A	7440-02-0	Nickel	µg/g	89.9	<2.00E-03	7.14E+03	8.36E+03	7.75E+03	15.7	183	3.61	n/a	--
S16R000405	Inorg.	A	7440-30-1	Niobium	µg/g	89.2	<6.00E-03	<10.8	<10.9	n/a	n/a	32.0	10.8	n/a	Ub
S16R000405	Inorg.	A	7723-14-0	Phosphorus	µg/g	90.4	<0.0130	2.37E+04	1.83E+04	2.10E+04	25.5	-631	23.5	n/a	--
S16R000405	Inorg.	A	7439-92-1	Lead	µg/g	97.9	<0.0130	440	509	474	14.5	105	23.5	n/a	--
S16R000405	Inorg.	A	7440-05-3	Palladium	µg/g	92.5	<0.0120	<21.7	<21.7	n/a	n/a	93.7	21.7	n/a	U
S16R000405	Inorg.	A	7440-10-0	Praseodymium	µg/g	98.8	<0.0260	<46.9	<47.1	n/a	n/a	105	46.9	n/a	U
S16R000405	Inorg.	A	7440-17-7	Rubidium	µg/g	91.3	<0.0570	<103	<103	n/a	n/a	94.5	103	n/a	U
S16R000405	Inorg.	A	7440-16-6	Rhodium	µg/g	90.9	<0.0120	<21.7	<21.7	n/a	n/a	94.5	21.7	n/a	U
S16R000405	Inorg.	A	7440-18-8	Ruthenium	µg/g	92.5	<5.00E-03	<9.03	<9.05	n/a	n/a	94.9	9.03	n/a	U
S16R000405	Inorg.	A	7704-34-9	Sulfur	µg/g	90.3	0.0479	791	937	864	16.9	105	50.6	n/a	B
S16R000405	Inorg.	A	7440-36-0	Antimony	µg/g	88.6	<0.0180	<32.5	<32.6	n/a	n/a	84.1	32.5	n/a	U
S16R000405	Inorg.	A	7782-49-2	Selenium	µg/g	91.1	<0.0300	<54.2	<54.3	n/a	n/a	94.7	54.2	n/a	U

