



U.S. DEPARTMENT OF  
**ENERGY**

PNNL-19425

Prepared for the U.S. Department of Energy  
under Contract DE-AC05-76RL01830

# **Hanford Site Tank 241-C-108 Residual Waste Contaminant Release Models and Supporting Data**

KJ Cantrell  
KM Krupka  
KN Geiszler

BW Arey  
HT Schaef

June 2010



**Pacific Northwest**  
NATIONAL LABORATORY

*Proudly Operated by **Battelle** Since 1965*

## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights.** Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY

*operated by*

BATTELLE

*for the*

UNITED STATES DEPARTMENT OF ENERGY

*under Contract DE-AC05-76RL01830*

Printed in the United States of America

Available to DOE and DOE contractors from the

Office of Scientific and Technical Information,

P.O. Box 62, Oak Ridge, TN 37831-0062;

ph: (865) 576-8401

fax: (865) 576-5728

email: [reports@adonis.osti.gov](mailto:reports@adonis.osti.gov)

Available to the public from the National Technical Information Service,

U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161

ph: (800) 553-6847

fax: (703) 605-6900

email: [orders@ntis.fedworld.gov](mailto:orders@ntis.fedworld.gov)

online ordering: <http://www.ntis.gov/ordering.htm>



This document was printed on recycled paper.



# **Hanford Site Tank 241-C-108 Residual Waste Contaminant Release Models and Supporting Data**

KJ Cantrell  
KM Krupka  
KN Geiszler

BWArey  
HT Schaef

June 2010

Prepared for  
Washington River Protection *Solutions*, LLC and  
the U.S. Department of Energy  
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory  
Richland, Washington 99352

## Summary

The U.S. Department of Energy (DOE) Office of River Protection prime contractor, Washington River Protection *Solutions*, LLC (WRPS), is responsible for the storage, retrieval, and disposal of Hanford Site tank waste. As part of this effort, WRPS contracted with Pacific Northwest National Laboratory to conduct testing for selected key contaminants present in residual waste remaining in the single-shell tank 241-C-108 (C-108). Release model data will be used to support tank closure performance assessments that will be performed by WRPS. This report presents the results from laboratory characterization, testing, and analysis of a tank C-108 post modified sluicing residual waste composite sample (designated 20578). The tank C-108 residual waste sample was taken following an assessment that determined through material balance data the modified retrieval sluicing system deployed at the tank could no longer efficiently retrieve waste (met the limit of the technology). These studies were completed to characterize concentration and form of contaminant of interest in the residual waste; assess the leachability of contaminants from the solids; and develop release models for contaminants of interest. It is expected that additional retrieval processing will take place. As a result, the sample analyzed here is not expected to represent final retrieval sample. Total elemental analysis using the U.S. Environmental Protection Agency (EPA) acid digestion method, indicates that tank C-108 partial retrieval residual waste (sample 20578) is composed of the following major elements Na at 15.5%, Al at 12.3%, P at 4.0%, Fe at 1.6%, Ni at 0.22%, U at 0.18%, and Mn at 0.13%. Concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  were determined to be 0.636  $\mu\text{g/g}$  waste (0.0108  $\mu\text{Ci/g}$ ) and 1870  $\mu\text{g/g}$  waste ( $6.27 \times 10^{-4}$   $\mu\text{Ci/g}$ ). Average concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  were determined to be  $1.24 \times 10^{-4}$   $\mu\text{Ci/g}$  ( $^{237}\text{Np}$ ),  $2.90 \times 10^{-2}$   $\mu\text{Ci/g}$  ( $^{239}\text{Pu}$ ), and not detected ( $^{241}\text{Am}$ ). The concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  were found at significant concentrations in tank C-108 residual waste, with averages of  $1.69 \times 10^4$   $\mu\text{Ci/g}$  ( $^{137}\text{Cs}$ ) and  $1.22 \times 10^5$   $\mu\text{Ci/g}$  ( $^{90}\text{Sr}$ ). The results determined from the fusion dissolution procedure were generally very similar to those determined with the EPA acid digestion method.

Solid phase characterization by x-ray diffraction (XRD) indicated the presence of the following phases in the as-received tank C-108 composite sample: gibbsite [ $\text{Al}(\text{OH})_3$ ], natrophosphate [ $\text{Na}_7(\text{PO}_4)_2\text{F} \cdot 19\text{H}_2\text{O}$ ], sodium fluoride phosphate hydrate [ $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$ ], sodium phosphate ( $\text{Na}_3\text{PO}_4$ ), nickel oxide hydroxide ( $\text{NiOOH}$ ), and nitratine ( $\text{NaNO}_3$ ). Solid phase characterization by scanning electron microscopy/energy dispersive spectrometry (SEM/EDS) analyses indicated the majority of the particles in the as-received tank C-108 composite sample are composed of Al-Na-O-P-F, Al-Na-O-P, Na-O-P-F, Na-O-P, Al-Na-O, and Na-O, all in variable proportions. Note that all phases could also potentially contain H and C, which cannot be quantified by EDS. Combining the XRD with the SEM/EDS results, the following conclusions can be made regarding the presence of solid phases in the tank C-108 residual waste composite sample:

- Gibbsite was the major phase present and appears to be coated and mixed with variable amounts of phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate
- Fe-oxides occurred as a minor phase. Its particles were coated and mixed with variable amounts of other solid phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate
- Minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu, were associated with the Fe-oxides

- Most likely the Ni occurred as a separate phase (NiOOH) coating the iron oxide
- Small amounts of a U–Na–O phase(s), most likely čejkaite and/or sodium uranate or similar phase were also present
- Sr was occasionally associated with a Na–O–P phase, possibly as NaSrPO<sub>4</sub> or another similar phase.

It is also noteworthy that particles composed of U–Na–O had morphologies that were very similar to particles that were identified as čejkaite in a previous study conducted with samples of unleached tank C-203 preretrieval waste (Deutsch et al. 2007).<sup>1</sup>

Analysis of the tank C-108 solids that remained after leaching with double-deionized (DDI) water and calcite (CaCO<sub>3</sub>) saturated DDI water indicated that the majority of the more soluble phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate were largely dissolved away, leaving relatively clean gibbsite and Fe oxides particles with only minor amounts of surface coatings. The elemental compositions of many of the particles analyzed are close to the composition of pure gibbsite. As was the case for the unleached samples, the Fe-oxides often hosted minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu. Particles containing Sr with an elemental composition of SrNaPO<sub>4</sub> were also occasionally identified in the DDI water and CaCO<sub>3</sub> saturated DDI water leached samples.

The solids that were leached with Ca(OH)<sub>2</sub> saturated DDI water were quite different than the DDI water and CaCO<sub>3</sub> saturated DDI leached samples. The surfaces of these particles were coated with precipitated materials and desiccation cracks were evident. XRD indicated only the presence of gibbsite. The SEM/EDS results suggested the following phases are likely present:

- Gibbsite as the dominant phase with coatings of one or more phases with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) and Ca(OH)<sub>2</sub>
- Lesser amounts of Fe-oxide(s) with coatings of one or more phase(s) with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) and Ca(OH)<sub>2</sub>
- Ni and traces of other metals such as Pb, Zn, and Cu which were often associated with the Fe-oxide(s).

Thermodynamic modeling suggested that the DDI and CaCO<sub>3</sub> saturated leachate solutions were close to saturation with respect to Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> (am), while the Ca(OH)<sub>2</sub> saturated leachate solutions were near equilibrium with respect to Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> (c). These results suggest that any čejkaite that was present in the as-received sample from tank C-108 residual waste was converted to these less soluble phases as a result of contact with the relatively dilute leachate solutions. The leachate solutions were also determined to be significantly undersaturated with respect to gibbsite [Al(OH)<sub>3</sub>], and other Al oxyhydroxides. This is likely due to slow dissolution kinetics of these phases. Additional useful saturation index results were limited due to analytical results that were below the quantification limit.

---

<sup>1</sup> Deutsch WJ, KM Krupka, MJ Lindberg, KJ Cantrell, CF Brown, and HT Schaefer. 2007. *Hanford Tanks 241-C-203 and 241-C-204: Residual Waste Contaminant Release Model and Supporting Data*. PNNL-14903, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

The leachability of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  was relatively high in the tank C-108 residual waste. For the 1-month single-contact DDI leachants, the average leachable  $^{99}\text{Tc}$  concentration was  $0.506\text{ }\mu\text{g/g}$  ( $0.00860\text{ }\mu\text{Ci/g}$ ) and the average leachable  $^{238}\text{U}$  concentration was  $825\text{ }\mu\text{g/g}$  ( $2.77 \times 10^{-4}\text{ }\mu\text{Ci/g}$ ). These values equate to an average of 80% ( $^{99}\text{Tc}$ ) and 44% ( $^{238}\text{U}$ ) compared to the totals determined by EPA acid digestion. For the 1-month single-contact  $\text{Ca}(\text{OH})_2$  leachants, the average leachable  $^{99}\text{Tc}$  concentration was  $0.522\text{ }\mu\text{g/g}$  ( $0.00887\text{ }\mu\text{Ci/g}$ ) and the average leachable  $^{238}\text{U}$  concentration was  $52.1\text{ }\mu\text{g/g}$  ( $1.75 \times 10^{-5}\text{ }\mu\text{Ci/g}$ ). These values are an average of 82% ( $^{99}\text{Tc}$ ) and 2.8% ( $^{238}\text{U}$ ) of the totals. Compared to the leachable concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  determined in the 1-month DDI water leachates, the percent leachable  $^{99}\text{Tc}$  is about the same, whereas the percent leachable  $^{238}\text{U}$  is dramatically lower. This suggests the tank C-108 residual waste in contact with  $\text{Ca}(\text{OH})_2$  leachant may result in the formation of a Ca-U phase with a lower solubility such as becquerelite [ $\text{Ca}(\text{UO}_2)_6\text{O}_4(\text{OH})_6(\text{H}_2\text{O})_8$ ]. For the 1-month single-contact  $\text{CaCO}_3$  leachants, the average leachable  $^{99}\text{Tc}$  concentration was  $0.627\text{ }\mu\text{g/g}$  ( $0.0107\text{ }\mu\text{Ci/g}$ ) and the average leachable  $^{238}\text{U}$  concentration was  $648\text{ }\mu\text{g/g}$  ( $2.18 \times 10^{-4}\text{ }\mu\text{Ci/g}$ ). These values equate to an average of 99% ( $^{99}\text{Tc}$ ) and 35% ( $^{238}\text{U}$ ) compared to the totals determined by EPA acid digestion. These values are similar to those of the DDI leachants.

## Acknowledgments

The authors wish to acknowledge M. Connelly and S. J. Eberlein at Washington River Protection *Solutions*, LLC (Richland, Washington) for providing project funding and technical guidance. We greatly appreciate the technical reviews provided by M. Connelly, M. P. Bergeron, L. Fort, J. G. Field, and D. M. Nguyen of Washington River Protection *Solutions*, LLC, and N. P. Qafoku (PNNL). The authors would also like to thank S. R. Baum, E. T. Clayton, and I. V. Kutnyakov (all from PNNL) for completing the chemical analyses of the solution samples from our studies. We are particularly grateful to H. E. Matthews (PNNL) for completing the editorial review and K. R. Neiderhiser (PNNL) for final word processing of this technical report.

## Acronyms and Abbreviations

AMU	atomic mass unit
ASTM	American Society for Testing and Materials
BSE	backscattered electron
CCV	continuing calibration verification
CFR	<i>Code of Federal Regulations</i>
DDI	double-deionized (water)
DOE	U.S. Department of Energy
Dup (or dup)	duplicate
EDS	energy dispersive spectrometry
EPA	U.S. Environmental Protection Agency
EQL	estimated quantification limit
GEA	gamma energy analysis
GWB	Geochemist's Workbench <sup>®1</sup>
HASQARD	Hanford Analytical Services Quality Assurance Requirements Document
HDW	Hanford Defined Waste
IC	ion chromatography (chromatograph)
ICDD	International Center for Diffraction Data
ICP-MS	inductively coupled plasma-mass spectrometry (spectrometer)
ICP-OES	inductively coupled plasma-optical emission spectroscopy (same as ICP-AES)
JCPDS	Joint Committee on Powder Diffraction Standards
NIST	National Institute of Standards and Technology
ND	not detected
LSC	liquid scintillation counting
PDF <sup>™</sup>	powder diffraction file
pH	measure of the acidity of a solution, where pH is the negative of the logarithm of the activity of H <sup>+</sup> in solution
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
C-108	single-shell tank 241-C-108 (Hanford Site)
SE	secondary electron
SEM	scanning electron microscopy (or microscope)
SI	saturation index
SRM	standard reference material
SST	single-shell tank

---

<sup>1</sup> The Geochemist's Workbench is a registered trademark of the University of Illinois.

TBP	tributyle phosphate process waste
TIC	total inorganic
TOC	total organic
TC	Total C
XRD	X-ray powder diffractometry analysis (commonly called X-ray diffraction)
WRPS	Washington River Protection <i>Solutions</i> , Inc.

## Units of Measure

$\theta$	angle of incidence (Bragg angle)
$^{\circ}\text{C}$	temperature in degrees Celsius [ $T(^{\circ}\text{C}) = T(\text{K}) - 273.15$ ]
cm	centimeter
ft	foot
g	gram
gallon	gallon
in.	inch
L	liter
$\mu$	micro (prefix, $10^{-6}$ )
$\mu\text{Ci}$	microcurie
$\mu\text{g}$	microgram
$\mu\text{m}$	micrometer
M	molarity, mol/L
mL	milliliter
mM	molarity, millimol/L
rpm	revolution per minute
$I/I_0$	relative intensity of an x-ray powder diffractometry analysis peak to the most intense peak
$\lambda$	wavelength
wt%	weight percent



# Contents

Summary .....	iii
Acknowledgments.....	vii
Acronyms and Abbreviations .....	ix
Units of Measure.....	xi
1.0 Introduction .....	1.1
1.1 Work Scope.....	1.1
1.2 C-100 Series Tank Description .....	1.2
2.0 Materials and Laboratory Test Methods.....	2.1
2.1 Tank C-108 Residual Waste Samples .....	2.1
2.2 Tier 1 Tests.....	2.2
2.2.1 Residual Waste Composition by Fusion Analysis and Acid Digestion.....	2.2
2.2.2 Moisture Content.....	2.3
2.2.3 Carbon Analysis .....	2.4
2.2.4 Leach Tests.....	2.4
2.2.5 pH.....	2.6
2.2.6 Alkalinity.....	2.6
2.2.7 Anion Analysis.....	2.6
2.2.8 Cations and Trace Metals.....	2.6
2.2.9 <sup>237</sup> Np and <sup>239</sup> Pu Analysis .....	2.7
2.2.10 Radiochemical Analysis.....	2.7
2.3 X-Ray Powder Diffractometry .....	2.8
2.4 Scanning Electron Microscopy/Energy Dispersive Spectrometry Analysis .....	2.11
2.5 Geochemical Modeling .....	2.12
3.0 Laboratory Results.....	3.1
3.1 Residual Waste Composition .....	3.1
3.1.1 EPA Acid Digestion Results .....	3.2
3.1.2 Fusion Results .....	3.5
3.1.3 Comparison with Results Reported by the 222-S Laboratory.....	3.8
3.1.4 Carbon Analysis Results .....	3.9
3.2 Double-Deionized Water-Leach Tests .....	3.9
3.2.1 1-Month Single-Contact Double-Deionized Water-Leach Tests .....	3.9
3.2.2 Periodic Replenishment Double-Deionized Water-Leach Tests .....	3.14
3.3 Ca(OH) <sub>2</sub> Solution Leach Tests .....	3.19
3.3.1 1-Month Single-Contact Ca(OH) <sub>2</sub> Solution Leach Tests .....	3.19
3.3.2 Periodic Replenishment Ca(OH) <sub>2</sub> Solution Leach Tests.....	3.23

3.4	CaCO <sub>3</sub> Solution Leach Tests .....	3.28
3.4.1	1-Month Single-Contact CaCO <sub>3</sub> Solution Leach Tests .....	3.28
3.4.2	Periodic Replenishment CaCO <sub>3</sub> Solution Leach Tests .....	3.33
3.5	X-Ray Diffraction Results .....	3.37
3.6	Scanning Electron Microscopy/Energy Dispersive Spectrometry Results .....	3.40
3.7	Geochemical Modeling .....	3.45
4.0	Contaminant Release Models .....	4.1
4.1	Uranium, Chromium, and <sup>99</sup> Tc Release Models .....	4.1
5.0	Discussion .....	5.1
6.0	References .....	6.1
	Appendix A – Leach Test Results – Solution Concentration .....	A.1
	Appendix B – X-Ray Diffraction Patterns for Unleached and Leached Samples of Residual Waste from Tank C-108 .....	B.1
	Appendix C – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Composite Subsample from Unleached Residual Waste from Tank C-108 .....	C.1
	Appendix D – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Green Nodule Subsample from Unleached Residual Waste from Tank C-108 .....	D.1
	Appendix E – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Black Nodule Subsample from Unleached Residual Waste from Tank C-108 .....	E.1
	Appendix F – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Crystal Nodule Subsample from Unleached Residual Waste from Tank C-108 .....	F.1
	Appendix G – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Deionized Water-Leached Residual Waste from Tank C-108 .....	G.1
	Appendix H – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Ca(OH) <sub>2</sub> Leached Residual Waste from Tank C-108 .....	H.1
	Appendix I – Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for CaCO <sub>3</sub> Leached Residual Waste from Tank C-108 .....	I.1

## Figures

1.1	Hanford Site 241-C Tank Farm .....	1.3
1.2	Generalized Profile View of a 100-Series Tank Such as Tank C-108.....	1.4
2.1	Tank C-108 Residual Waste .....	2.1
2.2	Exploded Schematic View of the XRD Sample Holder .....	2.10
2.3	XRD Pattern for Collodion Film Measured in the Absence of Any Residual Waste Material .....	2.11
3.1	As-Measured and Background Subtracted XRD Patterns for Composite Subsample from the As-Received Tank C-108 Residual Waste .....	3.39
3.2	Backscattered Electron SEM Image of the As-Received Composite Tank C-108 Residual Waste Sample.....	3.43
3.3	Backscattered Electron SEM Image of DDI Water-Leached Sample, Tank C-108 Residual Waste.....	3.44
3.4	Backscattered Electron SEM Image of Ca(OH) <sub>2</sub> Leached Sample, Tank C-108 Residual Waste.....	3.45
4.1	Source Release Model Development for Long-Term Performance Assessments .....	4.1

## Tables

2.1	Tank C-108 Sample Provided to PNNL by 222-S Laboratory .....	2.1
2.2	Digestion Factors for Residual Waste Solids Used for the KOH-KNO <sub>3</sub> Fusion Treatment and EPA Acid Digestion Treatment .....	2.3
2.3	pH, Ca, and Alkalinity, for Initial Leachant Solutions .....	2.5
3.1	Moisture Contents of Tank C-108 Residual Waste Samples .....	3.1
3.2	Residual Waste Composition Determined by EPA Acid Digestion and ICP-OES Analysis.....	3.3
3.3	Residual Waste Composition Determined by EPA Acid Digestion and ICP-MS Analysis.....	3.4
3.4	Concentrations of <sup>99</sup> Tc and <sup>238</sup> U Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion and ICP-MS Analysis.....	3.5
3.5	Concentrations of <sup>237</sup> Np, <sup>239</sup> Pu, and <sup>241</sup> Am Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion and ICP-MS Analysis .....	3.5
3.6	Concentrations of <sup>137</sup> Cs, and <sup>90</sup> Sr Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion.....	3.5
3.7	Residual Waste Composition Determined by Fusion Dissolution and ICP-OES Analysis .....	3.6
3.8	Residual Waste Composition Determined by Fusion Dissolution and ICP-MS Analysis .....	3.6
3.9	Concentrations of <sup>99</sup> Tc and <sup>238</sup> U Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution and ICP-MS Analysis .....	3.7

3.10	Concentrations of $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution and ICP-MS Analysis.....	3.7
3.11	Concentrations of $^{137}\text{Cs}$ , and $^{90}\text{Sr}$ Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution .....	3.8
3.12	Comparison of PNNL Results with Those Determined by the 222-S Laboratory .....	3.8
3.13	Carbon Analysis Results of Tank C-108 Residual Waste Samples .....	3.9
3.14	Residual Waste to Double-Deionized Water Ratios Used in Single-Contact Leach Tests.....	3.10
3.15	The pH and Alkalinity Values for Single-Contact Double-Deionized Water Leachates.....	3.10
3.16	Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples .....	3.10
3.17	Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples .....	3.11
3.18	Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples.....	3.11
3.19	Leachable Metal Concentrations in Waste Determined by ICP-OES in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples .....	3.12
3.20	Leachable Metal Concentrations in Waste Determined by ICP-MS in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples .....	3.13
3.21	Leachable Anion Concentrations Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste .....	3.14
3.22	Residual Waste to Double-Deionized Water Ratios Used in the Periodic Replenishment Leach Tests .....	3.14
3.23	Average pH and Alkalinity Values for Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste .....	3.15
3.24	Average Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.15
3.25	Average Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.16
3.26	Average Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.16
3.27	Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.17
3.28	Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.17
3.29	Average Leachable Anion Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste.....	3.18
3.30	Residual Waste to $\text{Ca}(\text{OH})_2$ Solution Ratios Used in Single-Contact Leach Tests .....	3.19
3.31	The pH and Alkalinity Values for Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates .....	3.19
3.32	Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations Determined from Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.20
3.33	Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations Determined from Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.20

3.34	Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations Determined from Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.21
3.35	Leachable Metals Concentrations Determined by ICP-OES in Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste Samples .....	3.21
3.36	Leachable Metals Concentrations Determined by ICP-MS in Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste Samples .....	3.22
3.37	Leachable Anion Concentrations Determined from Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.23
3.38	Residual Waste to $\text{Ca}(\text{OH})_2$ Solution Ratios Used in the Periodic Replenishment Leach Tests .....	3.23
3.39	Average pH and Alkalinity Values for Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates .....	3.24
3.40	Average Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations Determined from Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.24
3.41	Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations Determined from Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.25
3.42	Average Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations Determined from Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.25
3.43	Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.26
3.44	Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.27
3.45	Average Leachable Anion Concentrations Determined from Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachates from Tank C-108 Residual Waste .....	3.28
3.46	Residual Waste to $\text{CaCO}_3$ Solution Ratios Used in Single-Contact Leach Tests .....	3.28
3.47	The pH and Alkalinity Values for Single-Contact $\text{CaCO}_3$ Solution Leachates .....	3.29
3.48	Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations Determined from Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.29
3.49	Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations Determined from Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.30
3.50	Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations Determined from Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.30
3.51	Leachable Metals Concentrations Determined by ICP-OES in Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.31
3.52	Leachable Metals Concentrations Determined by ICP-MS in Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.31
3.53	Leachable Anion Concentrations Determined from Single-Contact $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.32
3.54	Residual Waste to $\text{CaCO}_3$ Solution Ratios Used in the Periodic Replenishment Leach Tests .....	3.33
3.55	Average pH and Alkalinity Values for Periodic Replenishment $\text{CaCO}_3$ Solution Leachates .....	3.33
3.56	Average Leachable $^{99}\text{Tc}$ and $^{238}\text{U}$ Concentrations Determined from Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.34

3.57	Leachable $^{137}\text{Cs}$ and $^{90}\text{Sr}$ Concentrations Determined from Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.34
3.58	Average Leachable $^{237}\text{Np}$ , $^{239}\text{Pu}$ , and $^{241}\text{Am}$ Concentrations Determined from Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.35
3.59	Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.35
3.60	Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.36
3.61	Average Leachable Anion Concentrations Determined from Periodic Replenishment $\text{CaCO}_3$ Solution Leachates from Tank C-108 Residual Waste .....	3.37
3.62	Summary of Phases that Match the XRD Patterns for the As-Received, Green Nodule, Black Nodule, Crystal Nodule, DDI Water-Leached, $\text{Ca}(\text{OH})_2$ -Leached, and $\text{CaCO}_3$ -Leached Samples of Tank C-108 Residual Waste .....	3.40
3.63	Summary of Phases Indicated by SEM/EDS and XRD in Tank C-108 Residual Waste Samples .....	3.41
3.64	Calculated Saturation Indices for Relevant Al-, Na-, Ca-, $\text{PO}_4^-$ , and U-Containing Minerals Based on Compositions of 1-Month Single-contact DDI Leachates of Tank C-108 Residual Waste Samples .....	3.46
3.65	Calculated Saturation Indices for Relevant Al-, Na-, Ca-, $\text{PO}_4^-$ , and U-Containing Minerals Based on Compositions of 1-Month Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachates of Tank C-108 Residual Waste Samples .....	3.47
3.66	Calculated Saturation Indices for Relevant Al-, Na-, Ca-, $\text{PO}_4^-$ , and U-Containing Minerals Based on Compositions of 1-Month Single-contact $\text{CaCO}_3$ Solution Leachates of C-108 Residual Waste Samples .....	3.48
4.1	Residual Waste and Maximum Contaminant Release Concentrations for the Tank C-108 Release Model .....	4.2

## 1.0 Introduction

Washington River Protection *Solutions*, LLC is the U.S. Department of Energy (DOE) Office of River Protection's prime contractor responsible for the storage, retrieval, and disposal of Hanford Site tank waste. As part of this effort, Washington River Protection *Solutions*, LLC contracted with Pacific Northwest National Laboratory (PNNL) to conduct testing for selected key contaminants present in residual waste remaining in the single-shell tank (SST) 241-C-108 (C-108) after waste retrieval. Release model data will be used to support tank closure performance assessments that will be performed by Washington River Protection *Solutions*, LLC.

Previous work to characterize and develop release models has been completed for final residual waste collected from five SSTs: C-106 (Deutsch et al. 2006, 2007a); C-202, C-203 (Deutsch et al. 2007c); C-103 (Cantrell et al. 2008a); and S-112 (Cantrell et al. 2008b). With the exception of one salt cake tank (S-112), all previous work describing characterization results and contaminant release models for final tank residual waste in SSTs were based on samples from sludge tanks. Tank C-108 is also a sludge tank.

This report presents the results of laboratory characterization, testing, analysis, and release model development for a composite sample (designated 20578) of residual waste collected from SST C-108 during the waste retrieval process after modified sluicing. Tank C-108 modified retrieval sluicing system operations were suspended because that retrieval system could no longer efficiently retrieve waste (met the limit of the technology). It is expected that additional retrieval processing will occur. As a result, the sample analyzed for these studies is not expected to represent final retrieval sample. These studies were completed to characterize the residual waste and assess the leachability of contaminants from the solids.

PNNL uses a tiered approach to the characterization and testing of the tank residual waste. Initial (Tier 1) laboratory tests include determination of the inventory of contaminants and bulk constituents of the residual waste, quantification of the concentrations of water-leachable contaminants and other waste components, and solids characterization with x-ray diffraction (XRD) and scanning electron microscopy/energy dispersive spectrometry (SEM/EDS) analyses. Tier 2 analyses are designed based upon the results of the Tier 1 tests, priorities, and funding resources. Examples of Tier 2 analyses include solubility tests, selective extractions, and application of synchrotron-based x-ray techniques and Mössbauer spectroscopy. Tier 1 results are reported here.

### 1.1 Work Scope

Initial (Tier 1) laboratory tests were conducted to characterize the residual waste and identify water-leachable constituents. The Tier 1 tests consisted primarily of acid digestions to measure elemental concentrations in the solid, and water leaching of contaminants from the residual waste to evaluate their mobility in infiltrating water. Water leaching was conducted with double deionized water (DDI), as well as  $\text{Ca}(\text{OH})_2$ -saturated DDI water, and  $\text{CaCO}_3$ -saturated DDI water solutions. The  $\text{Ca}(\text{OH})_2$  and  $\text{CaCO}_3$  saturated solutions were used to mimic the initial and final status of a tank chemical system in which the void space above the residual waste is filled with cementitious grout, which is a possible tank fill material at closure. XRD analysis of the as-received sample and water-leached solids was conducted to identify crystalline mineral phases present in the residual waste. SEM/EDS analyses were conducted to provide information on the morphologies and compositions of solid particles of the as-received and water-leached residual waste samples.

Laboratory results of Tier 1 testing on tank C-108 residual waste were used to develop source term models that describe the release of contaminants as infiltrating water contacts the solids in the future. These models simulate the geochemical system in the tank residual waste and take into account interactions between the solution phase and the contaminant-containing solids. The geochemical models will be used in future tank performance assessments.

## 1.2 C-100 Series Tank Description

The C Tank Farm was constructed from 1943 to 1944 in the 200 East Area and contains twelve 100-series and four 200-series, dish-bottom design SSTs (Brevick et al. 1997); see Figure 1.1. The 100-series tanks have a design capacity of 2,006,000 L (530,000 gal) and a diameter of 22.9 m (75 ft). Figure 1.2 presents a generalized profile view of a 100-series tank such as C-108 (RPP 2007).

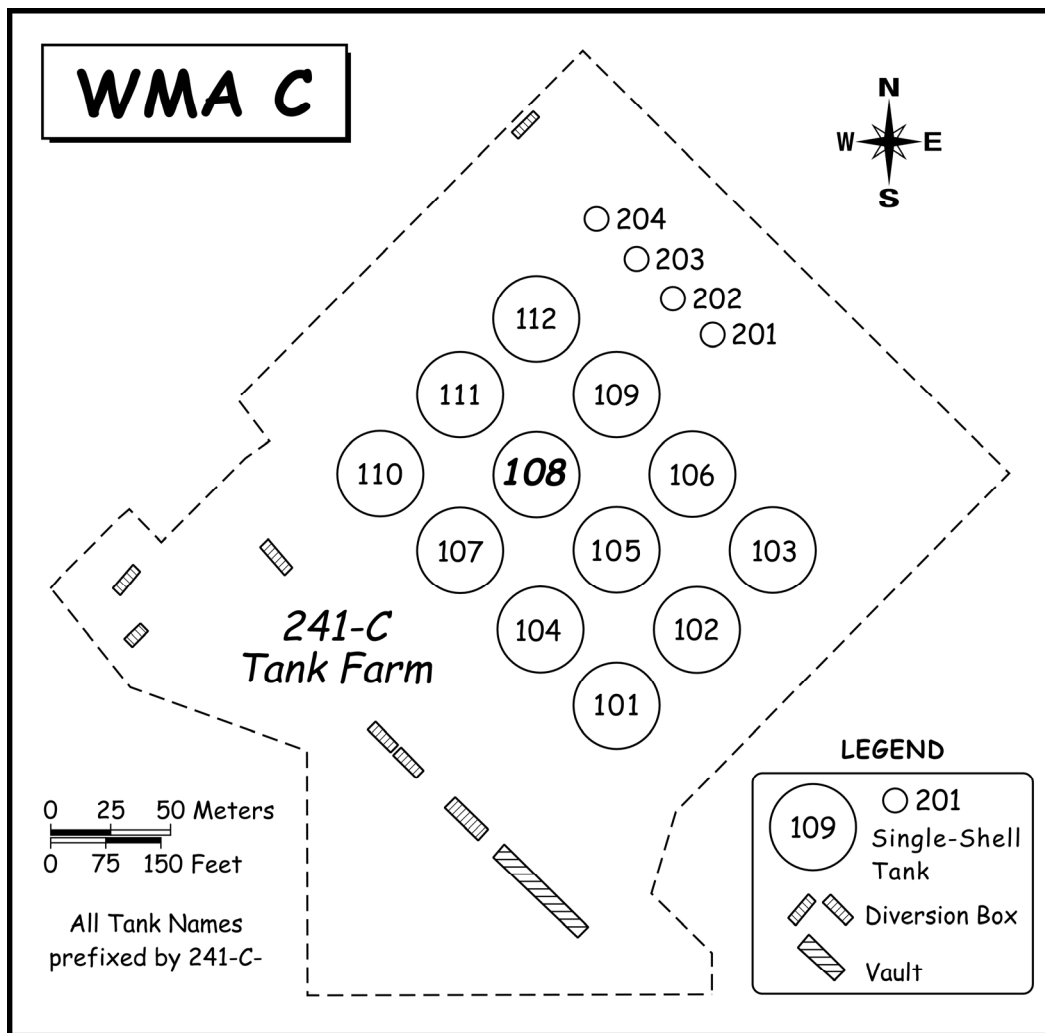
Although construction of tank C-108 was completed in 1944, the tank did not enter service until 1947. First-cycle decontamination (1C) waste from the bismuth phosphate process began cascading from tank 241-C-107 during 1947 (Agnew et al. 1997). Supernatant was pumped from tank C-108 during 1952, and the tank began receiving uranium recovery (UR) waste via the cascade line from tank 241-C-107. UR waste from tank C-108 was transferred to other tanks for in-tank ferrocyanide scavenging during 1956. A layering of solids settled from the UR waste in tank C-108. This layer would have been added to another layer of 1C solids predicted to have settled on the bottom of the tank during its early history. Tank C-108 subsequently received, staged, and then transferred out various waste streams; including but not limited to supernatant (most likely Plutonium-Uranium Extraction [PUREX] cladding waste [CWP] supernatant), waste from the Hot Semiworks Plant (HS), PUREX organic wash waste, ion exchange waste, reduction oxidation waste, N Reactor waste, decontamination waste, and laboratory waste.

Tank C-108 was removed from service in 1976 and saltwell pumping was completed in 1978, and intrusion prevention was completed on December 15, 1982. The tank was designated as interim stabilized on March 9, 1984 with roughly 250,000 L (66,000 gal) of sludge (deposited primarily during the 1950's and 1960's) and 42,000 L (11,000 gal) of drainable liquid remaining.

Tank C-108 underwent modified sluicing operations using tank 241-AN-106 supernatant starting on December 20, 2006, and completed on April 26, 2007. This first waste retrieval action had reached its limits leaving an estimated 25,700 L (6,800 gal) of waste remaining. The bulk of the remaining waste is comprised mostly of solids (hard heel) that are not mobilized by sluicing and are insoluble in the tank 241-AN-106 supernatant (Byrnes 2009).

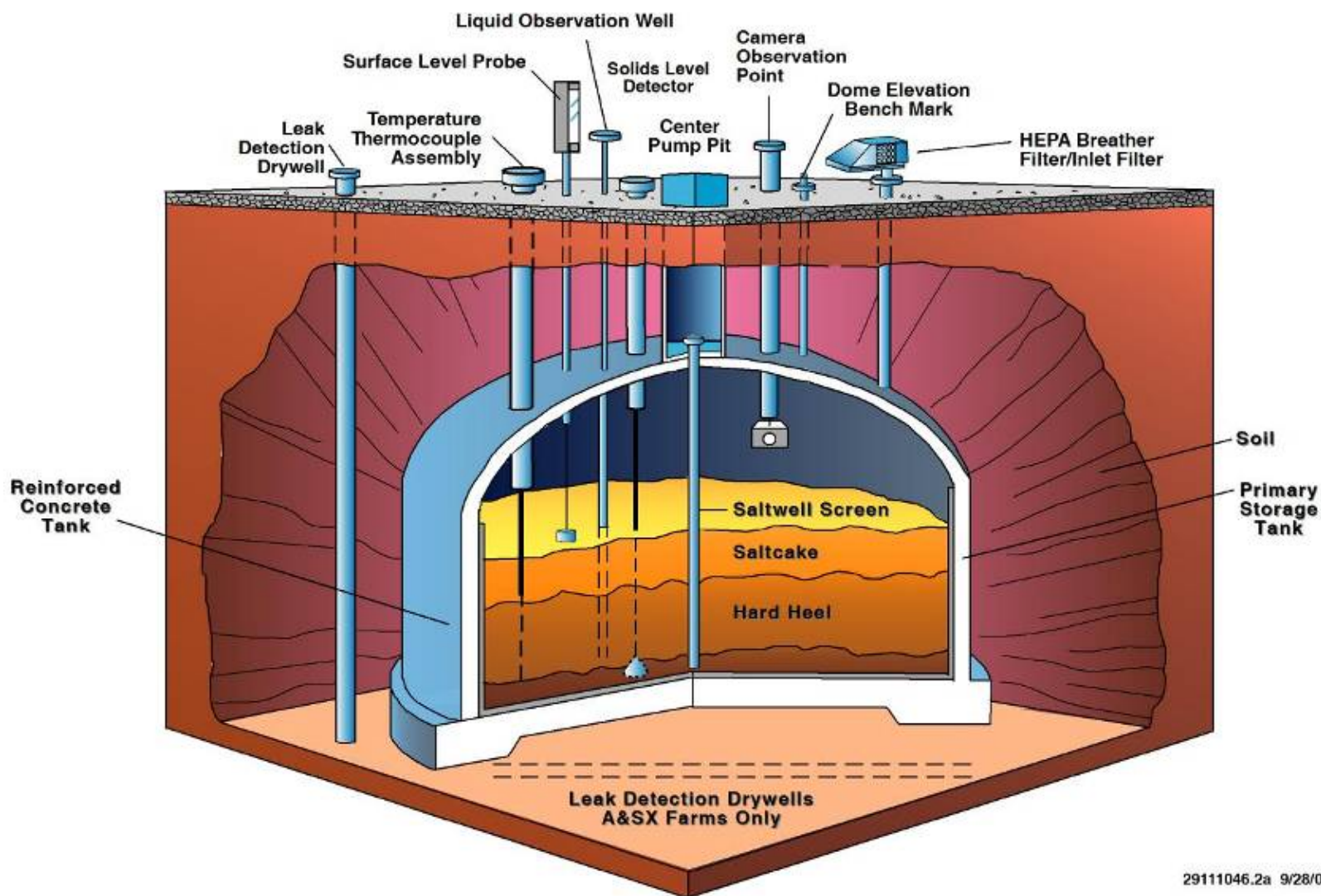
Subsequent to the modified sluicing effort, six solids grab samples were obtained from tank C-108 Riser 3 in July 2009 using the Off-Riser Sampling System and delivered to the 222-S Laboratory on the Hanford Site. A composite of these solid samples was received by PNNL in October 2009 for release model testing and characterization.





2009/DCL/C/001 (12/10)

**Figure 1.1.** Hanford Site 241-C Tank Farm



**Figure 1.2.** Generalized Profile View of a 100-Series Tank Such as Tank C-108 (Parker and Barton 2007)

## 2.0 Materials and Laboratory Test Methods

This section describes residual waste samples provided to PNNL and the analytical methods and various tests used to characterize the material and measure contaminant release.

### 2.1 Tank C-108 Residual Waste Samples

Residual waste samples from tank C-108 were collected by Washington River Protection *Solutions*, LLC after modified sluicing during retrieval activities in July 2009. The material from tank C-108 (sample 20578) was provided to PNNL on October 22, 2009 (Table 2.1). The sample is a composite sample (S09R000138) composed of equal amounts of composite samples A (S09T005880), B (S09T005881), and C (S09T005882) (Bushaw 2009). Figure 2.1 shows the as-received sample 20578.

**Table 2.1.** Tank C-108 Sample Provided to PNNL by 222-S Laboratory

Sample Number	Jar Size (mL)	Labcore Number	Net Weight of Sample Received (g)
20578	125	S09R000140	60



**Figure 2.1.** Tank C-108 Residual Waste (Sample 20578). (Solids identified by circles A, B, and C are green, black, and crystal nodule subsamples, respectively, which are described further in Sections 3.5 and 3.6 and were analyzed by XRD and SEM/EDS).

## 2.2 Tier 1 Tests

Tier 1 tests used in this study are described in this section. Studies of tank waste samples are generally completed using a tiered approach similar to the one developed for investigating contaminant fate and transport issues associated with past SST leaks in the vadose zone (e.g., Brown et al. 2007). Such an approach allows for initial (Tier 1) screening of samples using relatively inexpensive analytical techniques. This is followed by data analysis to determine the need for further testing (Tier 2). All laboratory activities were conducted in accordance with the requirements of Title 10, *Code of Federal Regulations*, Part 830.120, “Quality Assurance” (10 CFR 830.120), and the *Hanford Analytical Services Quality Assurance Requirements Documents* (HASQARD) (DOE-RL 2007). These requirements were implemented using PNNL’s online quality assurance (QA) plan, *Conducting Analytical Work in Support of Regulatory Programs*. PNNL’s QA plan is based on the requirements of DOE Order 414.1C as described in PNNL’s Standards-Based Management System, the HASQARD relevant elements of NQA-1-2004, as well as recognized industry standards (e.g., U.S. Environmental Protection Agency [EPA], American Society for Testing and Materials [ASTM], and the American National Standards Institute). Note that some qualitative analyses, such as those obtained by EDS, are not conducted according to these standards because of the nature of their measurement and sample geometry and the lack of qualitative internal standards.

### 2.2.1 Residual Waste Composition by Fusion Analysis and Acid Digestion

Bulk compositions of residual waste solids were determined using an acidic digestion procedure [EPA SW-846 Method 3052 (EPA 1996) with substitutions]<sup>1</sup> and a KOH-KNO<sub>3</sub> fusion-dissolution procedure.<sup>2</sup> The substitutions to EPA SW-846 Method 3052 consist of 1) decreasing the concentrated nitric acid (HNO<sub>3</sub>) volume from 9 mL to 5 mL, and 2) increasing the double-deionized (DDI) water volume from the recommended volume of 0 to 5 mL up to 10 mL. This method was used to measure metals concentrations and a limited number of nonmetals (e.g., P and S) in the residual waste. This method is not appropriate for the anion concentrations (e.g., NO<sub>3</sub>, Cl, F, and BO<sub>3</sub>) because of the addition of acids used in the procedure and analyses. The anion compositions were measured separately in solutions obtained by water leaching of the solids (see Section 2.2.7).

In the acid digestion procedure, approximately 300 mg of the sample is placed in a Teflon<sup>®</sup> microwave digestion vessel; 10-mL water, 5-mL 16 M HNO<sub>3</sub>, 2-mL 12 M HCl, and 1-mL 29 M HF are added to the sample; and the vessel is sealed and placed in a microwave-assisted digestion system.<sup>3</sup> The samples are treated at the EPA-recommended temperatures and times. The sample is then allowed to cool, and 0.45 g of boric acid (H<sub>3</sub>BO<sub>3</sub>) is added to the digestate and shaken by hand. Samples are filtered through a 0.45-μm pore-size syringe filter prior to analysis. There were no solids visible when the digestions were complete.

The KOH-KNO<sub>3</sub> fusion-dissolution procedure is the most commonly used method for solubilization of Hanford Site tank waste samples for chemical analysis by inductively coupled plasma-mass

---

<sup>1</sup> Lindberg MJ. 2009. *Operation of the MARS 5 Microwave Accelerated Reaction System*. AGG-MARS-001, Rev. 2. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>2</sup> Lindberg MJ. 2006. *Solubilization of Metals from Solids Using a KOH-KNO<sub>3</sub> Fusion*. AGG-ESL-001, Rev. 1. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>3</sup> Teflon is a registered trademark of E.I. du Pont de Nemours and Company.

spectroscopy (ICP-MS) and other methods (De Lorenzo et al. 1994; Simpson 1994; Fiskum et al. 2000; Smith et al. 2001). Benefits of the KOH-KNO<sub>3</sub> fusion-dissolution procedure include effective metathesizing of insoluble salts into acid soluble hydroxides; completing sample fusion at relatively low temperature (550°C) compared to other fluxing agents, such as 1100°C for the LiBO<sub>2</sub> fluxing agent; and allowing use of Ni or Zr crucibles, as opposed to the more costly Pt crucibles, for the fusion. As used in these studies, 300 mg of tank waste solids was mixed with 6 mL of a 30% KOH and 3% KNO<sub>3</sub> solution as a fluxing agent in a Ni crucible. The crucible was then placed in an oven at 95°C and allowed to evaporate to dryness, after which it was covered and transferred to a muffle furnace preheated to 550°C. Fusion was accomplished by heating the sample/flux mixture for 60 minutes at 550°C. After 60 minutes, the crucible was removed from the furnace and allowed to cool to room temperature. The fused solid was then dissolved in DDI water. The resulting solution was transferred to a 50-mL centrifuge tube. The crucible was then triple-rinsed with a 1:1 mixture of concentrated H<sub>2</sub>SO<sub>4</sub> acid and 1M NaHSO<sub>3</sub>, and these solutions were also added to the centrifuge tube. An additional 5 to 15 mL of the H<sub>2</sub>SO<sub>4</sub>:NaHSO<sub>3</sub> solution was added to the centrifuge tube to facilitate total sample dissolution. After sample dissolution was complete, the final solution volume in each centrifuge tube was determined gravimetrically and corrected for solution density. An aliquot of this solution is then filtered with a 0.45-μm syringe filter and submitted for analysis.

Table 2.2 lists the digestion factors (wet solid-to-solution ratios) for residual waste samples 20578 used for the KOH-KNO<sub>3</sub> fusion treatment and the EPA acid digestions. These factors were calculated from the wet weight of waste material divided by the volume of extracting solution. The digestion factors were then multiplied by the percentage solids, as determined from moisture content analysis, to convert to a dry weight basis. The dissolved metal concentrations of the filtered solutions were then analyzed by a combination of methods, including ICP-MS and inductively coupled plasma-optical emission spectroscopy (ICP-OES). These analytical methods are described in Lindberg and Deutsch (2003).

**Table 2.2.** Digestion Factors for Residual Waste Solids Used for the KOH-KNO<sub>3</sub> Fusion Treatment and EPA Acid Digestion Treatment

Treatment	Sample Number	Dry Weight Corrected Digestion Factor (g/L)
KOH-KNO <sub>3</sub> fusion	20578	12.25
	20578 Dup1	12.01
	20578 Dup2	9.98
EPA Method 3052 acid digestion	20578	7.17
	20578 Dup	8.03
Dup = Duplicate.		

## 2.2.2 Moisture Content

The moisture contents of the tank waste samples were measured to calculate dry weight concentrations for constituents in the waste. Dry weight concentrations provide a consistent measurement unit for comparison purposes that eliminates the effect of variable water content on sample concentrations.

Gravimetric water content of the waste material was determined using the ASTM procedure D2216-98, *Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass* (ASTM 1998), with the following minor exceptions: 1) the volume of sample recommended was decreased due to radiological concerns; and 2) the sample was dried at a lower oven temperature (105°C) for a longer period of time to prevent dehydration of the solids.<sup>4</sup>

Residual waste samples were placed in tared containers, weighed, and dried in an oven until a constant weight was achieved, usually 24 to 48 hours. The container was then removed from the oven, sealed, cooled, and weighed. All measurements were performed using a calibrated balance. The gravimetric water content is computed as the percentage change in waste sample weight before and after oven drying (i.e.,  $[(\text{wet weight} - \text{dry weight}) / \text{dry weight}]$ ).

### **2.2.3 Carbon Analysis**

The analysis of the C content of solid and liquid samples is described in this section. Carbon analyses were conducted to determine total inorganic C (TIC) and total organic C (TOC) in the residual waste samples and liquid samples. TIC is used as an indirect measure of carbonate concentrations that are used for conducting the geochemical modeling (saturation index calculations).

#### **2.2.3.1 Carbon Content of Solids**

The C content of solid samples was determined by the hot persulfate method.<sup>5</sup> In this method, samples are treated by wet chemical oxidation by heating at 92°C to 95°C with a solid potassium persulfate oxidant and liquid Ag-ion catalyst. Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) is also used to convert C to CO<sub>2</sub> (carbon dioxide). The CO<sub>2</sub> is swept away by an O<sub>2</sub> carrier gas and measured in a UIC Coulometrics Acid Module. The method uses a two-step process allowing a separate measurement of TIC and TOC on the same sample. In this process, the sample is first acidified with heated sulfuric acid, converting inorganic carbonates to CO<sub>2</sub> (i.e., TIC analysis); then, the persulfate solids and Ag-catalyst solution is added and the organic C remaining in the sample is converted to CO<sub>2</sub> for TOC measurement. Total C (TC) for a solid sample is calculated from the sum of TIC and TOC.

### **2.2.4 Leach Tests**

#### **2.2.4.1 Single-Contact Leach Tests**

Leachable inorganic constituents in the residual waste were determined using DDI water, a fresh cement pore water simulant [i.e., Ca(OH)<sub>2</sub> saturated DDI water] and an aged cement pore water stimulant [i.e., calcite (CaCO<sub>3</sub>) saturated DDI water]. The tests were prepared by adding 30 mL of the leachant to a quantity of moist residual waste ranging from 0.200 to 0.600 g contained in a 50-mL polypropylene centrifuge tube. Concentrations on a dry residual waste mass basis were determined using the measured moisture content of the waste. The centrifuge tube was sealed, briefly shaken by hand, and then placed

---

<sup>4</sup> Lindberg MJ. 2005. *Soil Water Content*. AGG-WC-001, Rev. 0. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>5</sup> Baldwin DL. 2005. *Carbon Measured in Solids, Sludge, and Liquid Matrices*. RPG-CMC-385, Rev. 0. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

on a mechanical orbital shaker for 1 month. After being shaken for the predetermined time, the tube was placed in a centrifuge and spun at 4000 revolutions per minute (rpm) for 20 minutes. The supernatant was then carefully decanted and filtered through 0.45- $\mu\text{m}$  pore-size membrane. Aliquots of these extracts were preserved appropriately and subjected to quantitative chemical and radiochemical analysis. More details can be found in ASTM procedure D3987-85 *Standard Test Method for Shake Extraction of Solid Waste with Water* (ASTM 1999).

Simulated cement pore water solutions were used to evaluate the leachability of constituents from residual waste contacting cementitious grout that may be used to fill the tank during closure. A  $\text{Ca}(\text{OH})_2$ -saturated solution ( $\sim 1.4 \text{ g/L}$  @  $25^\circ\text{C}$ ) was prepared to simulate a leachant produced by fresh cement. Care was taken to minimize contact of the solution with air, because  $\text{CO}_2$  in air is very soluble in water at high pH and the resulting dissolved carbonate will precipitate  $\text{CaCO}_3$  in the  $\text{Ca}(\text{OH})_2$ -saturated solution. Air space in the containers was minimized and the vessel was tightly sealed to limit leakage of air into the vessel. The pH of an aliquot of the  $\text{Ca}(\text{OH})_2$  solution was measured as well as the dissolved calcium concentration. This solution was used to leach the sludges in the same manner as the abovementioned DDI water leachant.

A  $\text{CaCO}_3$ -saturated solution was used to simulate an aged cement pore water. The calcite-saturated solution was prepared by adding excess powdered calcite to deionized water and stirring or shaking the mixture for 24 hours. This solution was also used to leach the sludges in the same manner as the abovementioned DDI water and  $\text{Ca}(\text{OH})_2$  leachants.

Analysis results for the initial leachants solutions are provided in Table 2.3. Thermodynamic equilibrium modeling conducted with Geochemist's Workbench<sup>®</sup> indicates that the  $\text{Ca}(\text{OH})_2$  saturated leachant solution was significantly oversaturated with respect to portlandite [a mineral with the composition of  $\text{Ca}(\text{OH})_2$ ] with a saturation index (SI) value of 1.37. The  $\text{CaCO}_3$  saturated leachant solution was very near saturation with respect to calcite with an SI value of 0.02. See Section 2.5 for the definition of SI.

At the end of the extraction tests, the remaining residual waste was saved for solid phase analysis by XRD and SEM/EDS. Selected samples were also analyzed by synchrotron based x-ray absorption techniques.

**Table 2.3.** pH, Ca, and Alkalinity, for Initial Leachant Solutions

Sample Number	pH	Ca	Total Alkalinity (as $\text{CaCO}_3$ ) at pH 4.5 Endpoint
		( $\mu\text{g/L}$ )	( $\mu\text{g/L}$ )
DDI	7.80	(5.17E+01)	(7.83E+03)
$\text{Ca}(\text{OH})_2$ Saturated Solution	12.62	8.94E+05	2.01E+06
$\text{CaCO}_3$ Saturated Solution	7.82	5.04E+03	6.59E+04
Concentrations listed in parentheses were <EQL. EQL = Estimated quantitation limit.			



#### 2.2.4.2 Periodic Replenishment Leach Tests

Periodic replenishment tests were also conducted on the residual waste samples. These tests were conducted with DDI water,  $\text{Ca}(\text{OH})_2$ -saturated, and  $\text{CaCO}_3$ -saturated leachants using the similar procedures described above for the single-contact leach tests. In the replenishment tests, the leachant was removed and replaced with an equal volume of fresh solution repeatedly for five times total. The contact period was 7 days. At the end of the extraction period, the samples were prepared and handled in the same manner as the single-contact water extracts for each repetitive step.

#### 2.2.5 pH

The pH of the solutions was measured using EPA SW-846 Method 9040C (EPA 2004) with a modification.<sup>6</sup> The modification consists of using a solid-state pH electrode instead of the recommended glass electrode.

#### 2.2.6 Alkalinity

The sample alkalinity was measured by standard titration. A volume of standardized sulfuric acid was added to the sample to an endpoint of pH 4.5 to measure total alkalinity. Alkalinity is reported in terms of an equivalent mass of  $\text{CaCO}_3$ . The alkalinity procedure follows Standard Method 2320 B, “Alkalinity by Titration” (Clesceri et al. 1998).

#### 2.2.7 Anion Analysis

Anion analysis was performed using an ion chromatograph (IC) following the technical procedure AGG-IC-001 (Lindberg 2004).<sup>7</sup> Fluoride ( $\text{F}^-$ ), chloride ( $\text{Cl}^-$ ), bromide ( $\text{Br}^-$ ), nitrate ( $\text{NO}_3^-$ ), nitrite ( $\text{NO}_2^-$ ), phosphate ( $\text{PO}_4^{3-}$ ), and sulfate ( $\text{SO}_4^{2-}$ ) were separated on a Dionex®<sup>8</sup> AS17 column with a gradient elution technique from 1-mM to 35-mM NaOH and measured using a conductivity detector. This methodology is a substitution for EPA SW-846 Method 9056A (EPA 2007), with the exception of using gradient elution with NaOH instead of the recommended isocratic elution with a  $\text{HCO}_3$  buffer.

#### 2.2.8 Cations and Trace Metals

Major cation analysis (including Al, Si, Ca, Mg, Na, K, Fe, and Mn) was performed by ICP-OES following PNNL-AGG-ICP-AES (Baum 2008).<sup>9</sup> Selected trace metals analysis (<sup>99</sup>Tc and U isotopes) was performed by ICP-MS following PNNL-AGG-415 (Clayton 2008).<sup>10</sup>

---

<sup>6</sup> Valenta MM. 2009. *pH Measurement*. AGG-pH-001, Rev. 1. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>7</sup> Lindberg MM. 2004. *Determinations by Ion Chromatography (IC)*. AGG-IC-001. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>8</sup> Dionex is a registered trademark of Dionex Corporation.

<sup>9</sup> Baum SR. 2008. *Inductively Coupled Plasma -Optical Emission Spectrometry (ICP-OES) Analysis*. PNNL-AGG-ICP-AES, Rev. 2. Unpublished PNNL technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>10</sup> Clayton ET. 2008. *Inductively Coupled Plasma Mass Spectrophotometry (ICP-MS) Analysis*. PNNL-AGG-415, Rev. 2, Pacific Northwest National Laboratory, Richland, Washington.



For both ICP-OES and ICP-MS, high-purity calibration standards were used to generate calibration curves and to verify continuing calibration during the analysis. Dilutions of 10 and 5 times were made for each sample and analyzed to investigate and correct for matrix interferences.

### 2.2.9 $^{237}\text{Np}$ and $^{239}\text{Pu}$ Analysis

Use of ICP-MS is a widely accepted method for the determination of trace metals in solution and was used in this study for determining  $^{237}\text{Np}$  and  $^{239}\text{Pu}$ . The instrument requires user calibration using multi-element standards with concentrations ranging from 5 pg/mL to 20 ng/mL. One area of concern in using ICP-MS to measure actinide elements in tank waste extracts is the proximity in atomic mass of the elements of interest. It can be difficult to measure elements separated by only one atomic mass unit (AMU) when one element is present in trace quantities ( $^{237}\text{Np}$  and  $^{239}\text{Pu}$ ) while another element is present in macroscopic concentrations ( $^{238}\text{U}$ ). Under these circumstances, peak tailing from  $^{238}\text{U}$  can extend into the regions corresponding to  $^{237}\text{Np}$  and  $^{239}\text{Pu}$ , resulting in erroneously high reporting of total  $^{237}\text{Np}$  and  $^{239}\text{Pu}$ . Standard ICP-MS analysis of the acid extracts of tank C-108 residual waste material for uranium ranged from 18.3 to 12.1 mg/L. Previous studies indicate that these concentrations of uranium do not cause an interference at AMU of 237 or 239. With these low levels of uranium,  $^{237}\text{Np}$  and  $^{239}\text{Pu}$  were able to be quantitatively analyzed without special analytical techniques. The estimated quantification limits (EQLs) for  $^{237}\text{Np}$  and  $^{239}\text{Pu}$  were 0.9 and 2.3  $\mu\text{g/L}$ , respectively.

### 2.2.10 Radiochemical Analysis

In addition to the radionuclides  $^{99}\text{Tc}$ ,  $^{237}\text{Np}$ ,  $^{238}\text{U}$ , and  $^{239}\text{Pu}$  that were analyzed in solution by ICP-MS, short-lived radionuclides ( $^{137}\text{Cs}$  and  $^{90}\text{Sr}$ ) were analyzed by conventional counting methods as described below.

#### 2.2.10.1 $^{137}\text{Cs}$ Analysis

Cesium-137 was measured in solution extracts by GEA. The analyses were made using 60% efficient intrinsic-germanium gamma detectors. All germanium counters were efficiency calibrated for distinct geometries using mixed gamma standards traceable to the National Institute of Standards and Technology (NIST). Direct solids, acid extracts, and water extracts were analyzed for gamma energy. Spectral analysis was conducted using libraries containing most mixed-fission products, activation products, and natural decay products. Control samples were run throughout the analysis to ensure correct operation of the detectors. The controls contained isotopes with photo peaks spanning the full detector range and were monitored for peak position, counting rate, and full-width half-maximum. Details are found in an internal PNNL procedure.<sup>11</sup>

---

<sup>11</sup> Lindberg MJ. 2004. *Gamma Energy Analysis, Operation, and Instrument Verification using Genie2000 Support Software*. AGG-RRL-001, Rev. 3. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

### 2.2.10.2 <sup>90</sup>Sr Analysis

Aliquots of filtered acid extracts, fusions, and water extracts were diluted in 8 M HNO<sub>3</sub> and submitted for Sr separation and analysis by internal PNNL procedure.<sup>12</sup> A 0.1-5 mL aliquot of sample was spiked with <sup>85</sup>Sr tracer and passed through a SrSpec<sup>®</sup> column to capture Sr.<sup>13</sup> The columns were washed with 10 column volumes (20 mL) of 8 M nitric acid. The Sr was eluted from the SrSpec column into glass liquid scintillation vials using 15 mL of deionized water. The vials were placed under a heat lamp overnight to evaporate the water to dryness. A 15-mL Optifluor<sup>®</sup> scintillation cocktail was added to each vial. Gamma spectroscopy was used to determine the chemical yield from the added <sup>85</sup>Sr tracer. The samples were then analyzed by LSC to determine the amount of <sup>90</sup>Sr originally present in the residual waste sample. A matrix spike, a blank spike, a duplicate, and blanks were run with each sample set to determine the efficiency of the separation procedure as well as the purity of reagents.

## 2.3 X-Ray Powder Diffractometry

Standard bulk powder XRD techniques were used to identify crystalline phases present in the following tank C-108 residual waste samples:

- Composite subsample – A mixture of all phases visible in the jar sample of as-received residual waste.
- Composite duplicate subsample – A duplicate of the composite sample.
- Green nodule subsample – Handpicked particles of green colored solid(s) in the jar sample of as-received residual waste.
- Black nodule subsample – Handpicked particles of black colored solid(s) in the jar sample of as-received residual waste.
- Crystal nodule subsample – Handpicked particles of crystalline-appearing phase in the jar sample of as-received residual waste. This solid consists of rounded, dense-looking, tan grains that appear to consist primarily of a single phase and can be as much as a couple of centimeters in largest dimension. In the jar sample, these grains usually have some light tan, fine grain material adhering to their surfaces.
- One-month deionized water-leached sample – Solid material remaining from the 1-month, single-contact deionized water leach test.
- One month Ca(OH)<sub>2</sub> leached sample – Solid material remaining from the 1-month, single-contact Ca(OH)<sub>2</sub>-solution leach test.
- One month CaCO<sub>3</sub> leached sample – Solid material remaining from the 1-month, single-contact CaCO<sub>3</sub>-solution leach test.

Because the sizes of particles in the jar sample of as-received residual waste (see Figure 2.1) were generally too large to use as-is for XRD analysis, subsamples of the as-received residual waste were ground prior to preparation of the XRD mounts. The hazardous nature of this residual waste precluded

---

<sup>12</sup> Lindberg MJ. 2004. *Tc99 and Sr90 Analysis Using Eichrom TEVA-Spec and Sr-Spec Resin*. AGG-RRL-003, Rev. 3. Unpublished internal technical procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>13</sup> SrSpec is a registered trademark of Eichrom Technologies.

the grinding of these subsamples to a particle size range considered optimum for XRD analysis (less than ~10  $\mu\text{m}$ ). However, the ground solids used for the sample mounts did produce XRD patterns (see Section 3.5 and Appendix B) sufficient for phase identification. The composite and composite duplicate samples were taken from material previously heated to 90°C for 48 hours for moisture content analysis. After heating, solids were placed in two LSC sample vials and carefully ground in the vials with a scoopula. Particles of crystal, green, and black nodule solids were carefully handpicked from the jar residual waste sample, placed in separate LSC vials, and allowed to air dry. The purpose of handpicking the selected material was to maximize the amount of these visibly different types of solids in the respective XRD mounts. This process did not—nor was it expected to—produce samples that contained only a single particular solid phase. Due to the dose rates, coarse particle size, and supposed hardness (based on appearance) of the crystal, green, and black nodule solids, these subsamples were not ground in the LSC vials like the composite samples. To contain any solids dispersed during the grinding process, the subsamples of crystal, green, and black nodule solids were ground with aluminum oxide mortar and pestle that was placed inside a heavy duty plastic bag located in a fume hood. Each ground subsample was carefully removed from the mortar, placed in a separate LSC vial, and isolated from the mortar for eventual removal from the plastic containment bag. Based on resistance to grinding, the black nodule material appeared to be slightly harder than the crystal and green nodule solids, but all three solids were softer than initially presumed. These ground samples are the parent materials for the sample mounts prepared for XRD and SEM/EDS analyzes (discussed in next section).