**RPP-RPT-59874**  
**Appendix D**

**Table D-3. Pre-Column Solids Composite Data Summary Report. (4 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000405	Inorg.	A	7440-21-3	Silicon	µg/g	87.8	0.0988	9.69E+02	1.11E+03	1.04E+03	13.3	148	21.7	n/a	Bb
S16R000405	Inorg.	A	7440-19-9	Samarium	µg/g	94.8	<0.0170	70.3	90.1	80.2	24.7	106	30.7	n/a	J
S16R000405	Inorg.	A	7440-31-5	Tin	µg/g	94.2	<8.00E-03	<14.4	<14.5	n/a	n/a	93.5	14.4	n/a	U
S16R000405	Inorg.	A	7440-24-6	Strontium	µg/g	91.3	<2.00E-03	60.4	72.3	66.4	17.9	95.4	3.61	n/a	--
S16R000405	Inorg.	A	7440-25-7	Tantalum	µg/g	92.9	<5.00E-03	<9.03	<9.05	n/a	n/a	0.0	9.03	n/a	Ub
S16R000405	Inorg.	A	13494-80-9	Tellurium	µg/g	88.7	<9.00E-03	<16.3	<16.3	n/a	n/a	89.9	16.3	n/a	U
S16R000405	Inorg.	A	7440-29-1	Thorium	µg/g	93.0	<0.0130	5.85E+03	7.07E+03	6.46E+03	18.9	239	23.5	n/a	--
S16R000405	Inorg.	A	7440-32-6	Titanium	µg/g	88.4	<2.00E-03	29.6	50.8	40.2	52.6	85.3	3.61	n/a	J
S16R000405	Inorg.	A	7440-28-0	Thallium	µg/g	90.2	<0.0150	<27.1	<27.2	n/a	n/a	86.9	27.1	n/a	U
S16R000405	Inorg.	A	7440-61-1	Uranium	µg/g	93.5	<0.0290	1.36E+04	1.69E+04	1.53E+04	21.6	436	52.4	n/a	--
S16R000405	Inorg.	A	7440-62-2	Vanadium	µg/g	89.1	<1.00E-03	8.96	7.66	8.31	15.7	97.9	1.81	n/a	J
S16R000405	Inorg.	A	7440-33-7	Tungsten	µg/g	93.5	<0.0160	<28.9	<29.0	n/a	n/a	86.1	28.9	n/a	U
S16R000405	Inorg.	A	7440-65-5	Yttrium	µg/g	92.6	<2.00E-03	7.86	11.0	9.44	33.5	100	3.61	n/a	J
S16R000405	Inorg.	A	7440-66-6	Zinc	µg/g	89.6	<0.0320	63.7	72.0	67.8	12.2	97.6	57.8	n/a	J
S16R000405	Inorg.	A	7440-67-7	Zirconium	µg/g	88.3	<1.00E-03	114	153	134	29.1	43.8	1.81	n/a	b
S16R000405	Inorg.	A	7440-46-2	Cesium	µg/g	102	<3.00E-04	4.04	4.69	4.36	14.9	97.7	0.0542	n/a	--
S16R000403	Inorg.	W	16984-48-8	Fluoride	µg/g	95.5	<1.00E-03	1.20E+04	1.22E+04	1.21E+04	1.37	-35.9	28.7	n/a	--
S16R000403	Inorg.	W	666-14-8	Glycolate	µg/g	94.8	<5.00E-03	<92.0	<93.4	n/a	n/a	99.6	92.0	n/a	U
S16R000403	Inorg.	W	71-50-1	Acetate	µg/g	97.1	<0.0100	292	318	305	8.66	99.6	161	n/a	J
S16R000403	Inorg.	W	12311-97-6	Formate	µg/g	94.8	<7.00E-03	499	503	501	0.697	89.5	241	n/a	J
S16R000403	Inorg.	W	16887-00-6	Chloride	µg/g	95.3	0.0110	519	536	528	3.19	106	92.0	n/a	B
S16R000403	Inorg.	W	14797-65-0	Nitrite	µg/g	97.8	0.0410	1.40E+04	1.46E+04	1.43E+04	4.03	145	402	n/a	b
S16R000403	Inorg.	W	14808-79-8	Sulfate	µg/g	103	0.0870	2.25E+03	2.27E+03	2.26E+03	0.994	99.9	264	n/a	B
S16R000403	Inorg.	W	338-70-5	Oxalate	µg/g	96.1	0.0220	475	494	484	3.86	108	149	n/a	BJ
S16R000403	Inorg.	W	24959-67-9	Bromide	µg/g	93.8	<6.00E-03	<86.2	<87.5	n/a	n/a	90.6	86.2	n/a	U
S16R000403	Inorg.	W	14797-55-8	Nitrate	µg/g	96.9	0.0920	2.76E+04	2.85E+04	2.81E+04	2.98	186	362	n/a	--
S16R000403	Inorg.	W	14265-44-2	Phosphate	µg/g	93.4	<0.0140	1.05E+05	1.07E+05	1.06E+05	1.37	-73.0	115	n/a	--
S16R000403	Inorg.	W	7772-98-7	Thiosulfate	µg/g	91.8	<6.00E-03	<149	<152	n/a	n/a	88.0	149	n/a	U
S16R000399	Rad.		NONE	Dose Rate	mRem/hr	n/a	n/a	316	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000405	Rad.	A	15594-54-4	Thorium-229	µg/g	n/a	<2.24E-06	<2.02	<2.03	n/a	n/a	n/a	2.02	n/a	IU
S16R000405	Rad.	A	TH-232	Thorium-232	µg/g	102	7.10E-05	6.50E+03	6.01E+03	6.26E+03	7.80	19500	5.80	n/a	--
S16R000405	Rad.	A	13968-55-3	Uranium-233	µg/g	95.0	<5.00E-05	6.97	7.79	7.38	11.1	102	0.903	n/a	J
S16R000405	Rad.	A	13966-29-5	Uranium-234	µg/g	n/a	<1.08E-06	<3.90	6.30	n/a	n/a	n/a	3.90	n/a	IU
S16R000405	Rad.	A	15117-96-1	Uranium-235	µg/g	96.7	8.64E-06	464	463	463	0.413	n/a	3.90	n/a	B
S16R000405	Rad.	A	13994-20-2	Neptunium-237	µg/g	101	<2.24E-06	0.900	1.03	0.966	13.6	247	0.202	n/a	J
S16R000405	Rad.	A	U-238	Uranium-238	µg/g	102	2.20E-04	1.64E+04	1.54E+04	1.59E+04	6.65	48300	7.55	n/a	B
S16R000407	Rad.		NONE	Dose Rate	mRem/hr	n/a	n/a	320	n/a	n/a	n/a	n/a	n/a	n/a	--
S16R000407	Rad.		15046-84-1	Iodine-129	µCi/g	110	<8.83E-06	6.51E-05	8.27E-05	7.39E-05	23.8	n/a	1.46E-05	14.5	--
S16R000409	Rad.	WR	14762-75-5	Carbon-14	µCi/g	100	<4.07E-06	2.90E-04	3.02E-04	2.96E-04	4.05	101	4.07E-06	3.128	--