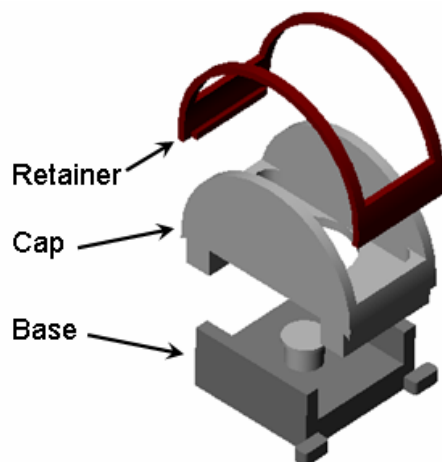
Because the residual waste samples are highly radioactive dispersible powders, it was necessary to prepare the XRD mounts of these samples inside a fume hood regulated for handling radioactive materials. Milligram quantities of each parent sample was transferred to separate LSC vials and mixed with a trace quantity of reference-material corundum powder ( $\alpha\text{-Al}_2\text{O}_3$ , alumina) (NIST SRM 676 [NIST 2005]). The reference-material corundum powder was added to each sample as an internal  $2\theta$  standard to correct for any observed peak shifts due to slight misalignments of the mounted samples. A couple drops of collodion solution were then added to each vial and the resulting slurry transferred by pipette onto a circular-shaped sample platform (1-cm [0.39-in.] diameter). The collodion solution consists of 2% nitrocellulose dissolved in amyl acetate, and is an x-ray amorphous, viscous binder commonly used to make random powder mounts for XRD when only a limited amount of sample is available. Each sample-containing platform was then placed on top of the post located on the base inside a disposable XRD specimen holder (Figure 2.2). The disposal specimen holder was designed specifically for safe handling of dispersible powders containing highly radioactive or hazardous materials (Strachan et al. 2003). After allowing each sample mount to air dry, the holder was assembled and a piece of Kapton<sup>®</sup> film was placed between the cap and the retainer.<sup>14</sup> The holder was sealed with wicking glue and removed from the fume hood for XRD analysis. For most samples insufficient corundum was added to allow for correction of any observed peak shifts. As an alternative, any observed peak shifts were corrected using gibbsite as a standard because significant quantities of gibbsite were known to occur in these samples.

Each sample was analyzed using a Scintag XRD unit equipped with a Peltier thermoelectrically cooled detector and a copper x-ray tube. The diffractometer was operated at 45 kV and 40 mA. Individual scans were obtained from 2° to 65° $2\theta$  with a step size of 0.02° and dwell time of 2 seconds. Scans were collected electronically and processed using the JADE<sup>®</sup> XRD pattern-processing software.<sup>15</sup>

---

<sup>14</sup> Kapton is a registered trademark of E.I. du Pont de Nemours and Company.

<sup>15</sup> Jade is a registered trademark of Materials Data, Inc.

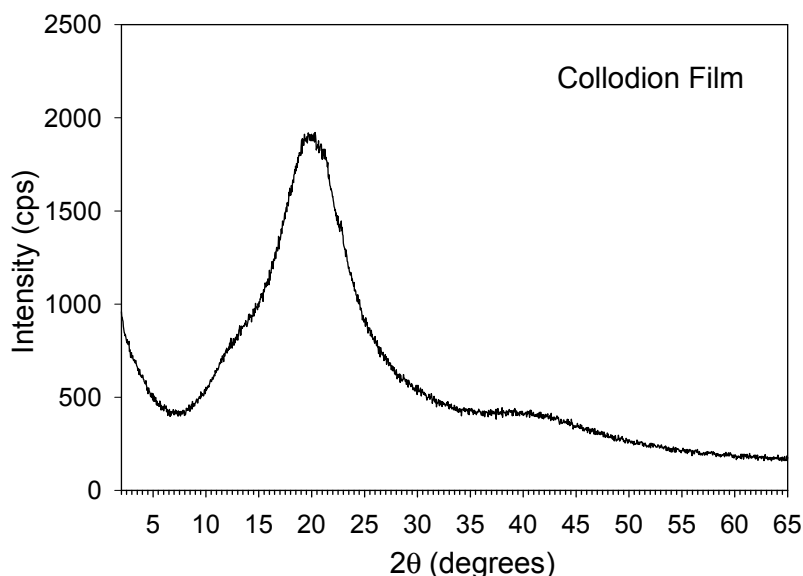


**Figure 2.2.** Exploded Schematic View of the XRD Sample Holder (Kapton<sup>®</sup> film not shown) (see Strachan et al. 2003)

A sample consisting of only a dry film of the collodion solution was previously prepared and analyzed by XRD by Krupka et al. (2004) so that its contribution relative to the background signals of the XRD patterns for the residual waste samples could be quantified. The resulting XRD pattern for the collodion solution film is shown in Figure 2.3. The most obvious feature of this diffraction pattern is the broad peak positioned between  $10^\circ$  and  $30^\circ 2\theta$ . The symmetry of this peak is characteristic of those resulting from the XRD of amorphous (noncrystalline) material. Although subtracting the collodion background from residual waste XRD patterns allows for better phase matching, this process may eliminate minor reflections and inconspicuous features of a pattern. Therefore, each as-measured XRD pattern was examined before and after background subtraction to ensure that the integrity of the pattern was maintained. For background subtraction, the JADE<sup>®</sup> software provides the user with control over the selection of background-subtraction points. This process allows a better fit to  $2\theta$  regions under broad reflections, such as those resulting from amorphous materials. On average, 30 to 40 background points were selected from each XRD pattern, and a cubic-spline curve was then fit through each set of points. Adjustments to this curve were made by selecting additional background points in regions of a pattern that were difficult to fit. Once a well-matched curve was fitted to a pattern, the background was subtracted from each as-measured XRD pattern, resulting in a smooth tracing.

Identification of the mineral phases in the background-subtracted patterns was based on a comparison of the XRD patterns measured for the residual waste samples with the mineral powder diffraction files (PDF<sup>™</sup>) published by the Joint Committee on Powder Diffraction Standards (JCPDS) International Center for Diffraction Data (ICDD). Generally, a crystalline phase must be present at greater than ~5-10 wt% of the total sample mass (greater than 1 wt% under optimum conditions) to be readily detected by XRD. In general, the measured peak intensities depend on several factors, including the combined mass of each crystalline phase in the sample. Due to the physical characteristics of these residual waste samples, such as high radioactivity, high dispersibility, and variable moisture content, the mass of residual waste combined with the collodion solution for each XRD mount could not be controlled or easily determined. Dissimilarities in mineral segregation (settling) resulting from the different densities of minerals mixed with the collodion solution and associated effects on relative peak intensities also influence the overall pattern intensity. The combined effect of these factors could have some effect on the

characteristic mineral peak intensities, which precluded quantitative comparisons of peak intensities for equivalent reflections in background-subtracted XRD patterns for different residual waste samples.



**Figure 2.3.** XRD Pattern for Collodion Film Measured in the Absence of Any Residual Waste Material (from Krupka et al. 2004)

## 2.4 Scanning Electron Microscopy/Energy Dispersive Spectrometry Analysis

SEM/EDS analyses were used to characterize the morphologies and compositions of solid phases present in the following tank C-108 residual waste samples:

- Composite sample
- Green nodule sample
- Black nodule sample
- Crystal nodule sample
- One month DDI water-leached sample
- One month  $\text{Ca}(\text{OH})_2$  leached sample
- One month  $\text{CaCO}_3$  leached sample.

Details of the samples listed above are described at the beginning of Section 2.3.

Each mount used for SEM/EDS consisted of double-sided C (carbon) tape attached to standard Al mounting stub. For each mount, small aliquots of each parent residual waste material were placed on the exposed upper surface of the C tape using a micro spatula. Each mount was then coated with C using a vacuum sputter-coater to improve the conductivity of the samples and thus the quality of the SEM images and EDS signals.

A JEOL JSM-840 SEM was used for high-resolution imaging of micrometer/submicrometer-sized particles in the residual waste samples. The EDS system provided qualitative elemental analysis for scanned areas of particles. The SEM is equipped with an INCA Energy EDS System<sup>16</sup> to automate the collection of EDS spectra over multi-micrometer-sized areas of an SEM-imaged sample. The EDS software was calibrated to a Cu reference standard mounted on a specimen holder. Operating conditions typically consisted of 20 keV for SEM imaging and 20 keV, 100 live seconds<sup>17</sup> for the EDS analyses. A limited number of SEM/EDS analyses of the 1-month deionized sample were also completed at 30 or 35 keV to check for the presence of the K $\alpha$  x-ray emission lines for Tc at 18.37 and 18.25 keV. The EDS analyses are limited to elements with atomic weights heavier than B (boron). Compositions determined by EDS are qualitative and have large uncertainties resulting from alignment artifacts caused by the variable sample and detector configurations that exist when different particles are imaged by SEM.

Electron micrographs of high-resolution secondary electron (SE) images and backscattered electron (BSE) images were obtained as digital images and stored in electronic format. To help identify particles that contain elements with large atomic numbers, such as U, the SEM was typically operated in the BSE mode. SE are low-energy electrons ejected from the probed specimen as a result of inelastic collisions with beam electrons, whereas BSEs are primary electrons emitted as a result of elastic collisions. BSE emission intensity is a function of the element's atomic number – the larger the atomic number, the brighter the signal. BSE images are obtained in exactly the same way as SE images.

The entire area of each SEM mount was examined by SEM at low magnification (typically 10 to 30 times) to identify those particles and surface features that were typical or unusual for the sample. During this examination, SEM micrographs were recorded at low magnification for one or more representative areas of the mount to provide a general perspective of the sizes, types, and distributions of particles that comprise each SEM mount. Within these imaged regions, additional SEM micrographs were recorded at greater magnifications to provide a more detailed representation of the particles' characteristics, and selected points on these particles were then analyzed by EDS. Depending on the perceived importance of such particles, regions on these particles were sometimes analyzed by SEM and EDS at even greater magnifications.

## 2.5 Geochemical Modeling

Geochemical modeling was used to determine mineral saturation indices (SIs) to identify solid phases potentially in equilibrium with the leachate compositions, and to evaluate the significance of aqueous complexation on the solubility of potentially important mineral phases. The SI is defined in Equation (3.1):

$$SI = \log (Q/K_{sp}) \quad (3.1)$$

where Q is the activity product and  $K_{sp}$  is the mineral solubility product at equilibrium at the temperature of interest. Minerals with SI values near zero (within  $\pm \sim 0.5$ ) are generally considered to be near

<sup>16</sup> Oxford Instruments, Concord, Massachusetts.

<sup>17</sup> Live time (real time less dead time) is when the EDS system is available to detect incoming x-ray photons. Dead time is the portion of the total analyzing time that is actually spent processing or measuring x-rays. While each x-ray pulse is being measured, the system cannot measure another x-ray that may enter the detector and is, therefore, said to be "dead."

equilibrium, more positive values are considered oversaturated, and more negative values are considered undersaturated with respect to the solution composition.

The React module of Geochemist's Workbench<sup>®</sup> (GWB) version 8.0 (Bethke and Yeakel 2009) was used to calculate the mineral SIs for these solutions. The thermodynamic database, thermo.com.V8.R6+.dat, used for the modeling calculations was modified to include solubility products for *čejkaite*  $[\text{Na}_4(\text{UO}_2)(\text{CO}_3)_3]$  (Felmy et al. 2005); *becquerelite*  $[\text{Ca}(\text{UO}_2)_6\text{O}_4(\text{OH})_6 \cdot 8\text{H}_2\text{O}]$  (Rai et al. 2002); Na diuranate hydrate  $[\text{Na}_2\text{U}_2\text{O}_7 \cdot x\text{H}_2\text{O}]$  (Yamamura et al. 1998); *andersonite*  $[\text{Na}_2\text{Ca}(\text{UO}_2)(\text{CO}_3)_3 \cdot 5\text{H}_2\text{O}]$  and *urancalcarite*  $[\text{Ca}(\text{UO}_2)_3(\text{CO}_3)(\text{OH})_6 \cdot 3\text{H}_2\text{O}]$  (Chen et al. 1999); *autunite*  $[\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 3\text{H}_2\text{O}]$  and *uranyl orthophosphate*  $[(\text{UO}_2)_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}]$  (Gorman-Lewis et al. 2009) and *natrophosphate*  $(\text{NaF} \cdot 2\text{Na}_3\text{PO}_4 \cdot 19\text{H}_2\text{O})$  (Weber et al. 2000); and the stability constant for the dissolved species  $\text{Ca}_2\text{UO}_2(\text{CO}_3)_3(\text{aq})$  (Kalmykov and Choppin 2000).

The utility of geochemical modeling is limited to phases that come to equilibrium within the extraction contact period, whose solubility and concentrations are such that they do not completely dissolve in the extraction fluid, and for which thermodynamic constants are available.

## 3.0 Laboratory Results

This section provides results of tests conducted on residual waste samples 20578 from tank C-108. Section 3.1 includes a description of the residual waste composition obtained from acid digestion methods. The results of the residual waste leach test and analysis of the residual waste supernatant in sample 20406 are described in Section 3.2. Residual waste characterization data generated by using XRD and SEM/EDS are discussed in Sections 3.3 and 3.4, respectively.

### 3.1 Residual Waste Composition

An important component of contaminant release rate calculations is an accurate measurement of the total concentrations of the contaminants in the source material. As described in Section 2.2.1, the total metals and radionuclide concentrations of the residual wastes were measured using an acid digestion method. The results of these analyses are described in this section. The anionic (nonmetal) composition of the residual waste was estimated by water extraction as part of the Tier 1 analyses (Section 2.2.7). Tier 1 anion results are discussed in Section 3.2.

Table 3.1 lists the moisture content ( $[(\text{wet wt} - \text{dry wt})/\text{dry wt}] \times 100\%$ ) of the tank C-108 residual waste samples used for the EPA acid digestions. These values are used with the digestion factors (Table 2.2) to convert the solution analyses of the extracts from the treatments to dry weight solid concentrations. The moisture contents of the residual waste samples ranged from 39.0% to 40.1%.

**Table 3.1.** Moisture Contents of Tank C-108 Residual Waste Samples

Sample Number	Moisture Content
20578	39.01%
20578 Dup1	41.25%
20578 Avg	40.13%
Dup = Duplicate sample.	

Concentrations listed in parentheses in the following tables are defined as less than the EQL but greater than a zero instrument signal (concentration of zero). These values are reported for informational purposes only. They may reflect actual concentrations that are real but have larger associated uncertainties than values above the EQL or may reflect values that were calculated from the instrument's background signal and are not representative of actual residual waste composition. The EQL of an element is determined by analyzing a suite of continuing calibration verification (CCV) standards at the beginning and end of each analytical run. The lowest CCV standard that is within  $\pm 10\%$  of its certified value is multiplied by the dilution factor for the sample to determine the EQL for the element for the particular analytical run. The EQL may vary with each analysis, depending on sample matrix, dilution factors, and instrument performance. Concentration values listed as not detected (ND) in the tables refer to instrument measurements that are less than zero.



### 3.1.1 EPA Acid Digestion Results

Composition results for tank C-108 partial retrieval residual waste (sample 20578) determined using the EPA acid digestion method are presented in this section. Results are reported for the primary sample, a duplicate, and the average of the two. The results of elemental analyses determined by ICP-OES analysis are listed in Table 3.2. Boron values from acid digestion are not reported because boric acid was used in the digestion process. Among the suite of 30 elements that were analyzed, 21 elements were present above the instrumental detection limits in all replicates. These elements include Al, Ba, Be, Bi, Ca, Cr, Cu, Fe, Li, Mg, Mn, Ni, P, Pb, Sr, Zn, Na, S, Ti, Zr, and U. Si and Tl concentrations were not quantifiable due to matrix interferences. On a percentage basis, the dominant elemental components determined by ICP-OES are Na at 15.5%, Al at 12.3%, P at 4.0%, Fe at 1.6%, Ni at 0.22%, U at 0.18%, and Mn at 0.13%.

The elemental concentrations analyzed by ICP-MS are listed in Table 3.3. For some metals, ICP-MS can attain lower detection limits than ICP-OES, allowing more accurate measurements of trace metal concentrations in the residual waste samples. Among the trace elements listed in the tables, Cd, Cr, Ru, and Pb were present in concentrations greater than their respective EQLs. Ag could not be reported as a result of matrix interference in the samples. Comparisons of the ICP-MS results can be made with the ICP-OES results for Cr, Cu, and Pb. Good agreement between the ICP-MS and the ICP-OES results is observed for each of these metals, despite the fact that the ICP-EOS results for Cr and Cu and the ICP-MS result for Cu were below their respective EQLs.

The concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  measured by ICP-MS are listed in Table 3.4. The average  $^{99}\text{Tc}$  concentration was  $0.636\text{ }\mu\text{g/g}$  waste ( $0.0108\text{ }\mu\text{Ci/g}$ ). The average  $^{238}\text{U}$  concentration was  $1870\text{ }\mu\text{g/g}$  waste ( $6.27 \times 10^{-4}\text{ }\mu\text{Ci/g}$ ).

The concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  measured by ICP-MS are listed in Table 3.5. The analysis results indicate that concentrations of these radionuclides in tank C-108 residual waste are relatively low, with averages of  $1.24 \times 10^{-4}\text{ }\mu\text{Ci/g}$  ( $^{237}\text{Np}$ ),  $2.90 \times 10^{-2}\text{ }\mu\text{Ci/g}$  ( $^{239}\text{Pu}$ ), and not detected ( $^{241}\text{Am}$ ).

The concentrations of  $^{137}\text{Cs}$ , and  $^{90}\text{Sr}$  determined by GEA analysis ( $^{137}\text{Cs}$ ) and LSC analysis following radiochemical separation ( $^{90}\text{Sr}$ ) are listed in Table 3.6. The analysis results indicate that significant concentrations of these radionuclides occur in tank C-108 residual waste, with averages of  $16.9\text{ }\mu\text{Ci/g}$  ( $^{137}\text{Cs}$ ), and  $122\text{ }\mu\text{Ci/g}$  ( $^{90}\text{Sr}$ ).

**Table 3.2.** Residual Waste Composition Determined by EPA Acid Digestion and ICP-OES Analysis

Sample Number	Al	As	B	Ba	Be	Bi	Ca	Cd	Co	Cr
µg/g Dry Waste										
EPA Acid Digestion										
20578	1.41E+05	ND	NA	(1.16E+01)	(2.76E-01)	(1.98E+02)	(2.15E+02)	ND	ND	(2.40E+01)
20578 Dup1	1.05E+05	ND	NA	(9.35E+00)	(1.62E-01)	(8.11E+01)	(1.96E+02)	ND	ND	(3.04E+01)
20578 Average	1.23E+05	ND	NA	(1.05E+01)	(2.19E-01)	(1.40E+02)	(2.05E+02)	ND	ND	(2.72E+01)

Sample Number	Cu	Fe	K	Li	Mg	Mn	Mo	Ni	P	Pb
µg/g Dry Waste										
EPA Acid Digestion										
20578	(2.42E+01)	4.56E+03	(1.14E+02)	(7.19E+00)	(1.57E+01)	(2.73E+01)	ND	2.20E+03	4.03E+04	6.72E+02
20578 Dup1	(2.37E+01)	2.75E+04	ND	(1.31E+01)	(1.95E+00)	1.26E+02	ND	2.26E+03	3.93E+04	7.90E+02
20578 Avg	(2.40E+01)	1.60E+04	(1.14E+02)	(1.01E+01)	(8.82E+00)	1.26E+02	ND	2.23E+03	3.98E+04	7.31E+02

Sample Number	Se	Sr	V	Zn	Na	S	Ti	Zr	U
µg/g Dry Waste									
EPA Acid Digestion									
20578	ND	7.46E+02	ND	(7.85E+01)	1.61E+05	(7.78E+02)	(1.55E+01)	(1.76E+01)	1.66E+03
20578 Dup1	ND	5.59E+02	ND	(1.02E+02)	1.48E+05	(9.30E+02)	(6.88E+00)	(1.59E+01)	1.88E+03
20578 Avg	ND	6.53E+02	ND	(9.03E+01)	1.55E+05	(8.54E+02)	(1.12E+01)	(1.68E+01)	1.77E+03

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantification limit.

NA = Not analyzed.

ND = Not detected.

**Table 3.3.** Residual Waste Composition Determined by EPA Acid Digestion and ICP-MS Analysis

Sample Number	Cr – Total Based on		As – Total Based on	Se – Total Based on	Mo – Total Based on		
	<sup>52</sup> Cr	Cu - Total <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se	<sup>95</sup> Mo	<sup>97</sup> Mo	<sup>98</sup> Mo <sup>(b)</sup>
µg/g Dry Waste							
EPA Microwave Digestion							
20578	2.87E+01	(2.12E+01)	(8.41E-01)	ND	(7.07E+00)	(9.63E+00)	(4.19E+00)
20578 Dup1	3.30E+01	(2.30E+01)	(2.19E+00)	ND	(6.75E+00)	(1.06E+01)	(4.65E+00)
20578 Average	3.09E+01	(2.21E+01)	(1.52E+00)	ND	(6.91E+00)	(1.01E+01)	(4.42E+00)

(a) Cu results are based on the average of <sup>63</sup>Cu and <sup>65</sup>Cu.  
(b) The indicated isotope is the suggested isotope for use to quantify the total concentration of that element.

Sample Number	Ru – Total Based on Sum of		Cd – Total <sup>(c)</sup>	Sb – Total Based on	Pb – Total <sup>(d)</sup>
	<sup>101</sup> Ru	<sup>102</sup> Ru		<sup>121</sup> Sb	
	µg/g Dry Waste				
EPA Microwave Digestion					
20578	2.13E+01	9.94E+00	2.45E+00	(1.55E+00)	7.19E+02
20578 Dup1	1.93E+01	9.60E+00	2.77E+00	(9.01E-01)	8.39E+02
20578 Average	2.03E+01	9.77E+00	2.61E+00	(1.23E+00)	7.79E+02

(c) Cd results are based on the average of <sup>111</sup>Cd and <sup>114</sup>Cd.  
(d) Pb results are based on the average of <sup>206</sup>Pb and <sup>208</sup>Pb.  
Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

**Table 3.4.** Concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion and ICP-MS Analysis

Sample Number	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	μg/g Dry Waste		μCi/g Dry Waste	
EPA Acid Digestion				
20578	5.94E-01	1.79E+03	1.01E-02	6.01E-04
20578 Dup1	6.78E-01	1.94E+03	1.15E-02	6.52E-04
20578 Average	6.36E-01	1.87E+03	1.08E-02	6.27E-04
Dup = Duplicate.				

**Table 3.5.** Concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion and ICP-MS Analysis

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
<b>EPA Microwave Digestion</b>						
20578	1.19E-04	1.67E-01	2.76E-02	4.46E-01	ND	ND
20578 Dup1	1.29E-04	1.82E-01	3.03E-02	4.88E-01	ND	ND
20578 Average	1.24E-04	1.75E-01	2.90E-02	4.67E-01	ND	ND
Dup = Duplicate.						
ND = Instrument returned a zero or negative response.						

**Table 3.6.** Concentrations of  $^{137}\text{Cs}$ , and  $^{90}\text{Sr}$  Measured in Tank C-108 Residual Waste Determined by EPA Acid Digestion

Sample Number	<sup>137</sup> Cs	<sup>90</sup> Sr	<sup>137</sup> Cs	<sup>90</sup> Sr
	μg/g Dry Waste		μCi/g Dry Waste	
EPA Acid Digestion				
20578	0.148	1.000	12.9	140
20578 Dup1	0.240	0.736	20.9	103
20578 Average	0.194	0.868	16.9	122
Dup = Duplicate.				

### 3.1.2 Fusion Results

Composition results for tank C-108 partial retrieval residual waste (sample 20578) determined using the fusion dissolution method are presented in this section. Results are reported for the primary sample, two duplicates, and the average of the three. The results of elemental analyses determined by ICP-OES analysis are listed in Table 3.7. These results are quite comparable to those determined by the EPA acid digestion method (Table 3.2).

**Table 3.7.** Residual Waste Composition Determined by Fusion Dissolution and ICP-OES Analysis

Sample Number	Al	Bi	Ca	Cr	Fe	P	Sr
µg/g Dry Waste							
Fusion Digestion							
20875	1.26E+05	(2.95E+01)	(2.57E+02)	(2.30E+01)	3.59E+03	7.10E+04	8.08E+02
20875 Dup 1	2.21E+05	(2.16E+02)	(3.54E+02)	(2.92E+01)	8.99E+03	3.50E+04	7.72E+02
20875 Dup 2	1.46E+05	(1.39E+02)	(8.45E+01)	(3.49E+01)	5.53E+03	3.54E+04	7.50E+02
20875 Avg	1.64E+05	(1.28E+02)	(2.32E+02)	(2.90E+01)	6.04E+03	4.71E+04	7.77E+02

Concentrations listed in parentheses were <EQL.

Na, K, S were all used in the fusion preparation; data are not reported for these elements.

Dup = Duplicate.

EQL = Estimated quantitation limit.

Elemental concentrations analyzed by ICP-MS are listed in Table 3.8. For most of the elements, the fusion dissolution ICP-MS results are in reasonably good agreement with the EPA acid digestion ICP-MS results; notable exceptions are As and Sb. Because nearly all of the analysis results for these elements were below the quantification limit, the differences are not considered to be significant.

**Table 3.8.** Residual Waste Composition Determined by Fusion Dissolution and ICP-MS Analysis

Sample Number	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo
µg/g Dry Waste								
Fusion Digestion								
20578	2.34E+01	(2.44E+01)	(1.78E+01)	(1.13E+01)	8.64E+00	1.07E+01	8.86E+00	(4.51E+00)
20578 Dup1	3.30E+01	(3.44E+01)	(1.91E+01)	(1.64E+01)	(5.44E+00)	1.11E+01	1.22E+01	(6.17E+00)
20578 Dup2	4.93E+01	(5.04E+01)	(2.32E+01)	(2.10E+01)	(6.22E+00)	1.61E+01	1.79E+01	(7.93E+00)
20578 Avg	3.52E+01	(3.64E+01)	(2.00E+01)	(1.62E+01)	8.64E+00	1.26E+01	1.30E+01	(6.20E+00)

Sample Number	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
µg/g Dry Waste								
Fusion Digestion								
20578	1.37E+01	6.66E+00	8.49E+00	2.81E+00	1.65E+00	(2.29E-01)	3.32E+02	3.29E+02
20578 Dup1	1.99E+01	9.94E+00	1.17E+01	3.46E+00	2.33E+00	(2.98E-01)	4.64E+02	4.64E+02
20578 Dup2	3.20E+01	1.54E+01	1.67E+01	4.72E+00	2.80E+00	(2.71E-01)	6.07E+02	6.12E+02
20578 Avg	2.19E+01	1.07E+01	1.23E+01	3.66E+00	2.26E+00	(2.66E-01)	4.68E+02	4.68E+02

(a) ICP-MS mass of choice.

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

Concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  determined by fusion dissolution and ICP-MS analysis are listed in Table 3.9. The average  $^{99}\text{Tc}$  concentration was determined to be  $0.542\text{ }\mu\text{g/g}$  waste ( $0.00922\text{ }\mu\text{Ci/g}$ ). The average  $^{238}\text{U}$  concentration was determined to be  $1430\text{ }\mu\text{g/g}$  waste ( $4.80 \times 10^{-4}\text{ }\mu\text{Ci/g}$ ). These values are quite similar to those determined by EPA acid digestion and ICP-MS analysis.

**Table 3.9.** Concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution and ICP-MS Analysis

Contact Stage	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/g}$ Dry Waste		$\mu\text{Ci/g}$ Dry Waste	
<b>Fusion Digestion</b>				
20578	3.35E-01	1.06E+03	5.70E-03	3.56E-04
20578 Dup1	5.20E-01	1.41E+03	8.84E-03	4.74E-04
20578 Dup2	7.72E-01	1.82E+03	1.31E-02	6.12E-04
20578 Average	5.42E-01	1.43E+03	9.22E-03	4.80E-04
Dup = Duplicate.				

Concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  determined by fusion dissolution and ICP-MS analysis are listed in Table 3.10. The analysis results indicate that concentrations of these radionuclides in tank C-108 residual waste are relatively low, with averages of  $1.31 \times 10^{-4}\text{ }\mu\text{Ci/g}$  ( $^{237}\text{Np}$ ),  $2.30 \times 10^{-2}\text{ }\mu\text{Ci/g}$  ( $^{239}\text{Pu}$ ), and  $1.43 \times 10^{-1}\text{ }\mu\text{Ci/g}$  ( $^{241}\text{Am}$ ). These values are very comparable with those determined by EPA acid digestion and ICP-MS analysis.

**Table 3.10.** Concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution and ICP-MS Analysis

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g}$ Dry Waste	$\mu\text{g/g}$ Dry Waste	$\mu\text{Ci/g}$ Dry Waste	$\mu\text{g/g}$ Dry Waste	$\mu\text{Ci/g}$ Dry Waste	$\mu\text{g/g}$ Dry Waste
<b>Fusion Digestion</b>						
20578	9.80E-05	1.38E-01	1.84E-02	2.97E-01	(1.40E-01)	(4.13E-02)
20578 Dup1	1.33E-04	1.88E-01	2.41E-02	3.89E-01	(1.24E-01)	(3.65E-02)
20578 Dup2	1.61E-04	2.27E-01	2.66E-02	4.29E-01	(1.66E-01)	(4.88E-02)
20578 Average	1.31E-04	1.84E-01	2.30E-02	3.72E-01	(1.43E-01)	(4.22E-02)
Concentrations listed in parentheses were <EQL.						
Dup = Duplicate.						
EQL = Estimated quantitation limit.						

Concentrations of  $^{137}\text{Cs}$ , and  $^{90}\text{Sr}$  determined by fusion dissolution and GEA analysis ( $^{137}\text{Cs}$ ) and LSC analysis following radiochemical separation ( $^{90}\text{Sr}$ ) are listed in Table 3.11. The analysis results indicate that significant concentrations of these radionuclides occur in tank C-108 residual waste, with averages of  $13.7\text{ }\mu\text{Ci/g}$  ( $^{137}\text{Cs}$ ) and  $207\text{ }\mu\text{Ci/g}$  ( $^{90}\text{Sr}$ ). These values are similar to those determined by EPA acid digestion.

**Table 3.11.** Concentrations of  $^{137}\text{Cs}$ , and  $^{90}\text{Sr}$  Measured in Tank C-108 Residual Waste Determined by Fusion Dissolution

	<sup>137</sup> Cs	<sup>90</sup> Sr	<sup>137</sup> Cs	<sup>90</sup> Sr
Contact Stage	μg/g Dry Waste		μCi/g Dry Waste	
Fusion Digestion				
20578	0.194	1.07	16.9	150
20578 Dup1	0.143	2.11	12.4	296
20578 Dup2	0.134	1.25	11.7	175
20578 Average	0.157	1.48	13.7	207
Dup = Duplicate.				

### 3.1.3 Comparison with Results Reported by the 222-S Laboratory

Results determined in this study are compared with those of the 222-S Laboratory for a number of the most important constituents to evaluate consistency in Table 3.12. When possible (depending on analyte), the same digestion methods were used for the comparison. In general, the agreement between the results for all analytes determined by both laboratories is quite good. Minor differences that are observed could easily result from sample heterogeneity.

**Table 3.12.** Comparison of PNNL Results with Those Determined by the 222-S Laboratory. Sample S09R000138 is a composite of S09T005880, S09T005881, and S09T005882.

Analyte	Extraction Method	PNNL (S09R000138)	222-S A (S09T005880)	222-S B (S09T005881)	222-S C (S09T005882)
Al ( $\mu\text{g/g}$ )	fusion	1.26E+05	9.19E+04	1.30E+05	1.02E+05
Cr ( $\mu\text{g/g}$ )	fusion	(29.0)	<105	<104	<103
$^{137}\text{Cs}$ ( $\mu\text{Ci/g}$ )	fusion	13.7	20.2	7.68	4.80
Fe ( $\mu\text{g/g}$ )	fusion	6.04E+03	1.82E+04	3.91E+03	3.39E+03
Mn ( $\mu\text{g/g}$ )	fusion	ND	91.8	<62.3	<61.7
Na ( $\mu\text{g/g}$ )	fusion	1.55E+05 <sup>(a)</sup>	1.30E+05	1.23E+05	1.29E+05
Ni ( $\mu\text{g/g}$ )	fusion	2.33E+03 <sup>(a)</sup>	2.13E+03	1.63E+03	596
$^{237}\text{Np}$ ( $\mu\text{g/g}$ )	acid extn	0.175	<0.101	<0.102	0.119
P ( $\mu\text{g/g}$ )	fusion	4.71E+04	2.77E+04	3.32E+04	4.14E+04
$^{239}\text{Pu}$ ( $\mu\text{g/g}$ )	acid extn	0.467	<1.31	<1.33	<1.29
$^{90}\text{Sr}$ ( $\mu\text{Ci/g}$ )	fusion	207	195	82.5	171
$^{99}\text{Tc}$ ( $\mu\text{g/g}$ )	acid extn	0.636	0.589	0.393	0.237
$^{238}\text{U}$ ( $\mu\text{g/g}$ )	acid extn	1.87E+03	1.02E+03	1.13E+03	1.62E+03

Concentrations listed in parentheses were <EQL.

(a) Acid extn.

ND = Not detected.

EQL = Estimated quantitation limit.

### 3.1.4 Carbon Analysis Results

Carbon analysis results are presented in Table 3.13. Results of the carbon analysis indicate that measureable carbon in the tank C-108 residual waste occurs in the form of inorganic carbon. The average TIC concentration in tank C-108 residual waste was  $6.48 \times 10^3 \mu\text{g C/g}$ .

**Table 3.13.** Carbon Analysis Results of Tank C-108 Residual Waste Samples

Sample Number	TC	TOC	TIC	TIC
	$\mu\text{g C/g Dry Waste}$		$\mu\text{g CO}_3/\text{g}$	
20578	8.05E+03	<440	8.05E+03	4.02E+04
20578 Dup1	4.59E+03	<174	4.59E+03	2.29E+04
20578 Dup2	6.81E+03	<343	6.81E+03	3.41E+04
20578 Avg	6.48E+03		6.48E+03	3.24E+04
Avg = Average.				
Dup = Duplicate.				
TC = Total carbon.				
TIC = Total inorganic carbon.				
TOC = Total organic carbon.				
$\mu\text{g C/g}$ = Microgram of carbon per gram of dry residual waste.				
$\mu\text{g CO}_3/\text{g}$ = Microgram of $\text{CO}_3$ (carbonate) per gram of dry residual waste.				
< = Sample was less than the method detection limit.				

## 3.2 Double-Deionized Water-Leach Tests

Data obtained from DDI water-leach tests on residual waste sample 20578 are presented in this section. Tests were conducted on three replicate subsamples indicated as 20578, 20578 Dup1, and 20578 Dup2. The single-contact, water-leach tests were run with an equilibration time of 1 month. Periodic replenishment tests were conducted with a contact period of 7 days for a total of five replenishments. The concentrations of the constituents in the water extracts are tabulated in this section in terms of leachable solids concentration units ( $\mu\text{Ci/g-dry residual waste}$  or  $\mu\text{g/g-dry residual waste}$ ). Results for the three replicates are provided in the tables for the single-contact leach tests. Liquid phase concentration units ( $\mu\text{Ci/L}$  or  $\mu\text{g/L}$ ) are provided in Appendix A.

### 3.2.1 1-Month Single-Contact Double-Deionized Water-Leach Tests

#### 3.2.1.1 Residual Waste to Double-Deionized Water Ratios: Single-Contact Leach Tests

Dry residual waste concentrations used in single-contact DDI water leach tests are shown in (Table 3.14). These values were calculated from the moist weight of the added waste and moisture content of the residual waste (Table 3.1). The waste concentrations ranged from 7.40 to 8.70 g/L.



**Table 3.14.** Residual Waste to Double-Deionized Water Ratios Used in Single-Contact Leach Tests

Sample Number	Dry Waste to DDI Water Ratio (g/L)
20578	7.40
20578 Dup1	8.20
20578 Dup2	8.70
Dup = Duplicate.	

### 3.2.1.2 pH and Alkalinity – Single-Contact Double-Deionized Water Leachants

Alkalinities and pH values measured in replicate samples of 1-month DDI water leachates of tank C-108 residual waste are listed in Table 3.15. The pH values for the three replicates were very similar with an average of 12.7. The total alkalinity for the three replicates was also very similar with an average of  $7.52 \times 10^6$  µg/L Alkalinity (as CaCO<sub>3</sub>).

**Table 3.15.** The pH and Alkalinity Values for Single-Contact Double-Deionized Water Leachates

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g dry waste
20578	12.77	7.45E+06	1.00E+06
20578 Dup1	12.75	7.75E+06	9.42E+05
20578 Dup2	12.69	7.35E+06	8.43E+05
20578 Average	12.74	7.52E+06	9.28E+05
Dup = Duplicate.			

### 3.2.1.3 <sup>99</sup>Tc and <sup>238</sup>U – Single-Contact Double-Deionized Water Leachants

Dissolved concentrations of <sup>99</sup>Tc and <sup>238</sup>U determined in replicate 1-month DDI water leachates of tank C-108 residual waste are shown in Table 3.16. The average leachable <sup>99</sup>Tc concentration was 0.506 µg/g (0.00860 µCi/g) and the average leachable <sup>238</sup>U concentration was 825 µg/g ( $2.77 \times 10^{-4}$  µCi/g). These values equate to an average of 80% (<sup>99</sup>Tc) and 44% (<sup>238</sup>U) compared to the totals determined by EPA acid digestion (Table 3.4).

**Table 3.16.** Leachable <sup>99</sup>Tc and <sup>238</sup>U Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	µg/g Dry Waste		µCi/g Dry Waste	
20578	3.95E-01	7.07E+02	6.72E-03	2.38E-04
20578 Dup 1	5.19E-01	1.05E+03	8.82E-03	3.53E-04
20578 Dup 2	6.04E-01	7.19E+02	1.03E-02	2.42E-04
20578 Average	5.06E-01	8.25E+02	8.60E-03	2.77E-04
Dup = Duplicate.				

### 3.2.1.4 <sup>137</sup>Cs and <sup>90</sup>Sr – Single-Contact Double-Deionized Water Leachants

Dissolved concentrations of <sup>137</sup>Cs and <sup>90</sup>Sr determined in replicate 1-month DDI water leachates of tank C-108 residual waste are shown in Table 3.17. The average leachable <sup>137</sup>Cs concentration was 6.66 µCi/g ( $7.66 \times 10^{-2}$  µg/g) and the average leachable <sup>90</sup>Sr concentration was 4.93 µCi/g ( $3.52 \times 10^{-2}$  µg/g). These values equate to an average of 39% (<sup>137</sup>Cs) and 4.0% (<sup>90</sup>Sr) compared to the totals determined by EPA acid digestion (Table 3.4).

**Table 3.17.** Leachable <sup>137</sup>Cs and <sup>90</sup>Sr Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	<sup>137</sup> Cs	<sup>90</sup> Sr	<sup>137</sup> Cs	<sup>90</sup> Sr
	µg/g Dry Waste		µCi/g Dry Waste	
20578	1.06E-01	4.04E-02	9.20	5.66
20578 Dup 1	5.98E-02	4.13E-02	5.20	5.78
20578 Dup 2	6.43E-02	2.39E-02	5.59	3.35
20578 Average	7.66E-02	3.52E-02	6.66	4.93
Dup = Duplicate.				

### 3.2.1.5 Transuranics – Single-Contact Double-Deionized Water Leachants

Dissolved concentrations of <sup>237</sup>Np, <sup>239</sup>Pu, and <sup>241</sup>Am determined in the tank C-108 1-month DDI water leachates are shown in Table 3.18. The average leachable concentrations for <sup>237</sup>Np, <sup>239</sup>Pu, and <sup>241</sup>Am were  $4.01 \times 10^{-5}$  µCi/g,  $9.57 \times 10^{-3}$  µCi/g, and ( $1.46 \times 10^{-2}$  µCi/g), respectively. These values equate to an average percent leachable of 31% (<sup>237</sup>Np), 42% (<sup>239</sup>Pu), and (~10%) (<sup>241</sup>Am) compared to totals determined by fusion digestion (Table 3.10).

**Table 3.18.** Leachable <sup>237</sup>Np, <sup>239</sup>Pu, and <sup>241</sup>Am Concentrations in Waste Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	<sup>237</sup> Np		<sup>239</sup> Pu		<sup>241</sup> Am	
	µCi/g Dry Waste	µg/g Dry Waste	µCi/g Dry Waste	µg/g Dry Waste	µCi/g Dry Waste	µg/g Dry Waste
20578	3.24E-05	4.57E-02	7.75E-03	1.25E-01	(1.46E-02)	(4.29E-03)
20578 Dup1	5.43E-05	7.65E-02	1.28E-02	2.06E-01	(1.53E-02)	(4.49E-03)
20578 Dup2	3.37E-05	4.74E-02	8.18E-03	1.32E-01	(1.38E-02)	(4.06E-03)
20578 Average	4.01E-05	5.65E-02	9.57E-03	1.54E-01	(1.46E-02)	(4.28E-03)
Concentrations listed in parentheses were <EQL.						
EQL = Estimated quantitation limit.						
Dup = Duplicate.						

### 3.2.1.6 Metals – Single-Contact Double-Deionized Water Leachants

Concentrations of a number of metals, such as Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Si, Sr, Ti, Tl, V, Zn, and Zr, in the DDI water leachates were measured by ICP-OES. Among these, only about a dozen metals were present in measurable concentrations (Table 3.19). The concentrations listed within parentheses indicate values that are less than EQL. The most significant DDI water leachable elements in the tank C-108 residual waste samples were Na and P. The average water leachable concentration of Na was  $1.68 \times 10^5$  µg/g waste. The average water leachable concentration of P was  $5.06 \times 10^4$  µg/g waste. Significant concentrations of U (919 µg/g), Al (772 µg/g), S (643 µg/g), and Si (299 µg/g) were also leachable from these samples. Other elements with relatively low measurable leachability included Cr and Fe.

**Table 3.19.** Leachable Metal Concentrations in Waste Determined by ICP-OES in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	Al	Cr	Fe	P	Na	Si
Sample Number	µg/g Dry Waste					
20578	8.50E+02	(1.58E+01)	4.17E+01	6.01E+04	1.84E+05	4.08E+02
20578 Dup1	6.88E+02	2.01E+01	3.92E+01	4.68E+04	1.64E+05	2.31E+02
20578 Dup2	7.79E+02	2.42E+01	2.74E+01	4.49E+04	1.56E+05	2.58E+02
20578 Avg	7.72E+02	2.22E+01	3.61E+01	5.06E+04	1.68E+05	2.99E+02

	S	U	Ca	K	Ni	Sr
Sample Number	µg/g Dry Waste					
20578	5.65E+02	7.25E+02	(3.23E+01)	(1.90E+02)	(1.20E+00)	(1.59E+00)
20578 Dup1	6.48E+02	1.27E+03	(5.43E+01)	(2.20E+02)	(3.50E+00)	(1.99E+00)
20578 Dup2	7.16E+02	7.61E+02	(1.79E+01)	(2.30E+02)	(1.04E+01)	(2.27E+00)
20578 Avg	6.43E+02	9.19E+02	(3.48E+01)	(2.13E+02)	(5.03E+00)	(1.95E+00)

Concentrations listed in parentheses were <EQL.  
Avg = Average.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.

Concentrations of a number of trace metals determined by ICP-MS in the DDI water leachates are shown in Table 3.20. The analysis included the metals Cr, Cu, As, Se, Mo, Ru, Ag, Cd, Sb, and Pb. Of these, only Cr and Pb were present at concentrations above the quantification limit. The leachable concentrations determined for Cr and Pb were 19.5 µg/g and 4.88 µg/g.

**Table 3.20.** Leachable Metal Concentrations in Waste Determined by ICP-MS in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/g Dry Waste					
20578	1.74E+01	1.84E+01	(8.32E+00)	ND	(7.37E+00)	ND
20578 Dup1	1.85E+01	1.74E+01	(7.04E+00)	ND	(4.11E+00)	ND
20578 Dup2	2.25E+01	2.22E+01	(5.64E+00)	ND	(5.47E+00)	ND
20578 Avg	1.95E+01	1.93E+01	(7.00E+00)	ND	(5.65E+00)	ND

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/g Dry Waste					
20578	(3.66E+00)	(6.89E+00)	(2.64E+00)	1.42E+01	6.50E+00	6.13E+00
20578 Dup1	(4.54E+00)	(6.40E+00)	(2.40E+00)	1.80E+01	8.23E+00	7.66E+00
20578 Dup2	(5.53E+00)	7.37E+00	(3.06E+00)	1.92E+01	9.09E+00	7.98E+00
20578 Avg	(4.58E+00)	7.37E+00	(2.70E+00)	1.71E+01	7.94E+00	7.26E+00

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Sample Number	µg/g Dry Waste						
20578	(1.49E+00)	(1.57E+00)	ND	ND	ND	4.09E+00	4.10E+00
20578 Dup1	(1.08E+00)	(1.13E+00)	ND	ND	ND	6.10E+00	7.03E+00
20578 Dup2	(1.05E+00)	(1.08E+00)	ND	ND	ND	(2.84E+00)	3.50E+00
20578 Avg	(1.21E+00)	(1.26E+00)	ND	ND	ND	5.10E+00	4.88E+00

(a) ICP-MS mass of choice.  
Concentrations listed in parentheses were <EQL.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.2.1.7 Anions – Single-Contact Double-Deionized Water Leachants

Leachable concentrations of anions determined from the 1-month contact DDI water leachates measured by IC are shown in Table 3.21. The anion with the highest leachable concentration was phosphate ( $1.57 \times 10^5$  µg/g), followed by fluoride ( $1.62 \times 10^4$  µg/g), then nitrite ( $1.55 \times 10^4$  µg/g), and then nitrate ( $1.31 \times 10^4$  µg/g). The concentrations of sulfate and chloride were below the quantification limit.

**Table 3.21.** Leachable Anion Concentrations Determined from Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste

Sample Number	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	µg/g Dry Waste					
20578	1.95E+04	(7.81E+02)	(1.25E+04)	(9.04E+03)	(2.11E+03)	1.89E+05
20578 Dup1	1.47E+04	(8.87E+02)	1.45E+04	1.22E+04	(2.53E+03)	1.42E+05
20578 Dup2	1.45E+04	(7.56E+02)	1.65E+04	1.40E+04	(2.43E+03)	1.41E+05
20578 Avg	1.62E+04	(8.08E+02)	1.55E+04	1.31E+04	(2.36E+03)	1.57E+05

Concentrations listed in parentheses were <EQL.  
 Avg = Average.  
 Dup = Duplicate.  
 EQL = Estimated quantitation limit.

### 3.2.2 Periodic Replenishment Double-Deionized Water-Leach Tests

#### 3.2.2.1 Residual Waste to Double-Deionized Water Ratios: Periodic Replenishment Leach Tests

Dry residual waste concentrations used in periodic replenishment DDI water leach tests are shown in (Table 3.22). These values were calculated from the moist weight of the added waste and moisture content of the waste (Table 3.1). The waste concentrations ranged from 7.36 to 10.76 g/L.

**Table 3.22.** Residual Waste to Double-Deionized Water Ratios Used in the Periodic Replenishment Leach Tests

Sample Number	Dry Waste to DDI Water Ratio (g/L)
20578	7.36
20578 Dup1	7.81
20578 Dup2	10.76

Dup = Duplicate.

#### 3.2.2.2 pH and Alkalinity – Periodic Replenishment Double-Deionized Water Leachants

The average alkalinities and pH values measured in tank C-108 residual waste periodic replenishment DDI water leachates are listed in Table 3.23. The pH values decreased from a high of 11.5 in the first contacting solution to 6.4 in the fifth contacting solution. The total alkalinity decreased from a high of  $9.30 \times 10^6$  µg/L (as CaCO<sub>3</sub>) to a value that was below the quantification limit in the fifth contact ( $9.34 \times 10^3$  µg/L).

**Table 3.23.** Average pH and Alkalinity Values for Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g Dry Waste
1	11.5	9.30E+06	1.08E+06
2	8.0	2.40E+05	2.60E+04
3	7.4	8.10E+04	9.19E+03
4	7.6	4.81E+04	5.68E+03
5	6.4	(9.34E+03)	(4.21E+02)

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.

### 3.2.2.3 <sup>99</sup>Tc and <sup>238</sup>U – Periodic Replenishment Double-Deionized Water Leachants

Average concentrations of <sup>99</sup>Tc and <sup>238</sup>U measured in the periodic replenishment DDI water leachates are listed in Table 3.24. The vast majority of the leachable <sup>99</sup>Tc and <sup>238</sup>U in the tank C-108 residual waste was solubilized during the first contact stage. After the first contact, <sup>99</sup>Tc decreased to concentrations that were less than the quantification limit. The concentration of <sup>238</sup>U in the second stage decreased to less than 1% of the concentration in stage 1. During subsequent stages, the <sup>238</sup>U concentrations continued to decrease.

**Table 3.24.** Average Leachable <sup>99</sup>Tc and <sup>238</sup>U Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	µg/g Dry Waste		µCi/g Dry Waste	
1	3.29E-01	6.99E+02	5.59E-03	2.35E-04
2	(1.70E-02)	3.99E+00	(2.89E-04)	1.34E-06
3	(1.86E-03)	1.45E+00	(3.17E-05)	4.86E-07
4	ND	5.52E-01	ND	1.85E-07
5	(4.53E-02)	2.40E-01	(7.70E-04)	8.06E-08

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.2.2.4 <sup>137</sup>Cs and <sup>90</sup>Sr – Periodic Replenishment Double-Deionized Water Leachants

Average concentrations of <sup>137</sup>Cs and <sup>90</sup>Sr measured in the periodic replenishment DDI water leachates are listed in Table 3.25. The vast majority of the leachable <sup>137</sup>Cs in the tank C-108 residual waste was solubilized during the first contact stage. Although variable, <sup>90</sup>Sr appears to leach out at relatively constant concentrations for all contact stages.

**Table 3.25.** Average Leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
1	6.01E-02	6.62E-02	5.23	9.27
2	ND	1.87E-01	ND	26.2
3	ND	2.18E-01	ND	30.5
4	(1.46E-02)	1.44E-01	(1.27)	20.1
5	ND	7.86E-02	ND	11.0

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.2.2.5 Transuranics – Periodic Replenishment Double-Deionized Water Leachants

Average concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  measured in the periodic replenishment DDI water leachates are listed in Table 3.26. The highest concentrations of leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  that occurred in the tank C-108 residual waste was solubilized during the first contact stage. During subsequent stages, the concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  significantly decreased. Concentrations in subsequent stages decreased to concentrations that were nearly an order of magnitude less than those in the first stage or less than the quantification or detection limits.

**Table 3.26.** Average Leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
1	3.16E-05	4.45E-02	7.61E-03	1.23E-01	1.94E-02	5.69E-03
2	(1.04E-08)	(1.47E-05)	(4.50E-05)	(7.25E-04)	ND	ND
3	(1.21E-06)	(1.71E-03)	(1.14E-04)	(1.84E-03)	ND	ND
4	4.87E-06	6.87E-03	4.07E-04	6.57E-03	ND	ND
5	1.88E-06	2.65E-03	(1.01E-04)	(1.64E-03)	ND	ND

Values within parentheses were <EQL.  
EQL = Estimated quantification limit.  
ND = Instrument returned a zero or negative response.

### 3.2.2.6 Metals – Periodic Replenishment Double-Deionized Water Leachants

Average concentrations of elements measured by ICP-OES in the periodic replenishment DDI water leachates are listed in Table 3.27. The highest concentrations of leachable elements measured by ICP-OES that were solubilized from tank C-108 residual waste occurred during the first contact stage, with the exception of Sr and Si. During subsequent stages, the concentrations of Cr, P, Na, S, and U

decreased significantly or to levels that were below the quantification limit or not detectable. Decreases in Al, Fe, Sr, and Si were much less dramatic during subsequent stages.

**Table 3.27.** Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	Al	Cr	Fe	P	Sr	Na	Si	S	U
µg/g Dry Waste									
1	4.57E+02	1.42E+01	3.84E+01	6.85E+04	(8.38E-01)	2.00E+05	1.19E+02	5.45E+02	7.51E+02
2	1.26E+02	ND	1.90E+01	8.78E+02	1.78E+02	2.69E+03	1.46E+02	(2.29E+01)	(8.87E+00)
3	7.84E+01	ND	2.25E+01	1.58E+02	1.58E+02	4.21E+02	(5.94E+01)	(2.25E+01)	(6.24E+00)
4	2.19E+01	ND	(2.91E+00)	(2.37E+01)	8.79E+01	(8.14E+01)	(2.88E+01)	(2.15E+01)	(3.22E+00)
5	(1.95E+00)	(2.83E-01)	(5.37E-01)	(9.42E+00)	1.26E+02	(2.86E+01)	(1.55E+01)	(2.61E+00)	(5.24E+00)

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

Average concentrations of elements measured by ICP-MS in the periodic replenishment DDI water leachates are listed in Table 3.28. The highest concentrations of leachable trace metals measured by ICP-MS that were solubilized from tank C-108 residual waste occurred during the first contact stage. During subsequent stages, the concentrations of all the metals that were quantified—except Pb—decreased significantly or to levels that were below the quantification limit or not detectable. Decreases in Pb concentrations were much less dramatic during subsequent stages, except in the last stage where a large decrease in concentration occurred.

**Table 3.28.** Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste

Contact Stage	Cr <sup>(a)</sup>	Cu - Total <sup>(b)</sup>	As – Total Based on	Se – Total Based on	Mo – Total Based on		
			<sup>75</sup> As	<sup>82</sup> Se	<sup>95</sup> Mo	<sup>97</sup> Mo	<sup>98</sup> Mo <sup>(c)</sup>
			µg/g Dry Waste				
1	1.02E+01	7.11E+00	3.86E+00	8.03E-02	2.81E+00	3.63E+00	1.32E+00
2	5.10E-01	(1.96E-01)	ND	ND	(1.12E-01)	(1.39E-01)	(1.13E-01)
3	(8.51E-02)	(1.63E-01)	ND	ND	(5.31E-03)	(2.06E-02)	(2.95E-02)
4	(8.54E-02)	(5.60E-02)	ND	ND	(6.27E-03)	(1.52E-02)	(2.54E-02)
5	(2.43E-02)	(1.45E-02)	ND	<sup>(d)</sup>	(1.36E-02)	(2.56E-02)	(3.41E-02)

(a) Cr results are based on the average of <sup>52</sup>Cr and <sup>53</sup>Cr.

(b) Cu results are based on the average of <sup>63</sup>Cu and <sup>65</sup>Cu.

(c) The indicated isotope is the suggested isotope for use to quantify the total concentration of that element.

(d) Se results not reported for last replenishment due to QC failure.



**Table 3.28. (contd)**

Contact Stage	Ru – Total Based on Sum of		Ag <sup>(e)</sup>	Cd – Total <sup>(f)</sup>	Sb – Total Based on <sup>121</sup> Sb	Pb – Total <sup>(g)</sup>
	<sup>101</sup> Ru	<sup>102</sup> Ru				
	μg/g Dry Waste					
1	5.70E+00	2.55E+00	(1.26E-01)	2.08E-01	1.11E-01	2.80E+00
2	2.89E-01	1.28E-01	(1.85E-02)	8.23E-02	(2.97E-02)	2.10E+00
3	(1.90E-02)	6.42E-02	(2.36E-02)	8.81E-02	(3.17E-02)	2.01E+00
4	(1.26E-02)	6.37E-02	(1.81E-02)	(3.57E-02)	(2.38E-02)	7.24E-01
5	(1.49E-02)	5.90E-02	(1.70E-02)	7.30E-02	(2.55E-02)	(2.56E-02)

(e) Ag results are based on the average of <sup>107</sup>Ag and <sup>109</sup>Ag.

(f) Cd results are based on the average of <sup>111</sup>Cd and <sup>114</sup>Cd.

(g) Pb results are based on the average of <sup>206</sup>Pb and <sup>208</sup>Pb.

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.2.2.7 Anions – Periodic Replenishment Double-Deionized Water Leachants

Average concentrations of anions measured by IC in the periodic replenishment DDI water leachates are listed in Table 3.29. The highest concentrations of leachable anions that were solubilized from tank C-108 residual waste occurred during the first contact stage. During subsequent stages, the concentrations of all the anions that were quantified decreased to levels that were below the quantification limit or not detectable.

**Table 3.29. Average Leachable Anion Concentrations Determined from Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste**

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	μg/g Dry Waste					
1	2.23E+04	(6.92E+02)	1.28E+04	1.03E+04	(1.59E+03)	1.61E+05
2	ND	(6.21E+02)	(2.96E+03)	ND	(8.65E+02)	(1.06E+03)
3	ND	(6.26E+02)	ND	ND	(8.70E+02)	(4.18E+02)
4	ND	(6.44E+02)	ND	ND	(8.14E+02)	ND
5	ND	(1.90E+02)	ND	(1.21E+03)	(1.54E+03)	(6.28E+02)

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.3 Ca(OH)<sub>2</sub> Solution Leach Tests

#### 3.3.1 1-Month Single-Contact Ca(OH)<sub>2</sub> Solution Leach Tests

##### 3.3.1.1 Residual Waste to Ca(OH)<sub>2</sub> Solution Ratios: Single-Contact Leach Tests

Dry residual waste concentrations used in single-contact Ca(OH)<sub>2</sub> solution leach tests are shown in (Table 3.30). These values were calculated from the moist weight of the added waste and moisture content of the residual waste (Table 3.1). The waste concentrations ranged from 6.66 to 10.59 g/L.

**Table 3.30.** Residual Waste to Ca(OH)<sub>2</sub> Solution Ratios Used in Single-Contact Leach Tests

Sample Number	Dry Waste to Ca(OH) <sub>2</sub> Solution Ratio (g/L)
20578	6.66
20578 Dup1	8.84
20578 Dup2	10.59
Dup = Duplicate.	

##### 3.3.1.2 pH and Alkalinity – Single-Contact Ca(OH)<sub>2</sub> Solution Leachants

Alkalinities and pH values measured in replicate samples of 1-month Ca(OH)<sub>2</sub> solution leachates of tank C-108 residual waste are listed in Table 3.31. The pH values for the three replicates were similar with an average of 12.7. The total alkalinity for the three replicates was also similar with an average of  $9.32 \times 10^6$  µg/L alkalinity (as CaCO<sub>3</sub>).

**Table 3.31.** The pH and Alkalinity Values for Single-Contact Ca(OH)<sub>2</sub> Solution Leachates

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		(µg/L)	(µg/g waste)
20578	12.65	8.15E+06	1.22E+06
20578 Dup1	12.66	1.16E+07	1.30E+06
20578 Dup2	12.68	8.25E+06	7.78E+05
20578 Average	12.66	9.32E+06	1.10E+06
Dup = Duplicate.			

##### 3.3.1.3 <sup>99</sup>Tc and <sup>238</sup>U – Single-Contact Ca(OH)<sub>2</sub> Solution Leachants

Dissolved concentrations of <sup>99</sup>Tc and <sup>238</sup>U determined in replicate 1-month Ca(OH)<sub>2</sub> solution leachates of tank C-108 residual waste are shown in Table 3.32. The average leachable <sup>99</sup>Tc concentration is 0.522 µg/g (0.00887 µCi/g) and the average leachable <sup>238</sup>U concentration is 52.1 µg/g ( $1.75 \times 10^{-5}$  µCi/g). These values equate to an average of 82% (<sup>99</sup>Tc) and 2.8% (<sup>238</sup>U) compared to the

totals determined by EPA acid digestion (Table 3.4). Compared to the leachable concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  determined in the 1-month DDI water leachates (Table 3.16), the percent leachable  $^{99}\text{Tc}$  is about the same, whereas the percent leachable  $^{238}\text{U}$  is dramatically lower.

**Table 3.32.** Leachable  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations Determined from Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Sample Number	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
20578	5.50E-01	6.48E+01	9.35E-03	2.18E-05
20578 Dup 1	5.45E-01	6.10E+01	9.27E-03	2.05E-05
20578 Dup 2	4.71E-01	3.06E+01	8.01E-03	1.03E-05
20578 Average	5.22E-01	5.21E+01	8.87E-03	1.75E-05
Dup = Duplicate.				

### 3.3.1.4 $^{137}\text{Cs}$ and $^{90}\text{Sr}$ – Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachants

Dissolved concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  determined in replicate 1-month  $\text{Ca}(\text{OH})_2$  solution leachates of tank C-108 residual waste are shown in Table 3.33. The average leachable  $^{137}\text{Cs}$  concentration was  $6.62 \mu\text{Ci/g}$  ( $7.61 \times 10^{-2} \mu\text{g/g}$ ) and the average leachable  $^{90}\text{Sr}$  concentration was  $55.3 \mu\text{Ci/g}$  ( $0.395 \mu\text{g/g}$ ). These values equate to an average of 39% ( $^{137}\text{Cs}$ ) and 45% ( $^{90}\text{Sr}$ ) compared to the totals determined by EPA acid digestion (Table 3.4).

**Table 3.33.** Leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations Determined from Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Sample Number	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
20578	1.15E-01	1.14E+00	9.97	1.59E+02
20578 Dup 1	4.75E-02	6.94E-03	4.13	9.71E-01
20578 Dup 2	6.63E-02	4.24E-02	5.77	5.94E+00
20578 Average	7.61E-02	3.95E-01	6.62	5.53E+01
Dup = Duplicate.				

### 3.3.1.5 Transuranics – Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachants

Dissolved concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  determined in the tank C-108 1-month  $\text{Ca}(\text{OH})_2$  solution leachates are shown in Table 3.34. The average leachable concentrations for  $^{237}\text{Np}$  and  $^{241}\text{Am}$  were not detectable, and the  $^{239}\text{Pu}$  concentrations were very low and below the quantification limit.