**RPP-RPT-59874**  
**Appendix D**

**Table D-3. Pre-Column Solids Composite Data Summary Report. (4 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
S16R000409	Rad.	WR	10028-17-8	Tritium	µCi/g	94.0	<7.83E-05	2.05E-04	7.94E-05	1.42E-04	88.3	115	7.83E-05	47.461	J
S16R000493	Rad.	Z	ALPHA	Gross alpha	µCi/g	107	<9.06E-03	1.71	1.65	1.68	3.72	102	5.07E-03	3.47	--
S16R000493	Rad.	Z	CM-243/244	Curium-243/244	µCi/g	n/a	<1.37E-04	1.03E-03	1.15E-03	1.09E-03	10.7	n/a	1.52E-04	14.21	--
S16R000493	Rad.	Z	14596-10-2	Americium-241	µCi/g	99.8	<2.53E-04	0.415	0.416	0.415	0.361	n/a	1.80E-04	0.67	--
S16R000493	Rad.	Z	15510-73-3	Curium-242	µCi/g	n/a	<9.50E-05	2.07E-04	9.40E-05	1.50E-04	75.0	n/a	1.08E-04	31.92	J
S16R000493	Rad.	Z	13966-00-2	Potassium-40	µCi/g	n/a	<0.338	<0.413	<0.360	n/a	n/a	n/a	0.413	n/a	U
S16R000493	Rad.	Z	10198-40-0	Cobalt-60	µCi/g	96.3	<0.0188	0.0491	0.0433	0.0462	12.6	n/a	0.0256	17.90	--
S16R000493	Rad.	Z	14681-63-1	Niobium-94	µCi/g	n/a	<0.0179	<0.0584	<0.0713	n/a	n/a	n/a	0.0584	n/a	U
S16R000493	Rad.	Z	RU/RH-106	Ru/Rh-106	µCi/g	n/a	<0.395	<0.530	<0.334	n/a	n/a	n/a	0.530	n/a	U
S16R000493	Rad.	Z	14234-35-6	Antimony-125	µCi/g	119	<0.0453	<0.362	<0.485	n/a	n/a	n/a	0.362	n/a	U
S16R000493	Rad.	Z	15832-50-5	Tin-126	µCi/g	n/a	<0.0140	<0.156	<0.152	n/a	n/a	n/a	0.156	n/a	U
S16R000493	Rad.	Z	13967-70-9	Cesium-134	µCi/g	n/a	<0.127	<0.527	<0.556	n/a	n/a	n/a	0.527	n/a	U
S16R000493	Rad.	Z	10045-97-3	Cesium-137	µCi/g	96.7	<0.0165	112	108	110	3.81	n/a	0.0556	0.13	--
S16R000493	Rad.	Z	14683-23-9	Europium-152	µCi/g	n/a	<0.0350	<0.319	<0.359	n/a	n/a	n/a	0.319	n/a	U
S16R000493	Rad.	Z	15585-10-1	Europium-154	µCi/g	n/a	<0.0493	<0.118	<0.0961	n/a	n/a	n/a	0.118	n/a	U
S16R000493	Rad.	Z	14391-16-3	Europium-155	µCi/g	n/a	<0.0394	<0.195	<0.194	n/a	n/a	n/a	0.195	n/a	U
S16R000493	Rad.	Z	14913-50-9	Thallium-208	µCi/g	n/a	<0.0206	<0.0588	<0.100	n/a	n/a	n/a	0.0588	n/a	U
S16R000493	Rad.	Z	14913-49-6	Bismuth-212	µCi/g	n/a	<0.262	<0.784	<0.557	n/a	n/a	n/a	0.784	n/a	U
S16R000493	Rad.	Z	15092-94-1	Lead-212	µCi/g	n/a	<0.0331	<0.109	<0.218	n/a	n/a	n/a	0.109	n/a	U
S16R000493	Rad.	Z	14733-03-0	Bismuth-214	µCi/g	n/a	<0.0452	<0.183	<0.136	n/a	n/a	n/a	0.183	n/a	U
S16R000493	Rad.	Z	15067-28-4	Lead-214	µCi/g	n/a	<0.0277	<0.244	<0.275	n/a	n/a	n/a	0.244	n/a	U
S16R000493	Rad.	Z	13982-63-3	Radium-226	µCi/g	n/a	<0.201	<1.41	<3.48	n/a	n/a	n/a	1.41	n/a	U
S16R000493	Rad.	Z	14331-83-0	Actinium-228	µCi/g	n/a	<0.0623	<0.176	<0.285	n/a	n/a	n/a	0.176	n/a	U
S16R000493	Rad.	Z	14274-82-9	Thorium-228	µCi/g	n/a	<9.16	<31.6	<76.5	n/a	n/a	n/a	31.6	n/a	U
S16R000493	Rad.	Z	15065-10-8	Thorium-234	µCi/g	n/a	<0.763	<1.01	<1.50	n/a	n/a	n/a	1.01	n/a	U
S16R000493	Rad.	Z	13981-37-8	Nickel-63	µCi/g	90.7	6.18E-03	35.9	36.7	36.3	2.20	n/a	1.31E-03	0.205	--
S16R000493	Rad.	Z	14119-32-5	Plutonium-241	µCi/g	98.0	<0.0144	0.957	1.31	1.13	31.1	n/a	9.87E-03	3.042	c
S16R000493	Rad.	Z	PU-239/240	Plutonium-239/240	µCi/g	112	6.97E-04	1.31	1.36	1.33	3.75	n/a	2.77E-04	0.38	--
S16R000493	Rad.	Z	13981-16-3	Plutonium-238	µCi/g	n/a	<2.47E-04	0.0354	0.0386	0.0370	8.38	n/a	2.26E-04	2.31	--
S16R000493	Rad.	Z	13982-10-0	Plutonium-242	µCi/g	n/a	<1.80E-04	4.56E-03	5.79E-03	5.17E-03	23.7	n/a	1.36E-04	6.44	Yc
S16R000493	Rad.	Z	SR-89/90	Strontium-89/90	µCi/g	105	<9.05E-03	97.7	99.4	98.6	1.74	n/a	9.22E-03	0.511	--
S16R000493	Rad.	Z	14133-76-7	Technetium-99	µCi/g	107	<2.88E-03	0.109	0.119	0.114	8.25	n/a	1.53E-03	3.85	--