**Table 3.34.** Leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations Determined from Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
20578	ND	ND	(4.36E-04)	(7.04E-03)	ND	ND
20578 Dup1	ND	ND	ND	ND	ND	ND
20578 Dup2	ND	ND	(1.01E-05)	(1.63E-04)	ND	ND
20578 Average	ND	ND	(1.42E-04)	(2.28E-03)	ND	ND

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.3.1.6 Metals – Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachants

Concentrations of a number of metals, such as Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Si, Sr, Ti, Tl, V, Zn, and Zr, in the  $\text{Ca}(\text{OH})_2$  solution leachates were measured by ICP-OES. Among these, only about a dozen metals were present in measurable concentrations (Table 3.35). The concentrations listed within parentheses indicate values that are less than EQL. The most significant  $\text{Ca}(\text{OH})_2$  solution leachable elements in the tank C-108 residual waste samples were Na, P, and Al. High concentrations of Ca were also observed in some replicates; however, because Ca is in the leachant, it is not considered as leachable. The average water leachable concentration of Na was  $1.31 \times 10^5 \mu\text{g/g waste}$ . Both P and Ca concentrations were quite variable among the three replicates. This effect may have been related to the formation of a  $\text{Ca-PO}_4$  precipitate. The average water leachable concentration of Al was  $2.76 \times 10^3 \mu\text{g/g waste}$ .

**Table 3.35.** Leachable Metals Concentrations Determined by ICP-OES in Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	Al	Cr	Fe	P	Na	Si
	$\mu\text{g/g Dry Waste}$					
20578	2.53E+03	2.32E+01	(9.58E+00)	(4.66E+01)	1.39E+05	(5.99E+00)
20578 Dup1	3.75E+03	2.45E+01	4.18E+01	3.85E+03	1.63E+05	4.28E+02
20578 Dup2	2.00E+03	1.96E+01	(8.13E+00)	(2.81E+01)	9.08E+04	(4.57E+01)
20578 Avg	2.76E+03	2.24E+01	4.18E+01	3.85E+03	1.31E+05	4.28E+02

Sample Number	S	U	Ca	K	Ni	Sr
	$\mu\text{g/g Dry Waste}$					
20578	6.37E+02	6.76E+01	2.02E+03	(2.85E+02)	(4.68E+00)	4.63E+01
20578 Dup1	7.78E+02	6.51E+01	(1.20E+01)	(2.36E+02)	(5.48E+00)	ND
20578 Dup2	5.95E+02	3.32E+01	9.16E+02	2.10E+02	(6.82E+00)	(1.04E+01)
20578 Avg	6.70E+02	5.53E+01	1.47E+03	2.10E+02	(5.66E+00)	4.63E+01

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

Concentrations of a number of trace metals determined by ICP-MS in the  $\text{Ca}(\text{OH})_2$  solution leachates are shown in Table 3.36. The analysis included the metals Cr, Cu, As, Se, Mo, Ru, Ag, Cd, Sb, and Pb. Of these, only Cr and Ru were present at concentrations that were consistently above the quantification limit. The leachable concentrations determined for Cr and Ru were 24.1  $\mu\text{g/g}$  and 10.2  $\mu\text{g/g}$ .

**Table 3.36.** Leachable Metals Concentrations Determined by ICP-MS in Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{52}\text{Cr}^{(a)}$	$^{53}\text{Cr}$	$^{63}\text{Cu}$	$^{65}\text{Cu}^{(a)}$	$^{75}\text{As}$	$^{82}\text{Se}$
	$\mu\text{g/g}$ Dry Waste					
20578	2.18E+01	2.08E+01	(1.48E+00)	ND	(5.58E+00)	ND
20578 Dup1	2.79E+01	2.67E+01	(1.18E+01)	(5.51E+00)	(5.37E+00)	ND
20578 Dup2	2.25E+01	2.24E+01	(1.26E+00)	ND	(1.94E+00)	(9.02E-02)
20578 Avg	2.41E+01	2.33E+01	(4.85E+00)	(5.51E+00)	(4.30E+00)	(9.02E-02)

Sample Number	$^{95}\text{Mo}^{(a)}$	$^{97}\text{Mo}$	$^{98}\text{Mo}$	$^{101}\text{Ru}^{(a)}$	$^{102}\text{Ru}$	$^{104}\text{Ru}$
	$\mu\text{g/g}$ Dry Waste					
20578	(3.28E+00)	(5.56E+00)	(2.16E+00)	5.20E+00	2.03E+00	2.31E+00
20578 Dup1	6.21E+00	8.28E+00	(3.47E+00)	2.01E+01	9.37E+00	8.98E+00
20578 Dup2	(4.09E+00)	6.60E+00	(2.41E+00)	5.18E+00	2.35E+00	2.26E+00
20578 Avg	6.21E+00	7.44E+00	(2.68E+00)	1.02E+01	4.58E+00	4.52E+00

Sample Number	$^{107}\text{Ag}^{(a)}$	$^{109}\text{Ag}$	$^{111}\text{Cd}^{(a)}$	$^{114}\text{Cd}$	$^{121}\text{Sb}$	$^{206}\text{Pb}$	$^{208}\text{Pb}^{(a)}$
	$\mu\text{g/g}$ Dry Waste						
20578	(1.18E+00)	(1.24E+00)	ND	ND	ND	8.92E+00	9.96E+00
20578 Dup1	(1.05E+00)	(9.69E-01)	ND	ND	ND	ND	(2.81E-01)
20578 Dup2	(8.81E-01)	(8.51E-01)	ND	ND	ND	3.49E+00	4.00E+00
20578 Avg	(1.04E+00)	(1.02E+00)	ND	ND	ND	6.21E+00	6.98E+00

(a) ICP-MS mass of choice.  
Concentrations listed in parentheses were <EQL.  
Avg = Average.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.3.1.7 Anions – Single-Contact $\text{Ca}(\text{OH})_2$ Solution Leachants

Leachable concentrations of anions determined from the 1-month contact  $\text{Ca}(\text{OH})_2$  solution leachates measured by IC are shown in Table 3.37. The anions with the highest leachable concentrations were nitrate ( $1.15 \times 10^4 \mu\text{g/g}$ ) and nitrite ( $1.01 \times 10^4 \mu\text{g/g}$ ). Concentrations for most replicates for the other anions were generally below the quantification limit.

**Table 3.37.** Leachable Anion Concentrations Determined from Single-Contact Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste

Sample Number	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	µg/g Dry Waste					
20578	(1.47E+03)	(9.30E+02)	1.59E+03	(1.19E+04)	(2.49E+03)	(4.94E+02)
20578 Dup1	1.02E+04	(7.56E+02)	1.55E+04	1.22E+04	(2.55E+03)	(1.19E+04)
20578 Dup2	(8.11E+02)	(6.32E+02)	1.31E+04	1.08E+04	(1.91E+03)	ND
20578 Avg	1.02E+04	(7.73E+02)	1.01E+04	1.15E+04	(2.32E+03)	(6.20E+03)

Avg = Average.  
Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

### 3.3.2 Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leach Tests

#### 3.3.2.1 Residual Waste to Ca(OH)<sub>2</sub> Solution Ratios: Periodic Replenishment Leach Tests

The dry residual waste concentrations used in periodic replenishment Ca(OH)<sub>2</sub> solution leach tests are shown in (Table 3.38). These values were calculated from the moist weight of the added waste and moisture content of the waste (Table 3.1). The waste concentrations ranged from 7.37 to 7.65 g/L.

**Table 3.38.** Residual Waste to Ca(OH)<sub>2</sub> Solution Ratios Used in the Periodic Replenishment Leach Tests

Sample Number	Dry Waste to Ca(OH) <sub>2</sub> Solution Ratio (g/L)
20578	7.50
20578 Dup1	7.37
20578 Dup2	7.65

Dup = Duplicate.

#### 3.3.2.2 pH and Alkalinity – Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachants

Average alkalinities and pH values measured in tank C-108 residual waste periodic replenishment Ca(OH)<sub>2</sub> solution leachates are listed in Table 3.39. The pH values ranged from a high of 12.2 in the fifth stage contact to a low of 11.6 in the fourth contact. The total alkalinity decreased from a high of  $1.03 \times 10^7$  µg/L (as CaCO<sub>3</sub>) to  $(1.95 \times 10^6$  µg/L) in the fifth contact.

**Table 3.39.** Average pH and Alkalinity Values for Periodic Replenishment  $\text{Ca(OH)}_2$  Solution Leachates

Contact Stage	pH	Total Alkalinity (as $\text{CaCO}_3$ ) at pH 4.5 Endpoint	Total Alkalinity (as $\text{CaCO}_3$ ) at pH 4.5 Endpoint
		$\mu\text{g/L}$	$\mu\text{g/g Dry Waste}$
1	11.8	1.03E+07	1.37E+06
2	11.8	6.54E+06	1.59E+06
3	12.0	6.30E+06	2.91E+04
4	11.6	8.88E+06	1.18E+06
5	12.2	1.95E+06	8.80E+04

### 3.3.2.3 $^{99}\text{Tc}$ and $^{238}\text{U}$ – Periodic Replenishment $\text{Ca(OH)}_2$ Solution Leachants

The average concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  measured in the periodic replenishment  $\text{Ca(OH)}_2$  solution leachates are listed in Table 3.40. The vast majority of the leachable  $^{99}\text{Tc}$  and  $^{238}\text{U}$  in the tank C-108 residual waste was solubilized during the first contact stage. After the first contact,  $^{99}\text{Tc}$  decreased to concentrations that were less than the quantification limit and  $^{238}\text{U}$  concentrations decreased, but not in a consistent manner.

**Table 3.40.** Average Leachable  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations Determined from Periodic Replenishment  $\text{Ca(OH)}_2$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
1	5.59E-01	2.49E+02	9.51E-03	8.38E-05
2	(1.03E-02)	4.94E+00	(1.75E-04)	1.66E-06
3	ND	9.03E-01	ND	3.03E-07
4	(1.43E-03)	1.88E+00	(2.42E-05)	6.32E-07
5	(1.45E-02)	2.12E+00	(2.47E-04)	7.12E-07

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.3.2.4 $^{137}\text{Cs}$ and $^{90}\text{Sr}$ – Periodic Replenishment $\text{Ca(OH)}_2$ Solution Leachants

The average concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  measured in the periodic replenishment  $\text{Ca(OH)}_2$  solution leachates are listed in Table 3.41. Leachable concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  are relatively low in the first contact stage. The highest concentrations of the leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  occur in the second contact stage and decrease in subsequent stages.

**Table 3.41.** Leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations Determined from Periodic Replenishment  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
1	(3.29E-02)	2.50E-02	(2.86)	3.49E+00
2	1.10E-01	1.37E+00	9.53	1.92E+02
3	3.07E-02	2.99E-01	2.67	4.19E+01
4	ND	3.99E-02	ND	5.59E+00
5	ND	2.44E-02	ND	3.41E+00

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.3.2.5 Transuranics – Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachants

Dissolved concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  determined in the tank C-108 1-month  $\text{Ca}(\text{OH})_2$  solution leachates are shown in Table 3.42. Except for  $^{239}\text{Pu}$  during the first stage ( $7.53 \times 10^{-3} \mu\text{Ci/g}$ ), the leachable concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  were below the quantification or detection limit.

**Table 3.42.** Average Leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations Determined from Periodic Replenishment  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
1	(3.91E-05)	(5.51E-02)	7.53E-03	1.02E-01	(7.05E-02)	(2.07E-02)
2	(7.47E-06)	(1.05E-02)	(2.48E-04)	(4.01E-03)	ND	ND
3	(1.32E-05)	(1.86E-02)	(9.86E-04)	(1.59E-02)	ND	ND
4	(1.26E-05)	(1.78E-02)	(7.56E-04)	(1.22E-02)	ND	ND
5	(5.67E-06)	(7.99E-03)	(6.82E-05)	(1.10E-03)	ND	ND

Values within parentheses were <EQL.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

### 3.3.2.6 Metals – Periodic Replenishment $\text{Ca}(\text{OH})_2$ Solution Leachants

The average concentrations of elements measured by ICP-OES in the periodic replenishment  $\text{Ca}(\text{OH})_2$  solution leachates are listed in Table 3.43. The highest concentrations of leachable elements measured by ICP-OES that were solubilized from tank C-108 residual waste occurred during the first



contact stage, with the exception of Bi, Ca, and Sr. During subsequent stages the concentrations of Al, Cr, Fe, K, P, Na, Si, S, and U decreased significantly or to levels that were below the quantification limit or not detectable.

**Table 3.43.** Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	Al	Bi	Ca	Cr	Fe	K
$\mu\text{g/g Dry Waste}$						
1	2.20E+03	ND	(2.77E+01)	2.51E+01	4.11E+01	3.10E+02
2	4.08E+01	(3.84E+01)	6.51E+04	ND	(7.24E+00)	(6.64E+01)
3	ND	7.35E+01	7.88E+04	ND	(9.26E-01)	(6.09E+01)
4	(1.02E+00)	7.64E+01	1.16E+05	ND	2.59E+01	(6.15E+01)
5	(8.54E-01)	8.55E+01	1.16E+05	(2.33E+00)	(1.48E+00)	(4.64E+01)

Contact Stage	P	Sr	Na	Si	S	U
$\mu\text{g/g Dry Waste}$						
1	2.24E+03	ND	1.61E+05	2.53E+02	8.47E+02	2.36E+02
2	4.09E+02	6.42E+02	5.19E+03	1.87E+02	(1.20E+02)	(4.87E+00)
3	(7.66E+01)	2.64E+02	1.03E+03	ND	(2.88E+02)	(3.82E+00)
4	(7.86E+01)	1.80E+02	(1.68E+02)	ND	(3.26E+02)	(3.34E+00)
5	(8.83E+01)	1.74E+02	(1.42E+02)	ND	(2.70E+02)	(5.71E+00)

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

The average concentrations of elements measured by ICP-MS in the periodic replenishment  $\text{Ca}(\text{OH})_2$  solution leachates are listed in Table 3.44. The highest concentrations of leachable trace metals measured by ICP-MS that were solubilized from tank C-108 residual waste and were above the quantification limit occurred during the first contact stage, except for Pb. During subsequent stages the concentrations of all the trace metals—except Pb—decreased to levels that were below the quantification limit or not detectable.

**Table 3.44.** Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste

	Cr <sup>(a)</sup>	Cu – Total <sup>(b)</sup>	As – Total Based on	Se – Total Based on	Mo - Total Based on		
			<sup>75</sup> As	<sup>82</sup> Se	<sup>95</sup> Mo	<sup>97</sup> Mo	<sup>98</sup> Mo <sup>(c)</sup>
Contact Stage	µg/g Dry Waste						
1	2.49E+01	(6.61E+00)	(3.56E-01)	ND	7.81E+00	9.63E+00	(3.63E+00)
2	(8.96E-01)	(1.42E-01)	ND	ND	ND	(1.36E-01)	(3.13E-01)
3	(2.32E+00)	ND	ND	ND	ND	(1.09E-01)	(3.46E-01)
4	ND	ND	(2.69E+00)	(1.89E+00)	ND	ND	(3.81E-02)
5	(2.34E+00)	ND	ND	<sup>(d)</sup>	(3.06E-01)	(4.41E-01)	(5.32E-01)
	Ru – Total Based on Sum of		Ag <sup>(e)</sup>	Cd – Total <sup>(f)</sup>	Sb – Total Based on <sup>121</sup> Sb	Pb – Total <sup>(g)</sup>	
	<sup>101</sup> Ru	<sup>102</sup> Ru					
Contact Stage	µg/g Dry Waste						
1	1.52E+01	7.27E+00	(6.32E-01)	1.30E+00	(4.83E-01)	7.64E+00	
2	(3.08E-01)	(5.26E-01)	(3.63E-01)	(9.04E-01)	(4.34E-01)	2.82E+01	
3	(2.85E-01)	(4.67E-01)	(3.32E-01)	(4.77E-01)	(4.06E-01)	2.91E+01	
4	ND	(1.87E-01)	(1.64E+00)	ND	ND	2.00E+01	
5	(3.59E-01)	(5.56E-01)	(3.64E-01)	1.36E+00	(4.47E-01)	1.51E+01	

(a) Cr results are based on the average of <sup>52</sup>Cr and <sup>53</sup>Cr.  
(b) Cu results are based on the average of <sup>63</sup>Cu and <sup>65</sup>Cu.  
(c) The indicated isotope is the suggested isotope for use to quantify the total concentration of that element.  
(d) Se results not reported for last replenishment due to QC failure.  
(e) Ag results are based on the average of <sup>107</sup>Ag and <sup>109</sup>Ag.  
(f) Cd results are based on the average of <sup>111</sup>Cd and <sup>114</sup>Cd.  
(g) Pb results are based on the average of <sup>206</sup>Pb and <sup>208</sup>Pb.  
Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.  
ND = Instrument returned a zero or negative response.

### 3.3.2.7 Anions – Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachants

Average concentrations of anions measured by IC in the periodic replenishment Ca(OH)<sub>2</sub> solution leachates are listed in Table 3.45. The highest concentrations of leachable anions that were solubilized from tank C-108 residual waste and were above the quantification limit occurred during the first contact stage. During subsequent stages, the concentrations of all the anions decreased to levels that were below the quantification limit or not detectable.

**Table 3.45.** Average Leachable Anion Concentrations Determined from Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	µg/g Dry Waste					
1	8.63E+03	(8.55E+02)	1.64E+04	1.73E+04	(2.91E+03)	(1.75E+03)
2	ND	(7.13E+02)	(3.40E+03)	ND	(9.58E+02)	(2.07E+02)
3	ND	(1.11E+03)	(1.07E+03)	ND	(9.77E+02)	ND
4	ND	(7.74E+02)	ND	ND	(1.10E+03)	ND
5	ND	(2.68E+02)	ND	(1.04E+03)	(1.90E+03)	(4.68E+02)

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

## 3.4 CaCO<sub>3</sub> Solution Leach Tests

### 3.4.1 1-Month Single-Contact CaCO<sub>3</sub> Solution Leach Tests

#### 3.4.1.1 Residual Waste to CaCO<sub>3</sub> Solution Ratios: Single-Contact Leach Tests

Dry residual waste concentrations used in single-contact CaCO<sub>3</sub> solution leach tests are shown in Table 3.46. These values were calculated from the moist weight of the added waste and moisture content of the residual waste (Table 3.1). The waste concentrations ranged from 6.74 to 8.62 g/L.

**Table 3.46.** Residual Waste to CaCO<sub>3</sub> Solution Ratios Used in Single-Contact Leach Tests

Sample Number	Dry Waste to CaCO <sub>3</sub> Solution Ratio (g/L)
20578	8.62
20578 Dup1	6.87
20578 Dup2	6.74
Dup = Duplicate.	

#### 3.4.1.2 pH and Alkalinity – Single-Contact CaCO<sub>3</sub> Solution Leachants

Alkalinities and pH values measured in replicate samples of 1-month CaCO<sub>3</sub> solution leachates of tank C-108 residual waste are listed in Table 3.47. The pH values for the three replicates were very similar with an average of 12.7. The total alkalinity for the three replicates was also very similar with an average of  $6.46 \times 10^6$  µg/L alkalinity (as CaCO<sub>3</sub>).

**Table 3.47.** The pH and Alkalinity Values for Single-Contact CaCO<sub>3</sub> Solution Leachates

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		(µg/L)	(µg/g waste)
20578	12.76	8.06E+06	9.34E+05
20578 Dup1	12.67	6.23E+06	9.05E+05
20578 Dup2	12.69	5.09E+06	7.53E+05
20578 Average	12.71	6.46E+06	8.64E+05
Dup = Duplicate.			

### 3.4.1.3 <sup>99</sup>Tc and <sup>238</sup>U – Single-Contact CaCO<sub>3</sub> Solution Leachants

Dissolved concentrations of <sup>99</sup>Tc and <sup>238</sup>U determined in replicate 1-month CaCO<sub>3</sub> solution leachates of tank C-108 residual waste are shown in Table 3.48. The average leachable <sup>99</sup>Tc concentration is 0.627 µg/g (0.0107 µCi/g) and the average leachable <sup>238</sup>U concentration is 648 µg/g ( $2.18 \times 10^{-4}$  µCi/g). These values equate to an average of 99% (<sup>99</sup>Tc) and 35% (<sup>238</sup>U) compared to the totals determined by EPA acid digestion (Table 3.4).

**Table 3.48.** Leachable <sup>99</sup>Tc and <sup>238</sup>U Concentrations Determined from Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

Sample Number	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	µg/g Dry Waste		µCi/g Dry Waste	
20578	3.60E-01	4.62E+02	6.12E-03	1.55E-04
20578 Dup	5.23E-01	5.94E+02	8.89E-03	2.00E-04
20578 Dup	9.97E-01	8.89E+02	1.69E-02	2.99E-04
20578 Average	6.27E-01	6.48E+02	1.07E-02	2.18E-04
Dup = Duplicate.				

### 3.4.1.4 <sup>137</sup>Cs and <sup>90</sup>Sr – Single-Contact CaCO<sub>3</sub> Solution Leachants

Dissolved concentrations of <sup>137</sup>Cs and <sup>90</sup>Sr determined in replicate 1-month CaCO<sub>3</sub> solution leachates of tank C-108 residual waste are shown in Table 3.49. The average leachable <sup>137</sup>Cs concentration was 5.49 µCi/g ( $63.1 \times 10^{-2}$  µg/g) and the average leachable <sup>90</sup>Sr concentration was 13.1 µCi/g ( $9.36 \times 10^{-2}$  µg/g). These values equate to an average of 32% (<sup>137</sup>Cs) and 11% (<sup>90</sup>Sr) compared to the totals determined by EPA acid digestion (Table 3.4).

**Table 3.49.** Leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations Determined from Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Sample Number	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
20578	6.98E-02	2.37E-01	6.07	3.32E+01
20578 Dup	4.41E-02	3.24E-02	3.84	4.53E+00
20578 Dup	7.54E-02	1.13E-02	6.56	1.58E+00
20578 Average	6.31E-02	9.36E-02	5.49	1.31E+01
Dup = Duplicate.				

### 3.4.1.5 Transuranics – Single-Contact $\text{CaCO}_3$ Solution Leachants

Dissolved concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  determined in the tank C-108 1-month  $\text{CaCO}_3$  solution leachates are shown in Table 3.50. The leachable concentrations for  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  were all below the quantification or detection limits.

**Table 3.50.** Leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations Determined from Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
20578	ND	ND	(3.16E-03)	(5.09E-02)	ND	ND
20578 Dup1	ND	ND	(2.38E-03)	(3.84E-02)	ND	ND
20578 Dup2	(2.83E-05)	(3.99E-02)	(6.02E-03)	(9.71E-02)	ND	ND
20578 Average	(9.13E-06)	(1.29E-02)	(3.85E-03)	(6.21E-02)	ND	ND

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.4.1.6 Metals – Single-Contact $\text{CaCO}_3$ Solution Leachants

Concentrations of a number of metals, such as Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Si, Sr, Ti, Tl, V, Zn, and Zr, in the  $\text{CaCO}_3$  solution leachates were measured by ICP-OES. Among these, only about a dozen metals were present in measurable concentrations (Table 3.51). The concentrations listed within parentheses indicate values that are less than EQL. The most significant  $\text{CaCO}_3$  solution leachable elements in the tank C-108 residual waste samples were Na and P. The average water leachable concentration of Na was  $1.59 \times 10^5 \mu\text{g/g waste}$ . The average  $\text{CaCO}_3$  leachable concentration of P was  $4.54 \times 10^4 \mu\text{g/g waste}$ . Significant concentrations of Al (939  $\mu\text{g/g}$ ), S (806  $\mu\text{g/g}$ ), U (749  $\mu\text{g/g}$ ), and Si (389  $\mu\text{g/g}$ ) were also leachable from these samples.

**Table 3.51.** Leachable Metals Concentrations Determined by ICP-OES in Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

Sample Number	Al	Cr	Fe	P	Na	Si
µg/g Dry Waste						
20578	8.50E+02	1.58E+01	4.17E+01	6.01E+04	1.84E+05	4.08E+02
20578 Dup1	9.17E+02	1.94E+01	2.79E+01	4.93E+04	1.61E+05	5.14E+02
20578 Dup2	1.05E+03	4.30E+01	4.01E+01	2.67E+04	1.33E+05	2.46E+02
20578 Avg	9.39E+02	2.61E+01	3.66E+01	4.54E+04	1.59E+05	3.89E+02

Sample Number	S	U	Ca	K	Ni	Sr
µg/g Dry Waste						
20578	5.65E+02	7.25E+02	(3.23E+01)	(1.90E+02)	(1.20E+00)	(1.59E+00)
20578 Dup1	6.04E+02	5.97E+02	(1.85E+01)	(2.10E+02)	(1.73E+00)	(8.12E-01)
20578 Dup2	1.25E+03	9.25E+02	(5.31E+01)	(3.79E+02)	5.13E+01	(1.56E+00)
20578 Avg	8.06E+02	7.49E+02	(3.46E+01)	(2.60E+02)	5.13E+01	(1.32E+00)

Concentrations listed in parentheses were <EQL.  
 Avg = Average.  
 Dup = Duplicate.  
 EQL = Estimated quantitation limit.

Concentrations of a number of trace metals determined by ICP-MS in the CaCO<sub>3</sub> solution leachates are shown in Table 3.52. The analysis included the metals Cr, Cu, As, Se, Mo, Ru, Ag, Cd, Sb, and Pb. Of these only Cr, Ru, and Pb were present at concentrations above the quantification limit. The average leachable concentrations determined for Cr, Ru, and Pb were 24.7 µg/g, 20.6 µg/g, and 4.2 µg/g.

**Table 3.52.** Leachable Metals Concentrations Determined by ICP-MS in Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/g Dry Waste					
20578	1.32E+01	1.42E+01	(4.32E+00)	ND	(5.43E+00)	ND
20578 Dup1	1.60E+01	1.50E+01	(5.97E+00)	ND	(6.37E+00)	ND
20578 Dup2	4.49E+01	4.92E+01	(6.48E+00)	(3.43E-01)	(2.95E+00)	ND
20578 Avg	2.47E+01	2.61E+01	(5.59E+00)	(3.43E-01)	(4.92E+00)	ND

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/g Dry Waste					
20578	(2.72E+00)	(4.18E+00)	(1.67E+00)	1.15E+01	5.27E+00	5.10E+00
20578 Dup1	(4.66E+00)	(7.84E+00)	(2.87E+00)	1.80E+01	8.03E+00	7.20E+00
20578 Dup2	8.98E+00	1.37E+01	(5.06E+00)	3.22E+01	1.53E+01	1.44E+01
20578 Avg	8.98E+00	1.37E+01	(3.20E+00)	2.06E+01	9.53E+00	8.90E+00

**Table 3.52.** (contd)

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Sample Number	µg/g Dry Waste						
20578	(1.05E+00)	(9.62E-01)	ND	ND	ND	(2.68E+00)	3.47E+00
20578 Dup1	(1.19E+00)	(1.22E+00)	ND	ND	ND	(1.81E+00)	(2.52E+00)
20578 Dup2	(1.41E+00)	(1.38E+00)	ND	ND	ND	(3.67E+00)	4.57E+00
20578 1 Month Avg	(1.22E+00)	(1.19E+00)	ND	ND	ND	(2.72E+00)	4.02E+00

(a) ICP-MS mass of choice.

Concentrations listed in parentheses were <EQL.

Avg = Average

Dup = Duplicate

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.4.1.7 Anions – Single-Contact CaCO<sub>3</sub> Solution Leachants

Leachable concentrations of anions determined from the 1-month contact CaCO<sub>3</sub> solution leachates measured by IC are shown in Table 3.53. The anion with the highest leachable concentration was phosphate ( $1.36 \times 10^5$  µg/g), followed by nitrite ( $2.80 \times 10^4$  µg/g), nitrate ( $2.34 \times 10^4$  µg/g), and then fluoride ( $1.38 \times 10^4$  µg/g). The concentrations of sulfate and chloride were below the quantification limit.

**Table 3.53.** Leachable Anion Concentrations Determined from Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
Sample Number	µg/g Dry Waste					
20578	1.95E+04	(6.96E+02)	(1.03E+04)	(7.51E+03)	(1.93E+03)	1.89E+05
20578 Dup1	ND	(8.72E+02)	ND	ND	(1.03E+03)	(4.92E+02)
20578 Dup2	8.13E+03	(9.91E+02)	2.80E+04	2.34E+04	(3.83E+03)	8.30E+04
20578 1 Month Avg	1.38E+04	(8.53E+02)	2.80E+04	2.34E+04	(2.26E+03)	1.36E+05

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.4.2 Periodic Replenishment CaCO<sub>3</sub> Solution Leach Tests

#### 3.4.2.1 Residual Waste to Double-Deionized Water Ratios: Periodic Replenishment CaCO<sub>3</sub> Solution Leach Tests

The dry residual waste concentrations used in periodic replenishment CaCO<sub>3</sub> solution leach tests are shown in Table 3.54. These values were calculated from the moist weight of the added waste and moisture content of the waste (Table 3.1). The waste concentrations ranged from 7.15 to 8.36 g/L.

**Table 3.54.** Residual Waste to CaCO<sub>3</sub> Solution Ratios Used in the Periodic Replenishment Leach Tests

Sample Number	Dry Waste to CaCO <sub>3</sub> Solution Ratio (g/L)
20578	8.00
20578 Dup1	8.36
20578 Dup2	7.15
Dup = Duplicate.	

#### 3.4.2.2 pH and Alkalinity – Periodic Replenishment CaCO<sub>3</sub> Solution Leachants

Average alkalinities and pH values measured in tank C-108 residual waste periodic replenishment CaCO<sub>3</sub> solution leachates are listed in Table 3.55. The pH values decreased from a high of 12.7 in the first stage contact to 7.2 in the fifth contact. The total alkalinity decreased from a high of  $8.45 \times 10^6$  µg/L (as CaCO<sub>3</sub>) in the first contact to a low of  $2.39 \times 10^4$  µg/L in the fifth contact.

**Table 3.55.** Average pH and Alkalinity Values for Periodic Replenishment CaCO<sub>3</sub> Solution Leachates

Contact Stage	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g Dry Waste
1	12.7	8.45E+06	1.07E+06
2	9.5	5.43E+06	3.14E+04
3	7.4	3.39E+06	1.23E+06
4	7.6	1.11E+05	1.44E+04
5	7.2	2.39E+04	2.97E+03

#### 3.4.2.3 <sup>99</sup>Tc and <sup>238</sup>U – Periodic Replenishment CaCO<sub>3</sub> Solution Leachants

The average concentrations of <sup>99</sup>Tc and <sup>238</sup>U measured in the periodic replenishment CaCO<sub>3</sub> solution leachates are listed in Table 3.56. The vast majority of the leachable <sup>99</sup>Tc and <sup>238</sup>U in the tank C-108 residual waste was solubilized during the first contact stage. After the first contact, <sup>99</sup>Tc decreased to concentrations that were less than the quantification limit. The concentration of <sup>238</sup>U in the second stage



decreased to less than 1% of the concentration in stage 1. During subsequent stages, the  $^{238}\text{U}$  concentrations remained at concentrations that were fairly similar to those in stage 2.

**Table 3.56.** Average Leachable  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations Determined from Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
1	4.39E-01	5.55E+02	7.47E-03	1.86E-04
2	(2.23E-02)	4.86E+00	(3.79E-04)	1.63E-06
3	(7.02E-03)	6.24E+00	(1.19E-04)	2.10E-06
4	(7.28E-04)	4.21E+00	(1.24E-05)	1.41E-06
5	(4.11E-02)	2.98E+00	(6.99E-04)	1.00E-06

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.

#### 3.4.2.4 $^{137}\text{Cs}$ and $^{90}\text{Sr}$ – Periodic Replenishment $\text{CaCO}_3$ Solution Leachants

Average concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  measured in the periodic replenishment  $\text{CaCO}_3$  solution leachates are listed in Table 3.57. Similar to the  $\text{Ca}(\text{OH})_2$  leachates, the leachable concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  are relatively low in the first contact stage. The highest concentrations of the leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  occur in the second contact stage and then decrease in subsequent stages.

**Table 3.57.** Leachable  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations Determined from Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/g Dry Waste}$		$\mu\text{Ci/g Dry Waste}$	
1	4.83E-02	8.86E-02	4.20E+00	1.24E+01
2	1.18E-01	5.22E+00	1.03E+01	7.31E+02
3	4.96E-02	2.45E+00	4.32E+00	3.43E+02
4	2.95E-02	7.14E-01	2.57E+00	1.00E+02
5	ND	2.46E-01	ND	3.44E+01

Concentrations listed in parentheses were <EQL.  
EQL = Estimated quantitation limit.

#### 3.4.2.5 Transuranics – Periodic Replenishment $\text{CaCO}_3$ Solution Leachants

The average concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  measured in the periodic replenishment  $\text{CaCO}_3$  solution leachates are listed in Table 3.58. The highest concentrations of leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  that occurred in the tank C-108 residual waste was solubilized during the first contact stage.

During subsequent stages the concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  decreased significantly. Concentrations in subsequent stages decreased to concentrations that were nearly an order of magnitude less than those in the first stage or less than the quantification or detection limits.

**Table 3.58.** Average Leachable  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations Determined from Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$	$\mu\text{Ci/g Dry Waste}$	$\mu\text{g/g Dry Waste}$
1	6.50E-05	9.16E-02	1.01E-02	1.63E-01	(8.76E-02)	(2.58E-02)
2	(4.05E-06)	(5.70E-03)	(2.19E-04)	(3.54E-03)	ND	ND
3	ND	ND	(1.34E-05)	(2.15E-04)	ND	ND
4	(1.10E-06)	(1.55E-03)	(1.75E-04)	(2.82E-03)	ND	ND
5	(1.62E-05)	(2.28E-02)	(4.88E-04)	(7.87E-03)	ND	ND

Values within parentheses were <EQL.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

### 3.4.2.6 Metals – Periodic Replenishment $\text{CaCO}_3$ Solution Leachants

Average concentrations of elements measured by ICP-OES in the periodic replenishment  $\text{CaCO}_3$  solution leachates are listed in Table 3.59. The highest concentrations of leachable elements measured by ICP-OES that were solubilized from tank C-108 residual waste occurred during the first contact stage, with the exception of Bi, Ca, and Sr. During subsequent stages, the concentrations of Al, Cr, Fe, P, Na, S, and U decreased significantly or to levels that were below the quantification limit or not detectable. Decreases in Sr, K, and Si were much less dramatic during subsequent stages.

**Table 3.59.** Average Leachable Metals Concentrations Determined by ICP-OES in Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste

Contact Stage	Al	Bi	Ca	Cr	Fe	K
	$\mu\text{g/g Dry Waste}$					
1	5.68E+02	ND	(1.90E+01)	1.85E+01	7.66E+01	(1.97E+02)
2	8.21E+01	7.59E+01	2.90E+04	ND	(8.54E+00)	(4.40E+01)
3	3.57E+01	(4.66E-01)	6.63E+02	ND	(4.83E+00)	(2.09E+01)
4	2.63E+01	(5.57E+00)	7.62E+02	ND	(3.14E+00)	(2.29E+01)
5	(9.19E+00)	ND	7.39E+02	(4.98E-01)	(1.67E-01)	(1.53E+01)

**Table 3.59.** (contd)

	P	Sr	Na	Si	S	U
Contact Stage	µg/g Dry Waste					
1	6.06E+04	(8.38E-01)	1.88E+05	1.95E+02	6.01E+02	5.39E+02
2	5.29E+02	4.48E+02	7.78E+03	1.87E+02	(6.82E+01)	(9.13E+00)
3	(9.76E+01)	1.97E+02	3.45E+02	1.55E+02	(2.34E+01)	(1.07E+01)
4	(6.55E+01)	9.61E+01	(8.06E+01)	1.51E+02	(1.84E+01)	(8.98E+00)
5	(3.99E+01)	4.16E+01	(4.32E+01)	1.33E+02	ND	(7.38E+00)

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

The average concentrations of elements measured by ICP-MS in the periodic replenishment CaCO<sub>3</sub> solution leachates are listed in Table 3.60. Except for Pb, the highest concentrations of leachable trace metals measured by ICP-MS that were solubilized from tank C-108 residual waste occurred during the first contact stage. During subsequent stages, the concentrations of all the metals except Pb decreased significantly or to levels that were below the quantification limit or not detectable. Pb concentrations generally decreased during subsequent stages but not in a consistent fashion.

**Table 3.60.** Average Leachable Metals Concentrations Determined by ICP-MS in Periodic Replenishment CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

Contact Stage	Cr <sup>(a)</sup>	Cu – Total <sup>(b)</sup>	As – Total	Se – Total	Mo – Total Based on		
			Based on	Based on			
			<sup>75</sup> As	<sup>82</sup> Se	<sup>95</sup> Mo	<sup>97</sup> Mo	<sup>98</sup> Mo <sup>(c)</sup>
µg/g Dry Waste							
1	1.65E+01	(5.64E+00)	(1.36E+00)	ND	(4.72E+00)	7.63E+00	(3.23E+00)
2	(1.04E-01)	(4.51E-01)	ND	ND	(2.34E-01)	(5.00E-01)	(6.61E-01)
3	(4.02E-01)	(1.21E-01)	ND	ND	(3.34E-01)	(3.03E-01)	(4.75E-01)
4	(3.97E-01)	ND	ND	ND	(6.95E-02)	(3.76E-01)	(2.98E-01)
5	ND	ND	ND	<sup>(d)</sup>	(1.65E-01)	(4.35E-01)	(4.99E-01)

(a) Cr results are based on the average of <sup>52</sup>Cr and <sup>53</sup>Cr.

(b) Cu results are based on the average of <sup>63</sup>Cu and <sup>65</sup>Cu.

(c) The indicated isotope is the suggested isotope for use to quantify the total concentration of that element.

(d) Se results not reported for last replenishment due to QC failure.

**Table 3.60.** (contd)

	Ru – Total Based on Sum of		Ag <sup>(e)</sup>	Cd – Total <sup>(f)</sup>	Sb – Total Based on <sup>121</sup> Sb	Pb – Total <sup>(g)</sup>
	<sup>101</sup> Ru	<sup>102</sup> Ru				
Contact Stage	µg/g Dry Waste					
1	1.49E+01	6.93E+00	(6.29E-01)	1.52E+00	(5.39E-01)	4.23E+00
2	(3.67E-01)	(3.97E-01)	(3.57E-01)	1.12E+00	(4.23E-01)	2.42E+01
3	(2.53E-01)	(2.06E-01)	(3.37E-01)	(8.11E-01)	(3.81E-01)	(9.05E-01)
4	(2.29E-01)	(1.41E-01)	(3.17E-01)	(2.68E-01)	(3.50E-01)	(1.11E+00)
5	(2.80E-01)	(1.33E-01)	(3.35E-01)	1.30E+00	(4.01E-01)	(1.34E-01)

(e) Ag results are based on the average of <sup>107</sup>Ag and <sup>109</sup>Ag.

(f) Cd results are based on the average of <sup>111</sup>Cd and <sup>114</sup>Cd.

(g) Pb results are based on the average of <sup>206</sup>Pb and <sup>208</sup>Pb.

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

### 3.4.2.7 Anions – Periodic Replenishment CaCO<sub>3</sub> Solution Leachants

Average concentrations of anions measured by IC in the periodic replenishment CaCO<sub>3</sub> solution leachates are listed in Table 3.61. The highest concentrations of leachable anions that were solubilized from tank C-108 residual waste occurred during the first contact stage. During subsequent stages, the concentrations of all the anions that were quantified decreased to levels that were below the quantification limit or not detectable.

**Table 3.61.** Average Leachable Anion Concentrations Determined from Periodic Replenishment CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	µg/g Dry Waste					
1	1.97E+04	(7.56E+02)	1.45E+04	(9.92E+03)	(2.25E+03)	1.92E+05
2	ND	(7.05E+02)	(3.36E+03)	ND	(8.96E+02)	(4.95E+02)
3	ND	(7.06E+02)	ND	ND	(1.16E+03)	(4.10E+02)
4	1.33E+04	(7.28E+02)	1.19E+04	(2.78E+03)	(1.32E+03)	1.30E+05
5	ND	(1.59E+02)	ND	(1.26E+03)	(1.58E+03)	(6.92E+02)

Concentrations listed in parentheses were <EQL.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

## 3.5 X-Ray Diffraction Results

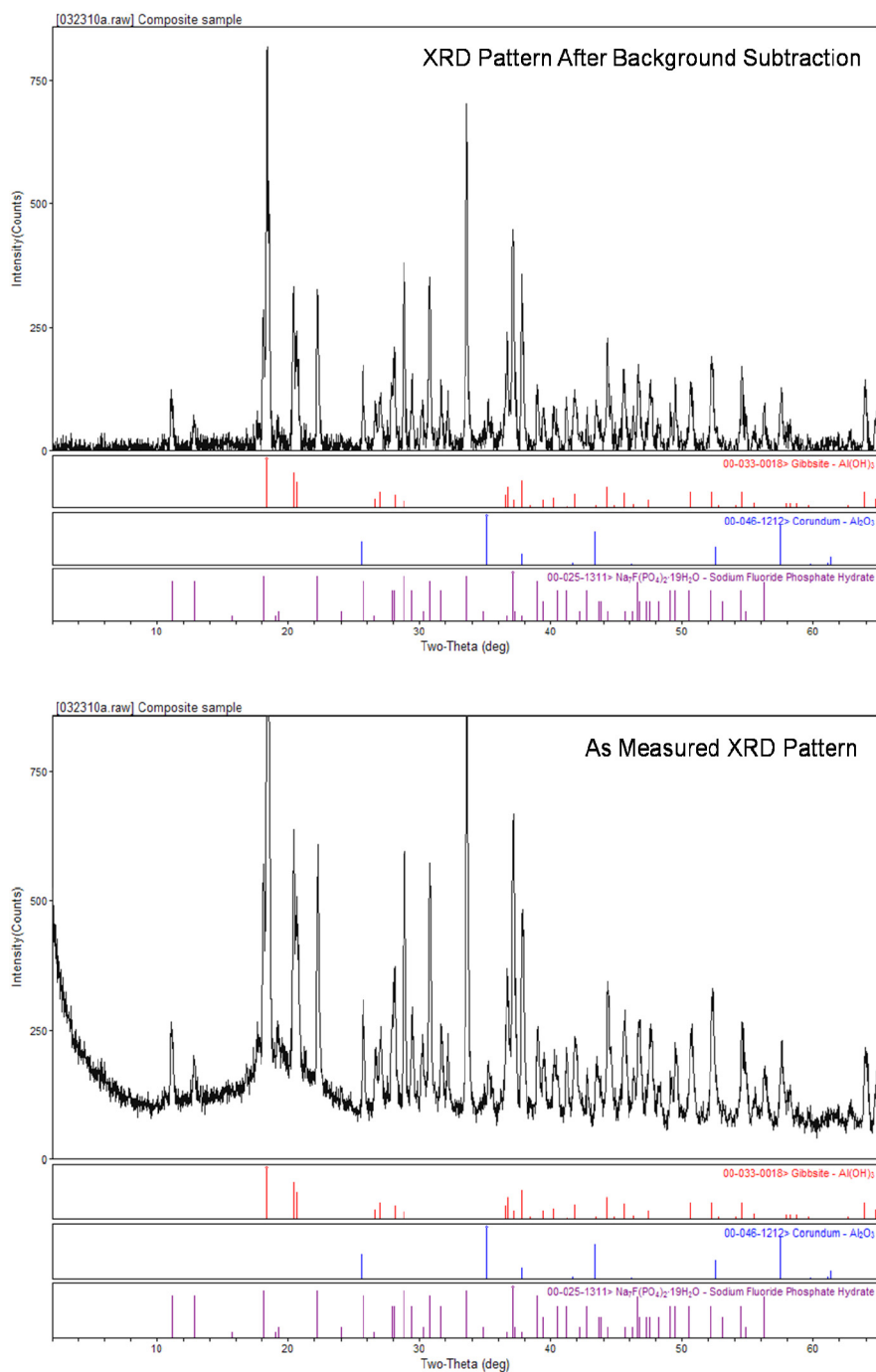
Bulk XRD patterns were measured on the as-received tank C-108 composite sample, three sub-samples selectively removed from as-received sample material (green, black, and crystal nodules), and

three water-leached samples (1-month DDI water, 1-month  $\text{Ca}(\text{OH})_2$ , and 1-month  $\text{CaCO}_3$ ) for a total of seven samples. Phase identification was based on a comparison of the peak reflections and intensities observed in each pattern to the mineral PDF<sup>TM</sup> published by the JCPDS ICDD. The following XRD patterns show, for comparison purposes, one or more schematic database (PDF) patterns considered for phase identification, such as gibbsite (PDF #00-033-0018) and corundum (PDF #00-010-0173). The XRD samples of tank C-103 residual waste included the addition of a trace quantity of reference-material corundum powder ( $\alpha\text{-Al}_2\text{O}_3$ , alumina) (NIST Standard Reference Material [SRM] 676) as an internal  $2\theta$  standard for each XRD pattern. The height of each line in the schematic PDF patterns represents the relative intensity of an XRD peak (i.e., the most intense [the highest] peak has a relative intensity  $[I/I_0]$  of 100%). A crystalline phase typically must be present at greater than ~5-10 wt% of the total sample mass (greater than 1 wt% under optimum conditions) to be readily detected by XRD. Phase identification from the XRD patterns was done in an iterative fashion by considering the compositions of phases identified by SEM/EDS (Section 3.6) in the unleached and leached residual waste samples.

Figure 3.1 shows typical XRD patterns for the as-received tank C-108 residual waste. The top image is the as-measured XRD pattern, and the bottom image is the XRD pattern corrected by background subtraction. The XRD pattern is shown as a function of degrees  $2\theta$  based on  $\text{Cu}_{K\alpha}$  radiation ( $\lambda=1.5406 \text{ \AA}$ ). The vertical axis in each pattern represents the intensity or relative intensity of the XRD peaks. All of the as-measured and corrected XRD patterns are included in Appendix B.

All of the as-measured XRD patterns (e.g., see top image in Figure 3.1) contain a broad diffraction profile (or hump) from approximately  $10$  to  $30^\circ 2\theta$ . This feature is indicative of diffraction from amorphous materials, which cannot be identified by XRD methods. For these samples, diffraction from the nitrocellulose binder is thought to be the main contributor to this broad profile. This is consistent with the SEM/EDS results (Section 3.6) which show that the majority of the particles (probably more than ~90%) have crystal faces and thus are likely crystalline. The maximum intensity (i.e., height) (see XRD patterns in Appendix B) of this broad diffraction profile varies between all samples (including unleached and leached), but not in any systematic fashion. Because the nitrocellulose binder is thought to be the main contributor to this broad profile (XRD pattern of a blank slide containing just binder is shown in Figure 2.3), the variability of the intensity of this broad diffraction profile in the different XRD patterns is likely due to variations in the relative amounts of nitrocellulose binder and waste solid used for each XRD mount. Given the difficulties associated with working with these radioactive materials, it is not possible to control the proportion of nitrocellulose binder to tank waste solid used for each XRD mount.

Table 3.62 lists the phases whose PDF database patterns match the corrected XRD patterns for the unleached and leached waste samples. Corundum is also present in all XRD patterns as it was added to all XRD samples as an internal  $2\theta$  standard. The XRD results indicate that all of the unleached and leached tank C-108 samples contain significant amounts of gibbsite  $[\text{Al}(\text{OH})_3]$ . In the as-received sample, sodium fluoride phosphate hydrate  $[\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}]$  was also identified. In the green nodule, sodium phosphate ( $\text{Na}_3\text{PO}_4$ ), nickel oxide hydroxide ( $\text{NiOOH}$ ), and nitratine ( $\text{NaNO}_3$ ) were also identified. In the black and crystal nodules, natrophosphate  $[\text{Na}_7(\text{PO}_4)_2\text{F} \cdot 19\text{H}_2\text{O}]$  was also identified. Beside gibbsite, no other crystalline phases were identified in any of the solution leached samples. This suggests that the solutions were effective at dissolving the bulk of the crystalline phases other than gibbsite.



**Figure 3.1.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for Composite Subsample from the As-Received Tank C-108 Residual Waste (Sample 20578)

**Table 3.62.** Summary of Phases that Match the XRD Patterns for the As-Received, Green Nodule, Black Nodule, Crystal Nodule, DDI Water-Leached, Ca(OH)<sub>2</sub>-Leached, and CaCO<sub>3</sub>-Leached Samples of Tank C-108 Residual Waste. (Corundum [ $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, PDF 00-010-0173] was also present in all XRD samples as it was added as an internal 2 $\theta$  standard.)

Phase Identification, Formula, and PDF Pattern Number	As- Received	Green Nodule	Black Nodule	Crystal Nodule	DDI Water- Leached	Ca(OH) <sub>2</sub> - Leached	CaCO <sub>3</sub> - Leached
Gibbsite Al(OH) <sub>3</sub> PDF 00-033-0018	X	X	X	X	X	X	X
Natrophosphate Na <sub>7</sub> (PO <sub>4</sub> ) <sub>2</sub> F·19H <sub>2</sub> O PDF 00-025-0831	--	--	X	X	--	--	--
Sodium fluoride phosphate hydrate Na <sub>7</sub> F(PO <sub>4</sub> ) <sub>2</sub> ·19H <sub>2</sub> O PDF 00-025-1311	X	--	--	--	--	--	--
Sodium phosphate Na <sub>3</sub> PO <sub>4</sub> PDF 00-025-1311	--	X	--	--	--	--	--
Nickel oxide hydroxide NiOOH PDF 00-006-0141	--	X	--	--	--	--	--
Nitratine NaNO <sub>3</sub> PDF 00-036-1474	--	X	--	--	--	--	--

X = Indicates that PDF pattern for that phase is consistent with the corrected XRD pattern for the corresponding tank C-108 waste sample.

### 3.6 Scanning Electron Microscopy/Energy Dispersive Spectrometry Results

Analysis by SEM/EDS was conducted on the as-received tank C-108 composite sample, three subsamples selectively removed from as-received sample material (green, black, and crystal nodules) and three water-leached samples (1-month DDI water, 1-month Ca(OH)<sub>2</sub>, and 1-month CaCO<sub>3</sub>) for a total of seven samples. Sample mount preparation and SEM/EDS analyses were completed as described in Section 2.4. Appendices C through I contain all SEM micrographs for the seven samples of tank C-108 residual waste and the tabulations of the elemental compositions derived from the EDS analyses of particles in these SEM-analyzed samples. All SEM micrographs (including those shown in this section) were collected using BSE emission to facilitate identification of particles that contain elements with large atomic numbers (i.e., the larger the atomic number, the brighter the signal contrast).

The SEM/EDS analysis results in terms of the likely phases based upon the elemental composition, morphology, and the XRD results for the tank C-108 residual waste samples are summarized in Table 3.63. The SEM micrographs and elemental EDS data in Appendices C through I were used to create this table. Figure 3.2 is a low-magnification micrograph that shows the morphologies, sizes, and

**Table 3.63.** Summary of Phases Indicated by SEM/EDS and XRD in Tank C-108 Residual Waste Samples

Compositions of Phases Identified by SEM/EDS	Phases Identified by XRD	Information Regarding Occurrence Based on SEM/EDS Analyses
Because H is not detectable by EDS and C was used for coating the SEM mounts, all of the phases listed below at being identified by SEM/EDS may also contain H and/or C.		
<b>As-Received Composite Residual Waste Sample</b>		
Al-Na-O-P-F Al-Na-O-P Na-O-P-F Na-O-P	Gibbsite Sodium fluoride phosphate hydrate	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Sr sometimes associated with the Na-O-P phase</li> <li>• Minor amounts of U, Fe, Ni, and Cr sometimes associated with this phase</li> </ul>
Al-Na-O	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of sodium carbonate, and sodium hydroxide</li> </ul>
Na-O	Nitratine	<ul style="list-style-type: none"> <li>• Likely nitratine, sodium carbonate and/or sodium hydroxide</li> </ul>
Na-U-O		<ul style="list-style-type: none"> <li>• Likely čejkaite and/or sodium uranate</li> </ul>
<b>Green Nodule</b>		
Al-Na-O-P-F Al-Na-O-P Na-O-P-F Na-O-P	Gibbsite Sodium phosphate Nitratine	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of sodium phosphate, natrophosphate, sodium fluoride phosphate hydrate, sodium carbonate, and sodium hydroxide</li> <li>• Sr sometimes associated with this phase</li> <li>• Minor amounts of U, Fe, Ni, and Cr sometimes associated with this phase</li> </ul>
Al-Na-O	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of sodium carbonate, and sodium hydroxide</li> </ul>
Na-O	Nitratine	<ul style="list-style-type: none"> <li>• Likely nitratine, sodium carbonate and/or sodium hydroxide</li> </ul>
Fe-Na-O	Nickel oxide hydroxide	<ul style="list-style-type: none"> <li>• Likely Fe-oxide with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Often associated with Ni and traces of other metals such as Pb, Zn, and Cu</li> </ul>
Na-U-O		<ul style="list-style-type: none"> <li>• Likely čejkaite and/or sodium uranate</li> </ul>
<b>Black Nodule</b>		
Al-Na-O-P-F Al-Na-O-P Na-O-P-F Na-O-P	Gibbsite Natrophosphate	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Minor amounts of U, Fe, Ni, and Cr sometimes associated with this phase</li> </ul>
Al-Na-O	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of sodium carbonate, and sodium hydroxide</li> </ul>
Na-O	--	<ul style="list-style-type: none"> <li>• Likely nitratine, sodium carbonate and/or sodium hydroxide</li> </ul>
Fe-Na-O	--	<ul style="list-style-type: none"> <li>• Likely Fe-oxide with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Often associated with Ni and traces of other metals such as Pb, Zn, and Cu</li> </ul>
Na-U-O	--	<ul style="list-style-type: none"> <li>• Likely čejkaite and/or sodium uranate</li> </ul>



**Table 3.63. (contd)**

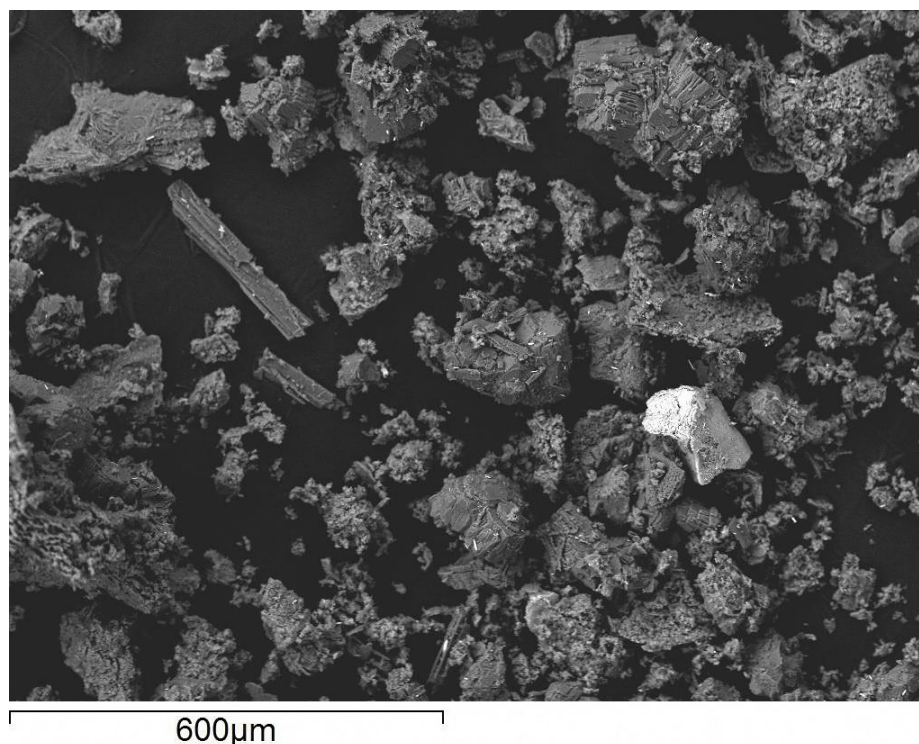
Compositions of Phases Identified by SEM/EDS	Phases Identified by XRD	Information Regarding Occurrence Based on SEM/EDS Analyses
<b>Crystal Nodule</b>		
Al–Na–O–P–F	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Sr sometimes associated with this phase</li> <li>• Minor amounts of U, Fe, Ni, and Cr sometimes associated with this phase</li> </ul>
Al–Na–O–P	Natrophosphate	
Na–O–P–F		
Na–O–P		
Al–Na–O	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of sodium carbonate, and sodium hydroxide</li> </ul>
Na–O	--	<ul style="list-style-type: none"> <li>• Likely nitratine, sodium carbonate and/or sodium hydroxide</li> </ul>
Fe–Na–O	--	<ul style="list-style-type: none"> <li>• Likely Fe-oxide with coatings of natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, and sodium hydroxide</li> <li>• Often associated with some Ni and traces of other metals such as Pb, Zn, and Cu</li> </ul>
Na–U–O	--	<ul style="list-style-type: none"> <li>• Likely čejkaite and/or sodium uranate</li> </ul>
<b>Water and CaCO<sub>3</sub> Leached Composite Residual Waste Sample</b>		
Al–O	Gibbsite	<ul style="list-style-type: none"> <li>• Gibbsite with minor amounts of residual Na–O–P coating</li> </ul>
Fe–O–Al–P–Na	--	<ul style="list-style-type: none"> <li>• Fe-oxide with minor amounts of residual Na–O–P coating</li> <li>• Often associated with Ni and traces of other metals such as Pb, Zn, and Cu</li> </ul>
Sr–Na–P–O	--	<ul style="list-style-type: none"> <li>• Likely SrNaPO<sub>4</sub></li> </ul>
<b>Ca(OH)<sub>2</sub> Leached Composite Residual Waste Sample</b>		
Ca–Al–Na–O–P–F	Gibbsite	<ul style="list-style-type: none"> <li>• Likely gibbsite with coatings of one or more of a phase with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) which likely includes Ca(OH)<sub>2</sub> and calcium phosphate</li> </ul>
Ca–O–P–F	--	<ul style="list-style-type: none"> <li>• Calcium phosphate and calcium phosphate fluoride</li> <li>• Sr sometimes associated with Ca–O–P</li> </ul>
Ca–O–P	--	<ul style="list-style-type: none"> <li>• Fe-oxide with coatings of one or more of a phase with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) which likely includes Ca(OH)<sub>2</sub></li> <li>• Often associated with Ni and traces of other metals such as Pb, Zn, and Cu</li> </ul>
Fe–O–Ca–P–Na	--	

surface textures of typical particles present in the composite sample of as-received tank C-108 residual waste. Analysis of the SEM micrographs—such as Figure 3.3—indicates the as-received tank C-108 residual waste is composed of a number of phases. The EDS analyses indicate that the majority of the particles shown in Figure 3.2 (as-received tank C-108 composite sample) are composed of Al–Na–O–P–F, Al–Na–O–P, Na–O–P–F, Na–O–P, Al–Na–O, and Na–O, all in variable proportions. Note that all phases could also potentially contain H and C, which cannot be quantified by EDS. Combining these results

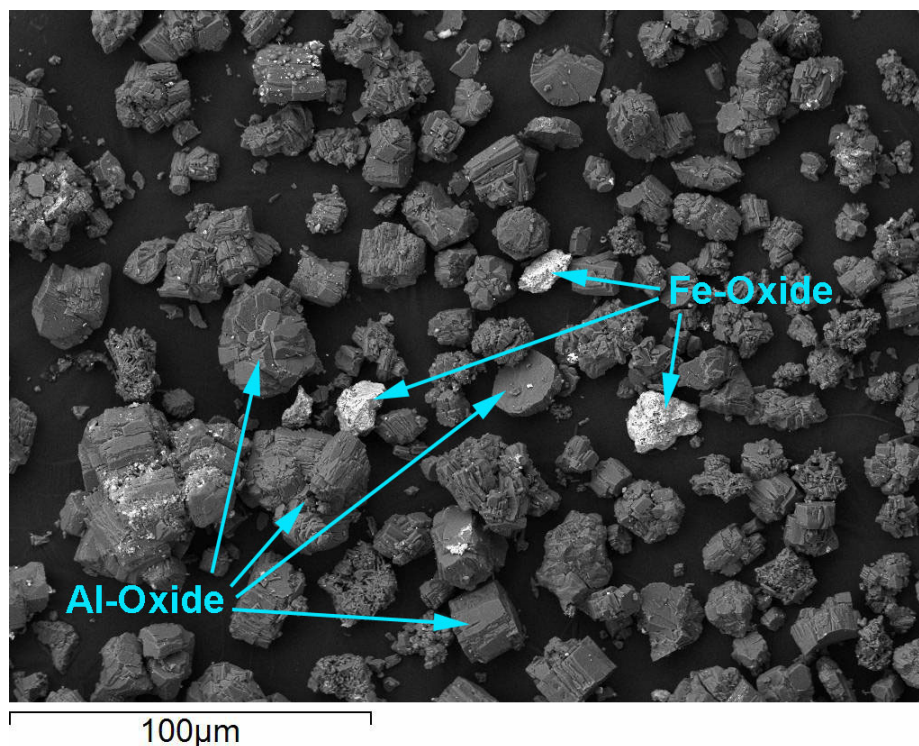
with the XRD results the following conclusions were made regarding the likely occurrence of phases in the tank C-108 residual waste composite sample:

- Gibbsite is a major phase present and appears to be coated and mixed with variable amounts of phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate.
- Sr is sometimes associated with a Na–O–P phase, possibly as  $\text{NaSrPO}_4$  or a similar phase.
- Minor amounts of U, Fe, Ni, and Cr are sometimes associated with the other phases indicated in the first bullet.
- Small amounts of a U–Na–O phase(s) occurs, likely čejkaite and/or sodium urinate.

The compositions determined by SEM/EDS for the green, black, and crystalline nodules are quite similar to those of the composite samples. In addition to the phases noted above, the green, black, and crystalline nodules contained one or more Fe-oxide phases that appear to be coated and mixed with variable amounts of gibbsite, natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate. The iron oxides are often associated with minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu.  $\text{NiOOH}$  was identified by XRD in the green nodule sample. It is possible that the Ni phase occurs as a separate phase sometimes coating the iron oxide. It is also noteworthy that for sample locations that were identified as containing U–Na–O phase(s), the morphology of these particles is very similar to čejkaite which were identified in unleached tank C-203 preretrieval waste (Deutsch et al. 2007b). The čejkaite in the unleached tank C-203 preretrieval waste was identifiable by XRD because of its much higher concentrations in this sample.