B = Applied when the preparation blanks are either <EQL or  $\leq 5\%$  of the measured concentration in the sample.

I = Applied when the result was determined via indirect calibration.

J = Applied to results that are considered estimates. Some examples of when a "J" flag may be applied include (but are not limited to):

- Result with concentration  $\geq$ MDL, but  $<$  the EQL
- Radiochemical result with counting uncertainty  $>30\%$
- An "unknown" constituent reported for an organic analysis.

U = Applied to analytes that were analyzed for, but were not detected, or were detected below the MDL

Y = Applied to results that require verbal descriptions or qualifying comments. This flag is used by the chemist, project coordinator, or other technical authority to identify data that is questionable or may be

**RPP-RPT-59874**  
**Appendix D**

**Table D-3. Pre-Column Solids Composite Data Summary Report. (4 pages)**

Sample Group: 20162592

Sample	State	A	CAS #	Analyte	Unit	Std % Rec	Blank	Result	Duplicate	Average	RPD	Spike % Rec	Det Limit	Count Error	Qual Flag
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inaccurate because of interferences, sampling problems, or instrumentation limitations.

a = Applied when the LCS has a percent recovery outside the customer or analytical method specified range. The "a" flag is not applied based on LCSD results.

b = Applied when the MS is outside the acceptance criterion of 75-125%.

c = Applied when the RPD between duplicate samples, LCSDs, or MSDs, is  $\geq 20\%$ . The exception is for ICP analysis on fusion digests, where the ICP RPD acceptance criterion for solids is  $\leq 35\%$ .

e = Applied when the percent difference between the serial dilution and sample is  $\geq 10\%$ .

RPP-RPT-59874

**APPENDIX E.**

**QUALITY ASSURANCE REVIEW DOCUMENT**

(3 pages, including cover sheet)

## RPP-RPT-59874

## Appendix E

## QA COMMENT/CONCERNS FROM REPORT REVIEW

Page 1 of 2

Reviewer: Jeanette Ardiente  
 PC: Tony Scott / Rob Schroeder

Document No.: 20162592  
 Project: HQ Test Bed  
 Review#: 16-09-03

ITEM #	DATA IN QUESTION – COMMENT/CONCERN	* LEVEL PTR/SUG/OBS	N	PC Comments
1	Has the client been notified that the tune failed? Please include e-mail or note in narrative that client was notified.	PTR		The client was not notified by the chemist. Also, the failure is not a "hard" failure. Verbiage in both SW-846 and the laboratory SOP used the word "should" - not "shall". A tailing factor greater than 2 indicates that the GC may need front-end maintenance including trimming the column and changing out the injection port liner and gold seal. The tailing factor was barely above 2.0 (2.08) however this did not affect the ability to properly quantitate and identify target compounds, as indicated by the passing CCV. This statement was added to the narrative.
2	Is there a reason that the ICAL checklist was not included? Please explain in narrative or on Data Review Checklist.	PTR		The ICAL received a first level review but did not receive a second level review therefore the peer review checklist was not completed by the second reviewer. In lieu of the second review, the ICAL curve fit and independent initial verification standard results were included in the package to support the data. This statement was added to the narrative.
3	On the SVOA LIQ/LIQ Extraction Bench Sheet, the analytical batch # and the concentration for LCS/MS/MSD spike mix is missing, please include if applicable.	SUG		The LCS/MS/MSD spike concentration is not listed on the bench sheet - only the book number and volume. The batch number was added to the bench sheet by the chemist.
4	For the sample, the "a" flag should be removed for everything except isobutyl alcohol, 2-ethoxyethanol, pyridine, hexachloroethane and hexachlorobutadiene.	PTR		Corrected by removing incorrect flags. Reprinted batch report for package.
5	The narrative lists, 2,4,5-trichlorophenol, 34&4-methylphenol, isobutyl alcohol and pyridine as not being recovered in the matrix spike, but it appears that 2-methylphenol, hexachlorobutadiene, and hexachloroethane were not recovered either. Please verify.	PTR		Corrected and "b" flagged corresponding compounds in sample result. Reprinted batch report.

C:\Users\h0000743\Documents\110916 Test bed SVOAs Review

## RPP-RPT-59874

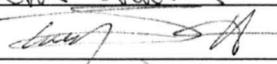
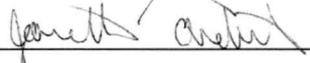
## Appendix E

## QA COMMENT/CONCERN FROM REPORT REVIEW

Page 2 of 2

Reviewer: Jeanette Ardiente  
 PC: Tony Scott / Rob Schroeder

Document No.: 20162592  
 Project: HQ Test Bed  
 Review#: 16-09-03

ITEM #	DATA IN QUESTION – COMMENT/CONCERN	* LEVEL PTR/SUG/OBS	N	PC Comments
6	Did 2,4-Dinitrotoluene, 2-ethoxyethanol, cyclohexanone, and/or nitrobenzene pass the MS and %RPD criteria? If so, then remove the "b" flag, if not, the mention in narrative. Also applies to 2-methylphenol, hexachlorobutadiene and hexachloroethane mentioned above.	PTR		Corrected and removed "b" flag from those compounds in the sample. Reprinted batch report.
7	I believe that the surrogate standards are not typically included in the DSR and should be turned off. Please verify.	PTR		Not familiar with removing surrogates from the DSR. Not sure how the DSR is generated.
*PTR = Prior to Data Release				*OBS = Observation
*SUG = QA Suggestion or Information				*N = Add to Narrative
NOTES:				
QA/QC Reviewer:				Date comments provided: 11-9-16
Project Coordinator:				Date comments received: 11-9-2016
QA/QC Reviewer:				Date comments resolved: 11-9-16