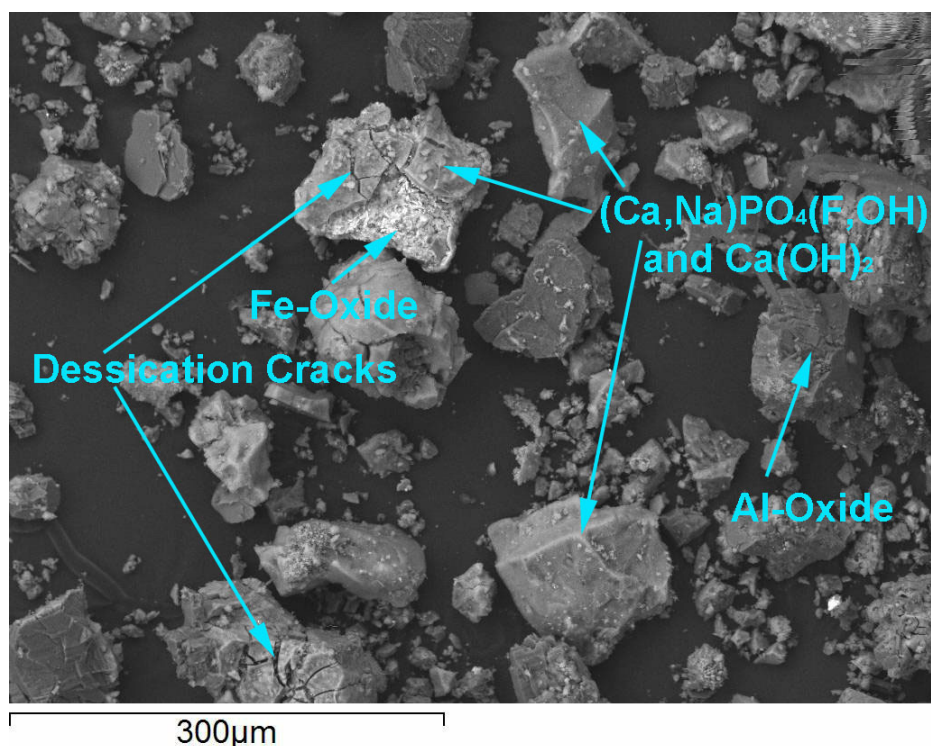
**Figure 3.2.** Backscattered Electron SEM Image of the As-Received Composite Tank C-108 Residual Waste Sample (Site 1)



**Figure 3.3.** Backscattered Electron SEM Image of DDI Water-Leached Sample (Site 1), Tank C-108 Residual Waste

Figure 3.3 is a low-magnification micrograph that shows the morphologies, sizes, and surface textures of typical particles present in the DDI water leach composite sample of tank C-108 residual waste. Analysis of the SEM micrographs—such as Figure 3.3—and the EDS results (Appendix G) indicate that the DDI water-leached tank C-108 residual waste is composed primarily of gibbsite with lesser amounts of Fe oxides. The SEM/EDS results for the  $\text{CaCO}_3$  leached samples were essentially the same as those for the DDI water-leached samples (Appendix I). It is apparent that leaching with DDI and  $\text{CaCO}_3$  results in the majority of the more soluble phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate being largely dissolved away, leaving relatively clean gibbsite and Fe oxides particles with only minor amounts of surface coatings remaining. The elemental compositions of many of the particles analyzed are close to the theoretical composition of gibbsite (25 atomic percent for Al and 75 atomic percent O); see Appendix G. As was the case for the unleached samples, the Fe-oxides are often associated with minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu. Particles containing Sr with an elemental composition of  $\text{SrNaPO}_4$  were also occasionally identified in the DDI and  $\text{CaCO}_3$  leached samples.

The appearance of the  $\text{Ca(OH)}_2$  leached samples is quite different than the DDI and  $\text{CaCO}_3$  leached samples (Figure 3.4). The surfaces of the particles appear to be coated and desiccation cracks are evident on several of the particles in the image.



**Figure 3.4.** Backscattered Electron SEM Image of  $\text{Ca}(\text{OH})_2$  Leached Sample (Site 1), Tank C-108 Residual Waste

The SEM/EDS results for the  $\text{Ca}(\text{OH})_2$  leached samples (Appendix H) suggest the following phases are likely present:

- Gibbsite as the dominant phase with coatings of one or more phases with the general composition of  $(\text{Ca},\text{Na})\text{PO}_4(\text{F},\text{OH})$  and  $\text{Ca}(\text{OH})_2$ .
- Lesser amounts of Fe-oxide(s) with coatings of one or more phase(s) with the general composition of  $(\text{Ca},\text{Na})\text{PO}_4(\text{F},\text{OH})$  and  $\text{Ca}(\text{OH})_2$ .
- The Fe-oxide(s) is often associated with Ni and traces of other metals such as Pb, Zn, and Cu.

None of these suggested phases apart from gibbsite were confirmed by XRD in the  $\text{Ca}(\text{OH})_2$  leached sample.

### 3.7 Geochemical Modeling

Calculated saturation indices for relevant Al-, Na-, Ca-,  $\text{PO}_4$ -, and U-containing minerals for the 1-month single-contact leachates for the tank C-108 residual waste samples are presented in this section. Minerals that are very highly undersaturated or oversaturated are not included. Minerals that are expected to form only at high temperature or under hydrothermal conditions are also not included. SI values that were calculated with estimated analytical data (below the quantification limit) for at least one constituent are indicated in red in the tables. For these cases, the SI values must be considered to have a higher degree of uncertainty.



Calculated mineral saturation indices for the 1-month single-contact DDI leachates for the tank C-108 residual waste samples are shown in Table 3.64. All three aluminum (oxy) hydroxide minerals listed in the table (diaspore, boehmite, and gibbsite) are undersaturated in the 1-month single-contact DDI leachates. The diaspore is closest to equilibrium; however, the solid phase characterization results did not indicate the presence of either diaspore or boehmite. The XRD results did indicate that gibbsite does occur in these samples. It is possible that boehmite and/or diaspore do occur at concentrations below the detection limit of XRD.

SI values for hydroxylapatite are variable, with sample 20578 being near saturation, sample 20578 Dup 1 being oversaturated, and sample 20578 Dup 2 being undersaturated. Whitlockite is very undersaturated in the 1-month single-contact DDI leachates as are calcite and aragonite. The amorphous form of sodium uranate [ $\text{Na}_2\text{U}_2\text{O}_7$  (am)] appears to be close to slightly oversaturated in the 1-month single-contact DDI leachates. The crystalline form [ $\text{Na}_2\text{U}_2\text{O}_7$  (c)] is highly oversaturated. Plattnerite also appears to be near to slightly oversaturated in the 1-month single-contact DDI leachates. Bunsenite is close to saturation to undersaturated in the 1-month single-contact DDI leachates.  $\text{Ni}(\text{OH})_2$  is significantly more undersaturated in the 1-month single-contact DDI leachates than bunsenite. The relatively high degree of variability for the SI values for the Ca and Ni containing compounds is because the concentration values for these metals were below the quantification limits, and as a result are estimated values subject to a higher degree of error.

**Table 3.64.** Calculated Saturation Indices for Relevant Al-, Na-, Ca-,  $\text{PO}_4$ -, and U-Containing Minerals Based on Compositions of 1-Month Single-contact DDI Leachates of Tank C-108 Residual Waste Samples

Phase	Sample 20578	Sample 20578 Dup1	Sample 20578 Dup2
	Saturation Index (unitless)		
Diaspore [ $\text{AlO}(\text{OH})$ ]	-0.83	-0.86	-0.72
Boehmite [ $\text{AlO}(\text{OH})$ ]	-1.23	-1.26	-1.12
Gibbsite [ $\text{Al}(\text{OH})_3$ ]	-1.43	-1.45	-1.31
Hydroxylapatite [ $\text{Ca}_5(\text{OH})(\text{PO}_4)_3$ ]	0.18	1.64	-0.74
Whitlockite [ $\text{Ca}_3(\text{PO}_4)_2$ ]	<-3.00	-2.49	<-3.00
Calcite ( $\text{CaCO}_3$ )	-1.81	-1.45	-1.93
Aragonite ( $\text{CaCO}_3$ )	-1.96	-1.60	-2.08
$\text{Na}_2\text{U}_2\text{O}_7$ (am)	0.46	0.82	0.44
$\text{Na}_2\text{U}_2\text{O}_7$ (c)	2.96	3.33	2.95
Plattnerite [ $\text{PbO}_4$ ]	0.46	0.76	0.82
Bunsenite [ $\text{NiO}$ ]	-1.23	-0.70	-0.14
$\text{Ni}(\text{OH})_2$	-1.51	-0.98	-0.42
SI values calculated with estimated analytical data (below the quantification limit) for at least one constituent are indicated in red.			

Calculated mineral saturation indices (unitless) for the 1-month single-contact  $\text{Ca}(\text{OH})_2$  solution leachates for tank C-108 residual waste samples are shown in Table 3.65. The three aluminum (oxy)

hydroxide minerals listed in Table 3.65 (diaspore, boehmite, and gibbsite) are much closer to saturation in the 1-month single-contact  $\text{Ca(OH)}_2$  solution leachates than in the 1-month single-contact DDI leachates. Diaspore is quite close to equilibrium in the  $\text{Ca(OH)}_2$  solution leachates. Solid phase characterization results did not verify the presence of this mineral. The XRD results did indicate that gibbsite does occur in these samples. It is possible that boehmite and/or diaspore occur at concentrations below the detection limit of XRD.

**Table 3.65.** Calculated Saturation Indices for Relevant Al-, Na-, Ca-,  $\text{PO}_4$ -, and U-Containing Minerals Based on Compositions of 1-Month Single-Contact  $\text{Ca(OH)}_2$  Solution Leachates of Tank C-108 Residual Waste Samples

Phase	Sample 20578	Sample 20578 Dup1	Sample 20578 Dup2
	Saturation Index (unitless)		
Diaspore $[\text{AlO(OH)}]$	-0.27	-0.00	-0.20
Boehmite $[\text{AlO(OH)}]$	-0.68	-0.40	-0.61
Gibbsite $[\text{Al(OH)}_3]$	-0.87	-0.60	-0.80
Hydroxylapatite $[\text{Ca}_5(\text{OH})(\text{PO}_4)_3]$	8.25	-0.14	6.14
Whitlockite $[\text{Ca}_3(\text{PO}_4)_2]$	0.98	<-3.00	-0.40
Calcite $(\text{CaCO}_3)$	1.62	-0.98	1.49
Aragonite $(\text{CaCO}_3)$	1.47	-1.12	1.35
$\text{Na}_2\text{U}_2\text{O}_7$ (am)	-2.30	-2.42	-2.46
$\text{Na}_2\text{U}_2\text{O}_7$ (c)	0.21	0.09	0.05
Plattnerite $[\text{PbO}_4]$	0.93	-0.52	0.70
Bunsenite $[\text{NiO}]$	-0.56	-0.39	-0.22
$\text{Ni(OH)}_2$	-0.84	-0.66	-0.50
SI values calculated with estimated analytical data (below the quantification limit) for at least one constituent are indicated in red.			

Hydroxylapatite is highly oversaturated in samples 20578 and 20578 Dup 2, and near saturation in sample 20578 Dup 1; however, because calcium in sample 20578 Dup 1 was below the quantification limit, this value is associated with greater uncertainty. Whitlockite is near saturation in samples 20578 and 20578 Dup 2 and very undersaturated in sample 20578 Dup1. Taking into account the uncertainty of sample 20578 Dup1 indicates that the solubility of whitlockite may control calcium and phosphate concentrations in the 1-month single-contact  $\text{Ca(OH)}_2$  solution leachates. For the samples with more reliable calcium concentrations (20578 and 20578 Dup 2), both calcite and aragonite are significantly oversaturated.

In contrast to the 1-month single-contact DDI leachates, the crystalline form of sodium uranate  $[\text{Na}_2\text{U}_2\text{O}_7 \text{ (c)}]$  is very close to saturation in the 1-month single-contact  $\text{Ca(OH)}_2$  solution leachates, whereas the amorphous form  $[\text{Na}_2\text{U}_2\text{O}_7 \text{ (am)}]$  is highly undersaturated. Discounting the result for sample 20578 Dup1, plattnerite appears to be somewhat oversaturated in the 1-month single-contact  $\text{Ca(OH)}_2$  solution leachates. Bunsenite is slightly undersaturated in the 1-month single-contact  $\text{Ca(OH)}_2$  solution leachates, while  $\text{Ni(OH)}_2$  is somewhat more undersaturated than bunsenite.

Calculated saturation indices (unitless) for the 1-month single-contact  $\text{CaCO}_3$  solution leachates for the C-108 residual waste samples are shown in Table 3.66. All three aluminum (oxy) hydroxide minerals listed in the table (diaspore, boehmite, and gibbsite) are undersaturated in the 1-month single-contact  $\text{CaCO}_3$  solution leachates. The diaspore is closest to equilibrium; however, the solid phase characterization results did not verify the presence of this mineral. The XRD results did indicate that gibbsite does occur in these samples. It is possible that boehmite and/or diaspore occur at concentrations below the detection limit of XRD.

**Table 3.66.** Calculated Saturation Indices for Relevant Al-, Na-, Ca-,  $\text{PO}_4$ -, and U-Containing Minerals Based on Compositions of 1-Month Single-contact  $\text{CaCO}_3$  Solution Leachates of C-108 Residual Waste Samples

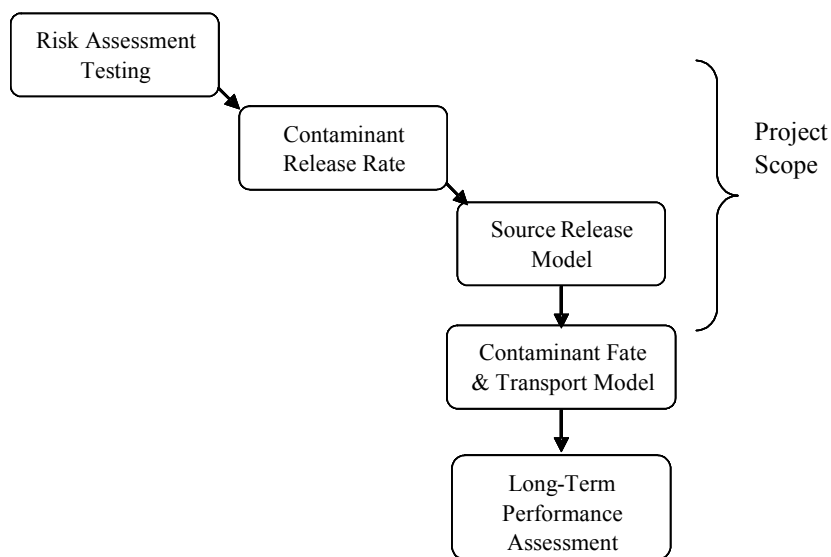
Phase	Sample 20578	Sample 20578 Dup1	Sample 20578 Dup2
	Saturation Index (unitless)		
Diaspore $[\text{AlO}(\text{OH})]$	-0.91	-0.71	-0.69
Boehmite $[\text{AlO}(\text{OH})]$	-1.31	-1.11	-1.09
Gibbsite $[\text{Al}(\text{OH})_3]$	-1.50	-1.31	-1.28
Hydroxylapatite $[\text{Ca}_5(\text{OH})(\text{PO}_4)_3]$	1.35	-0.48	1.49
Whitlockite $[\text{Ca}_3(\text{PO}_4)_2]$	-2.66	<-3.00	-2.58
Calcite ( $\text{CaCO}_3$ )	-1.58	-0.46	-1.49
Aragonite ( $\text{CaCO}_3$ )	-1.72	-0.60	-1.63
$\text{Na}_2\text{U}_2\text{O}_7$ (am)	0.23	0.18	0.45
$\text{Na}_2\text{U}_2\text{O}_7$ (c)	2.74	2.69	2.96
Plattnerite $[\text{PbO}_4]$	0.46	0.33	0.55
Bunsenite $[\text{NiO}]$	-0.76	-0.99	0.45
$\text{Ni}(\text{OH})_2$	-1.04	-1.27	0.18
SI values calculated with estimated analytical data (below the quantification limit) for at least one constituent are indicated in red.			

SI values for hydroxylapatite are variable, with samples 20578 and 20578 Dup 2 being oversaturated, and sample 20578 Dup 1 being slightly undersaturated. Whitlockite is very undersaturated in all the samples. Both calcite and aragonite are significantly undersaturated in all the samples. Meaningful conclusions regarding the saturation status of the calcium containing minerals is not possible as a result of the uncertainties in the calcium concentrations used for the SI calculations.

The amorphous form of sodium uranate  $[\text{Na}_2\text{U}_2\text{O}_7 \text{ (am)}]$  appears to be near to slightly oversaturated in the 1-month single-contact  $\text{CaCO}_3$  solution leachates. The crystalline form  $[\text{Na}_2\text{U}_2\text{O}_7 \text{ (c)}]$  is highly oversaturated. Plattnerite also appears to be near to slightly oversaturated in the 1-month single-contact  $\text{CaCO}_3$  solution leachates. Focusing on sample 20578 Dup2 for the nickel compounds suggests that  $\text{Ni}(\text{OH})_2$  is near saturation in the 1-month single-contact  $\text{CaCO}_3$  solution leachates, whereas bunsenite appears to be slightly oversaturated.

## 4.0 Contaminant Release Models

The primary objective of this project is to develop source release models for contaminants of concern present in residual tank waste. As shown in Figure 4.1, this consists of laboratory testing to produce contaminant release data and development of a conceptual source release model based on the contaminant release and associated solids characterization data. The release model can then be incorporated into a fate and transport model as part of long-term performance assessment for the tanks. This section describes the conceptual release models developed for the primary contaminants of concern ( $^{238}\text{U}$ , Cr, and  $^{99}\text{Tc}$ ) from the laboratory testing of residual waste collected from tank C-108 after modified sluicing.



**Figure 4.1.** Source Release Model Development for Long-Term Performance Assessments

### 4.1 Uranium, Chromium, and $^{99}\text{Tc}$ Release Models

Two tank scenarios have been considered as part of the development of the contaminant release models for retrieved tank C-108. In the first scenario, it is assumed that the tank is filled with a relatively inert material such as quartz ( $\text{SiO}_2$ ) sand or other material that does not significantly impact the chemistry of infiltration water that will contact the post-retrieval sludge. In this case, the composition of water contacting the post-retrieval sludge is assumed to be largely controlled by the solubility of  $\text{CaCO}_3$  (calcite). Calcite is a ubiquitous component of most Hanford Site vadose zone sediments and future-infiltrating water will likely equilibrate with this relatively soluble, reactive mineral.

In the second tank scenario, it is assumed the tanks are filled with a cementitious grout.  $\text{CaO}$  is a major component of cement and readily reacts with water to form portlandite [ $\text{Ca}(\text{OH})_2$ ]. It is assumed that, once the grout sets-up, some portion of its hydration product [ $\text{Ca}(\text{OH})_2$ ], will remain unreacted in the grout, and it will control the pH of the system. During this timeframe, the pH of the leaching solution generated by the grout is expected to be about 12.



As dissolved CO<sub>2</sub> in water contacts the grout, Ca(OH)<sub>2</sub> will react to form CaCO<sub>3</sub> (calcite). After all the available Ca(OH)<sub>2</sub> has been converted to calcite, the grout will be considered to have been aged. At this point, the characteristics of the leaching solution generated by water contacting grout will be largely controlled by the solubility of calcite and the partial pressure of CO<sub>2</sub> gas in the system. If the CO<sub>2</sub> partial pressure is the same as that in the atmosphere, the pH of the solution will be approximately 8.3.

An empirical solubility release model was selected to describe contaminant release for residual sludges in tank C-108. A solubility release model could also be applied for U using the solubility of Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> (am) to control the release concentration of U. CaCO<sub>3</sub> extract compositions are expected to provide the most representative release concentrations of contaminants for the first scenario in which the pore water is in equilibrium with calcite. Ca(OH)<sub>2</sub> extract compositions are expected to provide the most representative release concentrations of contaminants for the first phase of the second scenario (fresh cement) and the CaCO<sub>3</sub> extract compositions are expected to provide the most representative release concentrations of contaminants for the second phase of the second scenario (aged cement/grout in which the Ca(OH)<sub>2</sub> has been converted to CaCO<sub>3</sub>).

The maximum dissolved concentrations measured in the single-contact or sequential contact leaching experiments (Appendix A) as determined by ICP-MS were used to determine the maximum contaminant release concentrations for the release model. These values are provided in Table 4.1. For <sup>238</sup>U, Cr, and <sup>99</sup>Tc, the maximum values measured occurred in either the 1-month single-contact extraction or the first stage of the sequential contact leaching experiments. Total sludge concentrations shown in Table 4.1 were determined from the average of the acid digestion results by ICP-MS (Table 3.3 and Table 3.4).

In Table 4.1, scenario 1 and phase 2 of scenario 2 are indicated as calcite (CaCO<sub>3</sub>). Phase 1 of scenario 2 is indicated as fresh cement [Ca(OH)<sub>2</sub>]. The contaminant concentrations in the tank C-108 residual waste determined for the model are  $6.27 \times 10^{-4}$  µCi/g dry waste, 30.9 µg Cr/g dry waste, and 0.0108 µCi <sup>99</sup>Tc/g dry waste. The release concentration of <sup>238</sup>U for the calcite scenario is  $2.02 \times 10^{-3}$  µCi/L and  $1.06 \times 10^{-3}$  µCi/L for the fresh cement scenario. Cr release concentrations are similar for both scenarios at 291 µg/L for the calcite scenario and 247 µg/L for the fresh cement scenario. Release concentrations for <sup>99</sup>Tc are also similar for both scenarios at 0.115 µCi/L for the calcite scenario and 0.0901 µCi/L for the fresh cement scenario.

**Table 4.1.** Residual Waste and Maximum Contaminant Release Concentrations for the Tank C-108 Release Model

Release Scenario	Component	Waste Conc.	Max. Release Conc.
Calcite [CaCO <sub>3</sub> ]	U	$6.27 \times 10^{-4}$ µCi/g Dry Waste	$2.02 \times 10^{-3}$ µCi/L
Fresh cement [Ca(OH) <sub>2</sub> ]	U	$6.27 \times 10^{-4}$ µCi/g Dry Waste	$1.06 \times 10^{-3}$ µCi/L
Calcite [CaCO <sub>3</sub> ]	Cr	(30.9 µg/g Dry Waste)	291 µg/L
Fresh cement [Ca(OH) <sub>2</sub> ]	Cr	(30.9 µg/g Dry Waste)	247 µg/L
Calcite [CaCO <sub>3</sub> ]	<sup>99</sup> Tc	0.0108 µCi/g Dry Waste	0.115 µCi/L
Fresh cement [Ca(OH) <sub>2</sub> ]	<sup>99</sup> Tc	0.0108 µCi/g Dry Waste	0.0901 µCi/L

## 5.0 Discussion

The following discussion is associated with the results of laboratory characterization, testing, and analysis of a post tank C-108 sluicing composite sample (designated 20578). It is recognized that this residual waste sample was collected from tank C-108 remaining waste that could not be mobilized by sluicing forces and insoluble in the tank 241-AN-106 supernatant. Further retrieval technologies and methodologies will likely be deployed in tank C-108 to remove as much of the remaining 6,800 gallons residual waste as practical. As a result of these circumstances, the sample analyzed here is not expected to represent the final retrieval sample. The studies described here were completed to characterize the as sampled residual waste, assess the leachability of contaminants from the solids, and develop release models for contaminants of interest based on this interim waste sample (designated 20578). Should further waste retrieval operations take place at tank C-108, information assembled from this interim residual waste sample will be useful in assessing waste composition changes resulting from secondary waste retrieval technologies. Total elemental analysis using the U.S. Environmental Protection Agency (EPA) acid digestion method, indicates that tank C-108 partial retrieval residual waste (sample 20578) is composed of the following major elements Na at 15.5%, Al at 12.3%, P at 4.0%, Fe at 1.6%, Ni at 0.22%, U at 0.18%, and Mn at 0.13%. Concentrations of  $^{99}\text{Tc}$  and  $^{238}\text{U}$  were determined to be 0.636  $\mu\text{g/g}$  waste (0.0108  $\mu\text{Ci/g}$ ) and 1870  $\mu\text{g/g}$  waste ( $6.27 \times 10^{-4}$   $\mu\text{Ci/g}$ ). Average concentrations of  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  were determined to be  $1.24 \times 10^{-4}$   $\mu\text{Ci/g}$  ( $^{237}\text{Np}$ ),  $2.90 \times 10^{-2}$   $\mu\text{Ci/g}$  ( $^{239}\text{Pu}$ ), and not detected ( $^{241}\text{Am}$ ). The concentrations of  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  were found at significant concentrations in tank C-108 residual waste, with averages of  $1.69 \times 10^4$   $\mu\text{Ci/g}$  ( $^{137}\text{Cs}$ ) and  $1.22 \times 10^5$   $\mu\text{Ci/g}$  ( $^{90}\text{Sr}$ ). The results determined from the fusion dissolution procedure were generally very similar to those determined with the EPA acid digestion method.

Solid phase characterization by x-ray diffraction (XRD) indicated the presence of the following phases in the as-received tank C-108 composite sample: gibbsite [ $\text{Al}(\text{OH})_3$ ], natrophosphate [ $\text{Na}_7(\text{PO}_4)_2\text{F} \cdot 19\text{H}_2\text{O}$ ], sodium fluoride phosphate hydrate [ $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$ ], sodium phosphate ( $\text{Na}_3\text{PO}_4$ ), nickel oxide hydroxide ( $\text{NiOOH}$ ), and nitratine ( $\text{NaNO}_3$ ). Solid phase characterization by scanning electron microscopy/energy dispersive spectrometry (SEM/EDS) analyses indicated the majority of the particles in the as-received tank C-108 composite sample are composed of Al-Na-O-P-F, Al-Na-O-P, Na-O-P-F, Na-O-P, Al-Na-O, and Na-O, all in variable proportions. Note that all phases could also potentially contain H and C, which cannot be quantified by EDS. Combining the XRD with the SEM/EDS results, the following conclusions can be made regarding the presence of solid phases in the tank C-108 residual waste composite sample:

- Gibbsite was the major phase present and appears to be coated and mixed with variable amounts of phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate
- Fe-oxides occurred as a minor phase. Its particles were coated and mixed with variable amounts of other solid phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate
- Minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu, were associated with the Fe-oxides
- Most likely the Ni occurred as a separate phase ( $\text{NiOOH}$ ) coating the iron oxide

- Small amounts of a U–Na–O phase(s), most likely čejkaite and/or sodium uranate or similar phase were also present
- Sr was occasionally associated with a Na–O–P phase, possibly as NaSrPO<sub>4</sub> or another similar phase.

It is also noteworthy that particles composed of U–Na–O had morphologies that were very similar to particles that were identified as čejkaite in a previous study conducted with samples of unleached tank C-203 preretrieval waste (Deutsch et al. 2007).

Analysis of the tank C-108 solids that remained after leaching with DDI and calcite (CaCO<sub>3</sub>) saturated DDI water indicated that the majority of the more soluble phases such as natrophosphate, sodium fluoride phosphate hydrate, sodium phosphate, sodium carbonate, sodium hydroxide, and sodium nitrate were largely dissolved away, leaving relatively clean gibbsite and Fe oxides particles with only minor amounts of surface coatings. The elemental compositions of many of the particles analyzed are close to the composition of pure gibbsite. As was the case for the unleached samples, the Fe-oxides often hosted minor amounts of Ni and traces of other metals such as Pb, Zn, and Cu. Particles containing Sr with an elemental composition of SrNaPO<sub>4</sub> were also occasionally identified in the DDI water and CaCO<sub>3</sub> saturated DDI water leached samples.

The solids that were leached with Ca(OH)<sub>2</sub> saturated DDI water were quite different than the DDI water and CaCO<sub>3</sub> saturated DDI leached samples. The surfaces of these particles were coated with precipitated materials and desiccation cracks were evident. XRD indicated only the presence of gibbsite. The SEM/EDS results suggested the following phases are likely present:

- Gibbsite as the dominant phase with coatings of one or more phases with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) and Ca(OH)<sub>2</sub>
- Lesser amounts of Fe-oxide(s) with coatings of one or more phase(s) with the general composition of (Na, Ca)PO<sub>4</sub>(F,OH) and Ca(OH)<sub>2</sub>
- Ni and traces of other metals such as Pb, Zn, and Cu which were often associated with the Fe-oxide(s).

Thermodynamic modeling suggested that the DDI and CaCO<sub>3</sub> saturated leachate solutions were close to saturation with respect to Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> (am), while the Ca(OH)<sub>2</sub> saturated leachate solutions were near equilibrium with respect to Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> (c). These results suggest that any čejkaite that was present in the as-received sample from tank C-108 residual waste was converted to these less soluble phases as a result of contact with the relatively dilute leachate solutions. The leachate solutions were also determined to be significantly undersaturated with respect to gibbsite [Al(OH)<sub>3</sub>], and other Al oxyhydroxides. This is likely due to slow dissolution kinetics of these phases. Additional useful saturation index results were limited due to analytical results that were below the quantification limit.

The leachability of <sup>99</sup>Tc and <sup>238</sup>U was relatively high in the tank C-108 residual waste. For the 1-month single-contact DDI leachants, the average leachable <sup>99</sup>Tc concentration was 0.506 µg/g (0.00860 µCi/g) and the average leachable <sup>238</sup>U concentration was 825 µg/g (2.77 × 10<sup>-4</sup> µCi/g). These values equate to an average of 80% (<sup>99</sup>Tc) and 44% (<sup>238</sup>U) compared to the totals determined by EPA acid digestion. For the 1-month single-contact Ca(OH)<sub>2</sub> leachants, the average leachable <sup>99</sup>Tc concentration was 0.522 µg/g (0.00887 µCi/g) and the average leachable <sup>238</sup>U concentration was 52.1 µg/g (1.75 × 10<sup>-5</sup> µCi/g). These values are an average of 82% (<sup>99</sup>Tc) and 2.8% (<sup>238</sup>U) of the totals. Compared to the leachable concentrations of <sup>99</sup>Tc and <sup>238</sup>U determined in the 1-month DDI water leachates, the percent

leachable  $^{99}\text{Tc}$  is about the same, whereas the percent leachable  $^{238}\text{U}$  is dramatically lower. This suggests the tank C-108 residual waste in contact with  $\text{Ca}(\text{OH})_2$  leachant may result in the formation of a Ca-U phase with a lower solubility such as becquerelite  $[\text{Ca}(\text{UO}_2)_6\text{O}_4(\text{OH})_6(\text{H}_2\text{O})_8]$ . For the 1-month single-contact  $\text{CaCO}_3$  leachants, the average leachable  $^{99}\text{Tc}$  concentration was  $0.627 \mu\text{g/g}$  ( $0.0107 \mu\text{Ci/g}$ ) and the average leachable  $^{238}\text{U}$  concentration was  $648 \mu\text{g/g}$  ( $2.18 \times 10^{-4} \mu\text{Ci/g}$ ). These values equate to an average of 99% ( $^{99}\text{Tc}$ ) and 35% ( $^{238}\text{U}$ ) compared to the totals determined by EPA acid digestion. These values are similar to those of the DDI leachants.

## 6.0 References

10 CFR 830.120. "Nuclear Safety Management; Quality Assurance Requirements." *Code of Federal Regulations*, U.S. Department of Energy.

Agnew SF. 1993. *Analysis of the History of 241-C Farm*. LA-UR-93-3605, Rev. 0, Los Alamos National Laboratory, Los Alamos, New Mexico.

Agnew SF. 1997. *Hanford Tank Chemical and Radionuclide Inventories*. LA-UR-96-3860, Los Alamos National Laboratory, Los Alamos, New Mexico.

ASTM. 1998. *D2216-98 Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*. American Society for Testing and Materials, West Conshohocken, Pennsylvania.