RPP-RPT-59874

**APPENDIX F.**

**WRPS 222-S ACTIVITY TIMELINE ASSOCIATED WITH THE TEST BED PROJECT**

(2 pages, including cover sheet)

## RPP-RPT-59874

## Appendix F

**222-S Laboratory Timeline Associated with the Test Bed Project.**

Date	Event
8-04-2015	222-S Laboratory was contacted by One System (Chief Technology Office (CTO)) – Sample archive and possible treatability study mentioned.
10-27-2015	PNNL contacted 222-S Laboratory – Requested information from on ~4 kg of tank waste to. WRPS proved rough cost estimate in 12/2015 for compositing filtering and Cs removal for 23
3-17-2016	PNNL (contracted by CTO) contacted 222-S Laboratory – Discussed interest in 100 gallons of tank waste.
4-28-2016	222-S Laboratory enquired about achieve sample release from Tank Waste Characterization.
May 2016	One System discussions with 222-S Laboratory – Discussed sample identification and cost estimate for a 5 gallon tank waste composite.
June 2016	222-S Laboratory pursued looking into current stock of sRF resin.
6-15-2016	Draft schedule still showed PNNL performing the work to filter and remove cesium from a tank waste composite.
6-28-2016	Proposal sent to DOE-ORP with the 222-S Laboratory slated to filter and remove cesium from a tank waste composite.
7-18-2016	One System submitted their draft proposal to DOE-ORP.
7-28-2016	Formal request for specified achieve samples made.
8-02-2016	222-S Laboratory received a draft copy of the WIR from DOE-EM, revealing the intended end product.
8-04-2016	Tank Waste Characterization released the first group of samples for use.
8-05-2016	222-S Laboratory send out the draft treatability study designation form.
8-08-2016	222-S Laboratory funding and work authorization was approved.
8-08-2016 to 9-07-2016	222-S Laboratory wrote the test plan, completed treatability study documentation, obtained release of samples, procured equipment & resin, performed cold testing, loaded equipment into the hot cell and setting up the test stand.
9-07-2016	222-S Laboratory built the archive composite.
9-13-2016	222-S Laboratory began filtering the composite.
9-15-2016	222-S Laboratory loaded out pre-column composite analytical samples.
9-19-2016	222-S Laboratory began continuous operation to remove cesium from the composite.
9-22-2016	222-S Laboratory successfully completed the cesium removal process.
9-26-2016	WHL provided GEA data to indicate the successful removal of Cs-137. The cesium-depleted fractions were recombined, blended and subsampled for analysis.
9-26-2016	Nickel tracer requested to be added to all radiochemistry subsamples.
9-27-2016	222-S Laboratory loaded out the post-column subsample for WHL analysis.
10-05-2016	222-S Laboratory circulated the composite and then dispensed it into 1 L product bottles.
10-06-2016	222-S Laboratory loaded the 1 L product bottles out of the hot cell and packaged into a 30 gal. drum.
10-13-2016	222-S Laboratory identified natrophosphate in the post-filtration, pre-column precipitated solids.
10-19-2016	222-S Laboratory obtained requests for analysis of 6 additional SVOA and VOA analytes.
11-01-2016	Rework was requested on approximately 13 organics due to analysis not coming under reporting limits. 222-S Laboratory retrieved bottle S16R000331 from drum and removed an additional 664 g of cesium-depleted product from the bottle.
11-02-2016	WRPS investigated the regulatory limits necessary for RCRA §268.40 nonwastewater concentration and RCRA §268.48 nonwastewater standard. It was realized that the regulation limits previously provided should have been in units of $\mu\text{g}/\text{L}$ TCLP instead of $\mu\text{g}/\text{kg}$ . Therefore, the additional organic analysis (using 664 g of cesium-depleted product) was unnecessary.

# INFORMATION CLEARANCE REVIEW AND RELEASE APPROVAL

## Part I: Background Information

Title: Test Report for Cesium and Solids Removal from an 11.5L Composite of Archived Hanford Double Shell Tank Supernate for Off-Site Disposal

Publish to OSTI?  Yes  No

### Information Category:

Abstract  Journal Article  Summary  
 Internet  Visual Aid  Software  
 Full Paper  Report  Other

Trademark/Copyright "Right to Use" Information or Permission Documentation

Yes NA

Document Number: RPP-RPT-59874 Revision 0

Date: April 2017

Author: Doll, Stephanie R

## Part II: External/Public Presentation Information

Conference Name:

Sponsoring Organization(s): WRPS

Date of Conference:

Conference Location:

Will Material be Handed Out?  Yes  No Will Information be Published?  Yes  No (If Yes, attach copy of Conference format instructions/guidance.)

## Part III: WRPS Document Originator Checklist

Description	Yes	N/A	Print/Sign/Date
Information Product meets requirements in TFC-BSM-AD-C-01?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Document Release Criteria in TFC-ENG-DESIGN-C-25 completed? (Attach checklist)	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
If product contains pictures, safety review completed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

## Part IV: WRPS Internal Review

Function	Organization	Date	Print Name/Signature/Date
Subject Matter Expert	WRPS	05/15/2017	Doll, Stephanie R IDMS Data File att.
Responsible Manager	WRPS	04/03/2017	Cooke, Gary A IDMS Data File att.
Other:			

## Part V: IRM Clearance Services Review

Description	Yes	No	Print Name/Signature
Document Contains Classified Information?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	If Answer is "Yes," ADC Approval Required  Print Name/Signature/Date
Document Contains Information Restricted by DOE Operational Security Guidelines?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Reviewer Signature:  Print Name/Signature/Date
Document is Subject to Release Restrictions? <i>If the answer is "Yes," please mark category at right and describe limitation or responsible organization below:</i>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Document contains: <input type="checkbox"/> Applied Technology <input type="checkbox"/> Protected CRADA <input type="checkbox"/> Personal/Private <input type="checkbox"/> Export Controlled <input type="checkbox"/> Proprietary <input type="checkbox"/> Procurement – Sensitive <input type="checkbox"/> Patentable Info. <input type="checkbox"/> OOU <input type="checkbox"/> Predecisional Info. <input type="checkbox"/> UCNI <input type="checkbox"/> Restricted by Operational Security Guidelines <input type="checkbox"/> Other (Specify) _____
Additional Comments from Information Clearance Specialist Review?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Information Clearance Specialist Approval <div style="background-color: #90EE90; border: 1px solid black; padding: 2px; text-align: center;"> <b>APPROVED</b>  <i>By Janis Aardal at 2:14 pm, May 16, 2017</i> </div> Print Name/Signature/Date

When IRM Clearance Review is Complete – Return to WRPS Originator for Final Signature Routing (Part VI)

Page 1 of ~~2~~ 3

A-6003-508 (REV 4)

## INFORMATION CLEARANCE REVIEW AND RELEASE APPROVAL

### Part VI: Final Review and Approvals

Description	Approved for Release		Print Name/Signature
	Yes	N/A	
WRPS External Affairs	<input checked="" type="checkbox"/>	<input type="checkbox"/>	IDMS Data File att. Holloway, Jerry N
WRPS Office of Chief Counsel	<input checked="" type="checkbox"/>	<input type="checkbox"/>	IDMS Data File att. Roden, Mari L
DOE – ORP Public Affairs/Communications	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Marshall, Richard A /Call, Paula K
Other: ORP SME	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Burnett, Kaylin W /Smith, Sahid C
Other: DOE OCC	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Stubblebine, Scott D

Comments Required for WRPS-Indicate Purpose of Document:

This test report describes the 222-S Laboratory Washington River Protection Solutions LLC and WAI Hanford Laboratory results of the low activity waste treatment technology project, Radioactive Test Bed Initiative.

**APPROVED**

*By Janis Aardal at 2:15 pm, May 16, 2017*

**Approved for Public Release;  
Further Dissemination Unlimited**

### Information Release Station

Was/Is Information Product Approved for Release?  Yes  No

If Yes, what is the Level of Releaser?  Public/Unrestricted  Other (Specify) \_\_\_\_\_

Date Information Product Stamped/Marked for Release: 05/16/2017

Was/Is Information Product Transferred to OSTI?  Yes  No

**Forward Copies of Completed Form to WRPS Originator**

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