ASTM. 1999. *D3987-85 Standard Test Method for Shake Extraction of Solid Waste with Water*. American Society for Testing and Materials, West Conshohocken, Pennsylvania.

Bethke CM and S Yeakel. 2009. *The Geochemist's Workbench<sup>®</sup>, Release 8.0, Reference Manual*. Hydrogeology Program, University of Illinois, Urbana, Illinois.

Brevick CH, JL Stroup, and JW Funk. 1997. *Supporting Document for the Historical Tank Content Estimate for C Tank Farm*. WHC-SD-WM-ER-313, Rev. 1B, Fluor Daniel Hanford, Inc., Richland, Washington.

Brown CF, MM Valenta, RJ Serne, BN Bjornstad, DC Lanigan, C Iovin, RE Clayton, KN Geiszler, ET Clayton, IV Kutnyakov, SR Baum, MJ Lindberg, and RD Orr. 2007. *Characterization of Direct Push Vadose Zone Sediments from the T and TY Waste Management Areas*. PNNL-16649, Pacific Northwest National Laboratory, Richland, Washington.

Bushaw RC. 2009. *Final Report for Tank 241-C-108 Soil Samples Taken After Modified Sluicing*. RPP-RPT-43234, Rev. 0, Washington River Protection Solutions, LLC, Richland, Washington.

Byrnes IB. 2009. *Single-Shell Tank 241-C-108 Hard Heel Retrieval Technology Selection*. RPP-Plan-43858, Rev. 0, Washington River Protection Solutions, LLC, Richland, Washington.

Cantrell KJ, KM Krupka, WJ Deutsch, MJ Lindberg, HT Schaef, KN Geiszler and BW Arey. 2008a. *Hanford Tank 241-C-103 Residual Waste Contaminant Release Models and Supporting Data*. PNNL-16738, Pacific Northwest National Laboratory, Richland, Washington.

Cantrell KJ, KM Krupka, KN Geiszler, MJ Lindberg, BW Arey, and HT Schaef. 2008b. *Hanford Tank 241-S-112 Residual Waste Composition and Leach Test Data*. PNNL-17593, Pacific Northwest National Laboratory, Richland, Washington.

Chen F, RC Ewing, and SB Clark. 1999. "The Gibbs Free Energies and Enthalpies of Formation of U<sup>6+</sup> Phases: An Empirical Method of Prediction." *American Mineralogist* 84(4):650-664.

Clesceri LS, AE Greenberg, and AD Eaton. 1998. *Standard Methods for the Examination of Water and Wastewater*, 20th Edition. Standard Method 2320 B, “Alkalinity by Titration.” American Public Health Association, American Water Works Association, and Water Environment Federation, Washington, D.C.

De Lorenzo DS, AT DiCenso, DB Hiller, KW Johnson, JH Rutherford, DJ Smith, and BC Simpson. 1994. *Tank Characterization Reference Guide*. WHC-SD-WM-TI-648, Rev. 0, prepared for Westinghouse Hanford Company by Los Alamos Technical Associates, Kennewick, Washington.

Deutsch WJ, KM Krupka MJ Lindberg, KJ Cantrell, CF Brown, and HT Schaef. 2006. *Hanford Tanks 241-C-106: Impact of Cement Reactions on Release of Contaminant from Residual Waste*. PNNL-15544, Pacific Northwest National Laboratory, Richland, Washington.

Deutsch WJ, KM Krupka MJ Lindberg, KJ Cantrell, CF Brown, and HT Schaef. 2007a. *Hanford Tanks 241-C-106: Residual Waste Contaminant Release Model and Supporting Data*. PNNL-15187, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

Deutsch WJ, KM Krupka, MJ Lindberg, KJ Cantrell, CF Brown, and HT Schaef. 2007b. *Hanford Tanks 241-C-203 and 241-C-204: Residual Waste Contaminant Release Model and Supporting Data*. PNNL-14903, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

Deutsch WJ, KM Krupka MJ Lindberg, KJ Cantrell, CF Brown, SV Mattigod, HT Schaef, and BW Arey. 2007c. *Hanford Tanks 241-C-202 and 241-C-203 Residual Waste Contaminant Release Models and Supporting Data*. PNNL-16229, Pacific Northwest National Laboratory, Richland, Washington.

DOE-RL. 2007. *Hanford Analytical Services Quality Assurance Requirements Documents*. DOE/RL-96-68 Rev. 3, HASQARD, Volumes 1, 2, 3, and 4. U.S. Department of Energy, Richland Operations Office, Richland, Washington.

DOE Order 414.1C. 2005. “Quality Assurance.” U.S. Department of Energy, Washington, D.C.

Ecology, EPA, and DOE. 1996. *Hanford Federal Facility Agreement and Consent Order*, Milestone M-45-03C. Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

EPA. 1996. “Method 3052. Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices,” Rev. 0. *Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods*. EPA SW-846, Third Ed., 3000 series, pp. 3052-1–3052-20. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. Accessed May 14, 2010, at [http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/3\\_series.htm](http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/3_series.htm).

EPA. 2004. “Method 9040C, pH Electrometric Measurement” Rev. 3. In *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. EPA SW-846, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. Accessed May 14, 2010, at [http://www.epa.gov/sw-846/9\\_series.htm](http://www.epa.gov/sw-846/9_series.htm).

EPA. 2007. "Method 9056A, Determination of Inorganic Anions by Ion Chromatography," Rev. 1. In *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. EPA SW-846, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. Accessed May 14, 2010, at [http://www.epa.gov/sw-846/9\\_series.htm](http://www.epa.gov/sw-846/9_series.htm).

Felmy AR, Y Xia, and Z Wang. 2005. "The Solubility Product of  $\text{NaUO}_2\text{PO}_4 \cdot x\text{H}_2\text{O}$  Determined in Phosphate and Carbonate Solutions." *Radiochimica Acta* 93(7):401-408.

Fiskum SK, CJ Barinaga, JP Bramson, KJ Carson, and JR DesChane. 2000. *Inorganic and Radiochemical Analysis of 241-C-104 Tank Waste*. PNNL-13364 (WTP-RPT-007-Rev. 0) (formerly BNFL-RPT-043), Pacific Northwest National Laboratory, Richland, Washington.

Gorman-Lewis D, T Shvareva, K Kubatko, PC Burns, DM Wellman, B McNamara, JES Szymanowski, A Navrotsky, and JB Fein. 2009. "Thermodynamic Properties of Autunite, Uranyl Hydrogen Phosphate, and Uranyl Orthophosphate from Solubility and Calorimetric Measurements." *Environmental Science and Technology* 43:7416-7422.

Kalmykov S and GR Choppin. 2000. "Mixed  $\text{Ca}^{2+}/\text{UO}_2^{2+}/\text{CO}_3^{2-}$  Complex Formation at Different Ionic Strengths." *Radiochimica Acta* 88(9-11):603-606.

Krupka KM, WJ Deutsch, MJ Lindberg, KJ Cantrell, NJ Hess, HT Schaef, and BW Arey. 2004. *Hanford Tanks 241-AY-102 and 241-BX-101: Sludge Composition and Contaminant Release Data*. PNNL-14614, Pacific Northwest National Laboratory, Richland, Washington.

Lindberg MJ and WJ Deutsch. 2003. *Tank 241-AY-102 Data Report*. PNNL-14344, Pacific Northwest National Laboratory, Richland, Washington.

NIST. 2005. *Standard Reference Material 676, Alumina Internal Standard for Quantitative Analysis by X-ray Powder Diffraction*. National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, Maryland.

Parker DL and WB Barton. 2007. *Retrieval Data Report for Single-Shell Tank 241-S-112*. RPP-RPT-35112, CH2M HILL Hanford Group, Inc., Richland, Washington.

Rai D, AR Felmy, NJ Hess, VL LeGore, and DE McCready. 2002. "Thermodynamics of the  $\text{U(VI)}\text{-Ca}^{2+}\text{-Cl}^-\text{-OH}^-\text{-H}_2\text{O}$  System: Solubility Product of Becquerelite." *Radiochimica Acta* 90:495-503.

RPP. 2007. *Retrieval Data Report for Single-Shell Tank 241-C-103*. RPP-RPT-33060, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.

Simpson BC. 1994. *Tank 241-T-111 Characterization Report*. WHC-EP-0806, Westinghouse Hanford Company, Richland, Washington.

Smith GL, DJ Bates, RW Goles, LR Greenwood, RC Lettau, GF Piepel, MJ Schweiger, HD Smith, MW Urie, and JJ Wagner. 2001. *Vitrification and Product Testing of C-104 and AZ-102 Pretreated Sludge Mixed with Flowsheet Quantities of Secondary Wastes*. PNNL-13452, Pacific Northwest National Laboratory, Richland, Washington.

Strachan DM, HT Schaef, MJ Schweiger, KL Simmons, LJ Woodcock, and MK Krouse. 2003. "A Versatile and Inexpensive XRD Specimen Holder for Highly Radioactive or Hazardous Specimens." *Powder Diffraction* 18(1):23-28.

Weber CF, EC Beahm, DD Lee, and JS Watson. 2000. "A Solubility Model for Aqueous Solutions Containing Sodium, Fluoride, and Phosphate Ions." *Industrial & Engineering Chemistry Research* 39:518-526.

Yamamura T, A Kidtamura, A Fukui, S Nishikawa, T Yamamoto, and H Moriyama. 1998. "Solubility of U(VI) in Highly Basic Solutions." *Radiochimica Acta* 83:139-146.



## **Appendix A**

### **Leach Test Results – Solution Concentrations**

## Appendix A

### Leach Test Results – Solution Concentrations

This section provides results of leach test results on a solution concentration basis. Data in this form are used for the geochemical modeling in Section 3.7 and to develop contaminant release models (Section 4.0).

#### A.1 Double-Deionized Water-Leach Tests

##### A.1.1 One-Month Single-Contact Double-Deionized Water-Leach Tests

**Table A.1.** pH and Alkalinity Values for Single-Contact Double-Deionized Water Leachants

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g dry waste
20578	12.77	7.45E+06	1.00E+06
20578 Dup1	12.75	7.75E+06	9.42E+05
20578 Dup2	12.69	7.35E+06	8.43E+05
20578 Average	12.74	7.52E+06	9.28E+05
Dup = Duplicate.			

**Table A.2.** Dissolved <sup>99</sup>Tc and <sup>238</sup>U Concentrations in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	µg/L		µCi/L	
20578	2.93E+00	5.25E+03	4.99E-02	1.76E-03
20578 Dup1	4.27E+00	8.65E+03	7.26E-02	2.91E-03
20578 Dup2	5.27E+00	6.27E+03	8.96E-02	2.11E-03
20578 Average	4.16E+00	6.72E+03	7.07E-02	2.26E-03
Dup = Duplicate.				

**Table A.3.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578	7.85E-04	3.00E-04	6.83E-02	4.20E-02
20578 Dup1	4.92E-04	3.25E-04	4.28E-02	4.55E-02
20578 Dup2	5.61E-04	2.59E-04	4.88E-02	3.63E-02
20578 Average	6.12E-04	2.95E-04	5.33E-02	4.13E-02
Dup = Duplicate.				

**Table A.4.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578	2.41E-04	0.3394	5.78E-02	0.9315	(1.08E-01)	(0.0319)
20578 Dup1	4.47E-04	0.6298	1.05E-01	1.6975	(1.26E-01)	(0.0370)
20578 Dup2	2.94E-04	0.4137	7.15E-02	1.1528	(1.21E-01)	(0.0355)
20578 Average	3.27E-04	0.4610	7.82E-02	1.2606	(1.18E-01)	(0.0348)
Concentrations listed in parentheses were <EQL.						
Dup = Duplicate.						
EQL = Estimated quantitation limit.						

**Table A.5.** Dissolved Metal Concentrations Determined by ICP-OES in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Sample Number	Al	Bi	Ca	Cr	Fe	Ni	P
	$\mu\text{g/L}$						
20578	6.31E+03	ND	(2.40E+02)	1.17E+02	3.10E+02	(8.92E+00)	4.46E+05
20578 Dup1	5.66E+03	ND	(4.47E+02)	1.65E+02	3.23E+02	(2.89E+01)	3.85E+05
20578 Dup2	6.80E+03	ND	(1.56E+02)	2.11E+02	2.39E+02	(9.10E+01)	3.92E+05
20578 Average	6.26E+03	ND	(2.81E+02)	1.65E+02	2.90E+02	(4.29E+01)	4.08E+05

Sample Number	K	Si	Na	Sr	S	U
	$\mu\text{g/L}$					
20578	(1.41E+03)	3.03E+03	1.37E+06	(1.18E+01)	4.19E+03	5.38E+03
20578 Dup1	(1.82E+03)	1.90E+03	1.35E+06	(1.63E+01)	5.33E+03	1.05E+04
20578 Dup2	(2.00E+03)	2.25E+03	1.36E+06	(1.98E+01)	6.24E+03	6.64E+03
20578 1 Month Avg	(1.74E+03)	2.39E+03	1.36E+06	(1.60E+01)	5.26E+03	7.50E+03
Concentrations listed in parentheses were <EQL.						
Avg = Average.						
Dup = Duplicate.						
EQL = Estimated quantitation limit.						
ND = Instrument returned a zero or negative response.						

**Table A.6.** Dissolved Metal Concentrations Determined by ICP-MS in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/L					
20578	1.29E+02	1.37E+02	(6.18E+01)	ND	(5.47E+01)	ND
20578 Dup1	1.53E+02	1.43E+02	(5.80E+01)	ND	(3.38E+01)	ND
20578 Dup2	1.96E+02	1.94E+02	(4.93E+01)	ND	(4.77E+01)	ND
20578 Avg	1.59E+02	1.58E+02	(5.63E+01)	ND	(4.54E+01)	ND

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
20578	(2.72E+01)	(5.12E+01)	(1.96E+01)	1.06E+02	4.83E+01	4.55E+01
20578 Dup1	(3.74E+01)	(5.27E+01)	(1.98E+01)	1.48E+02	6.77E+01	6.30E+01
20578 Dup2	(4.83E+01)	6.43E+01	(2.67E+01)	1.68E+02	7.93E+01	6.96E+01
20578 Avg	(3.76E+01)	6.43E+01	(2.20E+01)	1.40E+02	6.51E+01	5.94E+01

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Sample Number	µg/L						
20578	(1.11E+01)	(1.17E+01)	ND	ND	ND	3.03E+01	3.04E+01
20578 Dup1	(8.91E+00)	(9.30E+00)	ND	ND	ND	5.03E+01	5.79E+01
20578 Dup2	(9.13E+00)	(9.39E+00)	ND	ND	ND	(2.48E+01)	3.06E+01
20578 Average	(9.71E+00)	(1.01E+01)	ND	ND	ND	4.03E+01	3.96E+01

(a) ICP-MS mass of choice.  
Concentrations listed in parentheses were <EQL.  
Avg = Average  
Dup = Duplicate.  
EQL = Estimated quantitation limit.  
ICP-MS = Inductively coupled plasma mass spectrometer.  
ND = Instrument returned a zero or negative response.

**Table A.7.** Dissolved Anion Concentrations in Single-Contact Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
Sample Number	µg/L					
20578	1.45E+02	(5.80E+03)	(9.26E+04)	(6.71E+04)	(1.57E+04)	1.40E+06
20578 Dup1	1.21E+02	(7.30E+03)	1.19E+05	1.01E+05	(2.08E+04)	1.17E+06
20578 Dup2	1.26E+02	(6.60E+03)	1.44E+05	1.22E+05	(2.12E+04)	1.23E+06
20578 Average	1.31E+05	(6.57E+03)	1.32E+05	1.11E+05	(1.92E+04)	1.27E+06

Concentrations listed in parentheses were <EQL.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.

### A.1.2 Periodic Replenishment Double-Deionized Water-Leach Tests

**Table A.8.** pH and Alkalinity Values for Periodic Replenishment Double-Deionized Water Leachants

Contact Stage	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g Dry Waste
20578 Stage 1	8.93	8.02E+06	1.08E+06
20578 Dup1 Stage 1	12.74	9.61E+06	1.22E+06
20578 Dup2 Stage 1	12.71	1.03E+07	9.52E+05
20578 Stage 2	7.27	1.40E+05	1.89E+04
20578 Dup1 Stage 2	7.63	1.48E+05	1.89E+04
20578 Dup2 Stage 2	9.24	4.33E+05	4.01E+04
20578 Stage 3	6.83	6.18E+04	8.36E+03
20578 Dup1 Stage 3	7.75	7.00E+04	8.92E+03
20578 Dup2 Stage 3	7.53	1.11E+05	1.03E+04
20578 Stage 4	6.55	4.94E+04	6.69E+03
20578 Dup1 Stage 4	6.67	4.53E+04	5.77E+03
20578 Dup2 Stage 4	9.55	4.94E+04	4.58E+03
20578 Stage 5	6.20	(9.06E+03)	(1.23E+03)
20578 Dup1 Stage 5	6.05	(9.06E+03)	(1.61E+01)
20578 Dup2 Stage 5	6.81	(9.89E+03)	(1.76E+01)

Concentrations listed in parentheses were <EQL.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.

**Table A.9.** Dissolved  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578 Stage 1	2.40E+00	5.69E+03	4.08E-02	1.91E-03
20578 Dup1 Stage 1	1.84E+00	3.92E+03	3.12E-02	1.32E-03
20578 Dup2 Stage 1	4.62E+00	8.96E+03	7.85E-02	3.01E-03
20578 Stage 2	1.56E-01	2.95E+01	2.66E-03	9.93E-06
20578 Dup1 Stage 2	9.96E-02	2.98E+01	1.69E-03	1.00E-05
20578 Dup2 Stage 2	1.85E-01	4.52E+01	3.15E-03	1.52E-05
20578 Stage 3	6.22E-03	2.65E+00	1.06E-04	8.89E-07
20578 Dup1 Stage 3	5.07E-03	2.55E+00	8.62E-05	8.56E-07
20578 Dup2 Stage 3	4.42E-02	3.95E+01	7.52E-04	1.33E-05
20578 Stage 4	ND	4.63E-01	ND	1.55E-07
20578 Dup1 Stage 4	1.04E-02	7.84E-01	1.78E-04	2.64E-07
20578 Dup2 Stage 4	ND	5.96E+00	ND	2.00E-06
20578 Stage 5	3.13E-01	9.17E-01	5.32E-03	3.08E-07
20578 Dup1 Stage 5	4.58E-01	1.32E+00	7.78E-03	4.43E-07
20578 Dup2 Stage 5	3.81E-01	2.59E+00	6.48E-03	8.69E-07

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

#### A.1.2.1 Elements $^{137}\text{Cs}$ and $^{90}\text{Sr}$ – Periodic Replenishment Double-Deionized Water Leachants

**Table A.10.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578 Stage 1	7.01E-04	8.71E-04	6.10E-02	1.22E-01
20578 Dup1 Stage 1	4.12E-04	3.81E-04	3.58E-02	5.34E-02
20578 Dup2 Stage 1	3.57E-04	3.59E-04	3.11E-02	5.03E-02
20578 Stage 2	ND	2.23E-03	ND	3.12E-01
20578 Dup1 Stage 2	ND	1.49E-03	ND	2.09E-01
20578 Dup2 Stage 2	ND	7.79E-04	ND	1.09E-01
20578 Stage 3	ND	1.84E-03	ND	2.58E-01
20578 Dup1 Stage 3	ND	1.56E-03	ND	2.18E-01
20578 Dup2 Stage 3	ND	2.26E-03	ND	3.16E-01
20578 Stage 4	ND	1.15E-03	ND	1.61E-01
20578 Dup1 Stage 4	ND	3.89E-04	ND	5.44E-02
20578 Dup2 Stage 4	(1.58E-04)	2.46E-03	(1.37E-02)	3.45E-01
20578 Stage 5	ND	1.44E-04	ND	2.01E-02
20578 Dup1 Stage 5	ND	3.56E-05	ND	4.98E-03
20578 Dup2 Stage 5	ND	8.71E-04	ND	1.22E-01

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

**Table A.11.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578 Stage 1	2.55E-04	0.3598	6.05E-02	0.9750	1.64E-01	0.0482
20578 Dup1 Stage 1	1.78E-04	0.2511	4.00E-02	0.6459	1.69E-01	0.0498
20578 Dup2 Stage 1	4.04E-04	0.5685	1.03E-01	1.6622	1.55E-01	0.0457
20578 Stage 2	ND	ND	(9.47E-05)	(0.0015)	ND	ND
20578 Dup1 Stage 2	ND	ND	(5.58E-04)	(0.0090)	ND	ND
20578 Dup2 Stage 2	(4.04E-06)	(0.0057)	(5.48E-04)	(0.0088)	ND	ND
20578 Stage 3	(7.99E-06)	(0.0112)	(1.15E-03)	(0.0185)	ND	ND
20578 Dup1 Stage 3	(1.24E-05)	(0.0175)	(5.64E-04)	(0.0091)	ND	ND
20578 Dup2 Stage 3	(1.05E-05)	(0.0148)	(1.24E-03)	(0.0201)	ND	ND
20578 Stage 4	4.15E-05	0.0585	3.01E-03	0.0486	ND	ND
20578 Dup1 Stage 4	3.25E-05	0.0457	(2.02E-03)	(0.0326)	(1.19E-02)	(0.0035)
20578 Dup2 Stage 4	(7.30E-06)	(0.0103)	(8.79E-04)	(0.0142)	ND	ND
20578 Stage 5	(1.28E-05)	(0.0180)	(1.41E-03)	(0.0228)	ND	ND
20578 Dup1 Stage 5	(2.85E-06)	(0.0040)	ND	ND	ND	ND
20578 Dup2 Stage 5	2.03E-05	0.0286	(1.45E-03)	(0.0234)	ND	ND

Values within parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

**Table A.12.** Dissolved Metal Concentrations Determined by ICP-OES in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	Al	Bi	Ca	Cr	Fe	Ni	P
Contact Stage	µg/L						
20578 Stage 1	3.70E+03	ND	(9.87E+01)	1.04E+02	3.32E+02	ND	4.74E+05
20578 Dup1 Stage 1	2.39E+03	ND	ND	7.72E+01	2.03E+02	ND	6.62E+05
20578 Dup2 Stage 1	6.11E+03	ND	(1.10E+02)	2.00E+02	4.81E+02	(2.26E-01)	6.15E+05
20578 Stage 2	5.84E+02	ND	(2.29E+01)	ND	(1.26E+02)	ND	3.38E+03
20578 Dup1 Stage 2	5.96E+02	ND	(3.83E+01)	ND	(6.57E+01)	ND	5.16E+03
20578 Dup2 Stage 2	2.40E+03	ND	(1.32E+02)	ND	2.06E+02	ND	1.65E+04
20578 Stage 3	(5.00E+01)	(3.44E+01)	(8.44E+01)	ND	(1.93E+01)	ND	(4.25E+02)
20578 Dup1 Stage 3	(9.51E+00)	ND	(6.67E+01)	ND	ND	ND	1.15E+03
20578 Dup2 Stage 3	8.47E+02	ND	(1.66E+02)	ND	2.43E+02	(3.08E+01)	1.82E+03
20578 Stage 4	(3.14E+01)	ND	(5.74E+02)	ND	(1.16E+01)	(3.04E+01)	(1.07E+02)
20578 Dup1 Stage 4	(7.05E+00)	ND	(5.91E+01)	ND	ND	ND	(1.47E+02)
20578 Dup2 Stage 4	2.36E+02	ND	(1.65E+02)	ND	(7.74E+01)	ND	(4.11E+02)
20578 Stage 5	(2.69E+00)	ND	(9.48E+01)	(2.82E+00)	(9.16E+00)	(1.33E+01)	(4.41E+01)
20578 Dup1 Stage 5	ND	ND	(1.68E+01)	(2.32E+00)	(2.86E-01)	(2.26E+00)	(8.85E+01)
20578 Dup2 Stage 5	(6.15E+01)	ND	(1.88E+02)	(1.87E+00)	(3.60E+00)	(5.90E+01)	(1.18E+02)

	K	Si	Na	Sr	S	U
Contact Stage	µg/L					
20578 Stage 1	(1.31E+03)	8.87E+02	1.41E+06	(1.12E+01)	4.06E+03	6.11E+03
20578 Dup1 Stage 1	(9.46E+02)	9.21E+02	1.82E+06	(3.29E+00)	(3.03E+03)	3.79E+03
20578 Dup2 Stage 1	(2.12E+03)	(6.63E+02)	1.91E+06	(6.31E+00)	5.85E+03	1.02E+04
20578 Stage 2	(2.01E+02)	9.42E+02	1.25E+04	6.65E+02	(2.26E+02)	(7.48E+01)
20578 Dup1 Stage 2	(2.94E+02)	(4.67E+02)	1.40E+04	1.13E+03	(1.21E+02)	(5.88E+01)
20578 Dup2 Stage 2	(2.77E+02)	1.78E+03	4.97E+04	3.25E+03	(2.46E+02)	(9.73E+01)
20578 Stage 3	(2.45E+02)	(6.40E+02)	2.40E+03	9.45E+02	(1.84E+02)	(4.75E+01)
20578 Dup1 Stage 3	(2.72E+02)	(1.94E+02)	2.76E+03	1.25E+03	(2.03E+02)	(3.53E+01)
20578 Dup2 Stage 3	(1.37E+02)	(7.23E+02)	6.34E+03	2.03E+03	(1.80E+02)	(8.44E+01)
20578 Stage 4	(1.76E+02)	(1.89E+02)	6.07E+02	5.07E+02	(1.24E+02)	(1.97E+01)
20578 Dup1 Stage 4	(3.77E+02)	(3.47E+00)	3.38E+02	1.82E+02	(2.13E+02)	(3.46E+01)
20578 Dup2 Stage 4	(1.89E+02)	(6.52E+02)	1.28E+03	1.86E+03	(2.22E+02)	(2.80E+01)
20578 Stage 5	(1.18E+02)	(5.22E+01)	1.93E+02	(5.23E+01)	(5.43E+01)	(5.72E+01)
20578 Dup1 Stage 5	(7.53E+01)	(8.51E-01)	1.38E+02	(6.15E-02)	(8.90E+01)	(3.42E+01)
20578 Dup2 Stage 5	(6.54E+01)	(4.23E+02)	4.56E+02	1.36E+03	ND	(3.94E+01)

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.



**Table A.13.** Dissolved Metal Concentrations Determined by ICP-MS in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/L					
S1	8.19E+01	8.34E+01	4.89E+01	(4.78E+00)	2.40E+01	ND
S1DUP1	5.55E+01	5.86E+01	6.91E+01	(7.27E+00)	3.43E+01	ND
S1DUP2	1.28E+02	1.37E+02	6.39E+01	(8.56E+00)	4.29E+01	(8.67E-01)
S2	(1.30E+00)	(1.36E+00)	(1.47E+00)	(7.56E-01)	ND	ND
S2DUP1	(7.77E-01)	(5.17E-01)	(7.78E-01)	ND	ND	ND
S2DUP2	5.24E+00	5.77E+00	(4.12E+00)	(2.16E+00)	ND	ND
S3	(8.59E-01)	(8.84E-01)	(1.69E+00)	(1.06E+00)	ND	ND
S3DUP1	(4.31E-02)	(1.97E-01)	(1.13E+00)	(5.59E-01)	ND	ND
S3DUP2	(1.22E+00)	(1.42E+00)	(2.39E+00)	(1.82E+00)	ND	ND
S4	(4.60E-01)	(3.28E-01)	(5.21E-01)	ND	ND	ND
S4DUP1	(1.23E-01)	ND	(5.30E-01)	(4.02E-02)	ND	ND
S4DUP2	(1.71E+00)	(1.58E+00)	(1.33E+00)	(7.76E-01)	ND	ND
S5	ND	ND	(2.00E-01)	ND	ND	(b)
S5DUP1	(1.02E-01)	(3.20E-01)	(1.54E-02)	ND	ND	(b)
S5DUP2	ND	(2.06E-01)	ND	ND	ND	(b)

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
S1	2.28E+01	2.98E+01	1.11E+01	4.80E+01	2.15E+01	2.11E+01
S1DUP1	1.54E+01	2.10E+01	7.95E+00	3.63E+01	1.68E+01	1.67E+01
S1DUP2	3.65E+01	4.52E+01	1.57E+01	6.46E+01	2.83E+01	2.94E+01
S2	(1.54E+00)	(1.67E+00)	(1.53E+00)	5.32E-01	4.69E-01	3.71E+00
S2DUP1	(1.83E-01)	(2.72E-01)	(3.43E-01)	(4.68E-01)	6.26E-01	6.59E+00
S2DUP2	(1.12E+00)	(1.70E+00)	(9.75E-01)	3.12E+00	2.59E+00	1.93E+01
S3	(1.77E-02)	(1.11E-01)	(2.36E-01)	(1.29E-01)	3.87E-01	5.30E+00
S3DUP1	(2.71E-02)	(1.74E-01)	(2.46E-01)	(1.17E-01)	4.84E-01	6.72E+00
S3DUP2	(1.09E-01)	(2.65E-01)	(2.73E-01)	(2.65E-01)	8.49E-01	1.04E+01
S4	(6.37E-02)	(2.05E-01)	(2.64E-01)	(1.04E-01)	(2.09E-01)	2.50E+00
S4DUP1	ND	(3.42E-04)	(9.85E-02)	(9.78E-02)	(1.13E-01)	7.01E-01
S4DUP2	(4.23E-02)	(1.92E-01)	(3.01E-01)	(1.22E-01)	6.88E-01	9.86E+00
S5	(1.86E-01)	(2.10E-01)	(3.08E-01)	(1.17E-01)	(7.95E-02)	(1.63E-01)
S5DUP1	(7.79E-02)	(1.58E-01)	(2.76E-01)	(1.13E-01)	(5.04E-02)	ND
S5DUP2	2.28E+01	(3.04E-01)	(2.78E-01)	(1.54E-01)	6.37E-01	8.44E+00

**Table A.13. (contd)**

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Contact Stage	μg/L						
20578 Stage 1	(1.21E+00)	(7.89E-01)	1.30E+00	1.23E+00	7.32E-01	2.22E+01	2.02E+01
20578 Dup1 Stage 1	(9.64E-01)	(6.22E-01)	1.94E+00	1.83E+00	(4.57E-01)	1.07E+01	1.01E+01
20578 Dup2 Stage 1	(1.86E+00)	(1.23E+00)	2.39E+00	2.18E+00	1.34E+00	4.57E+01	4.53E+01
20578 Stage 2	(1.90E-01)	(1.20E-01)	(5.42E-01)	5.10E-01	(2.32E-01)	2.05E+01	1.50E+01
20578 Dup1 Stage 2	(1.75E-01)	(1.04E-01)	(5.68E-01)	5.13E-01	(2.08E-01)	1.38E+01	1.21E+01
20578 Dup2 Stage 2	(2.30E-01)	(1.30E-01)	1.04E+00	1.06E+00	(3.37E-01)	2.78E+01	2.04E+01
20578 Stage 3	(1.63E-01)	(1.02E-01)	9.09E-01	8.79E-01	(2.48E-01)	1.76E+01	1.74E+01
20578 Dup1 Stage 3	(3.33E-01)	(2.65E-01)	6.07E-01	5.58E-01	(2.34E-01)	2.30E+00	2.47E+00
20578 Dup2 Stage 3	(1.93E-01)	(1.26E-01)	(4.38E-01)	5.42E-01	(3.41E-01)	4.39E+01	2.86E+01
20578 Stage 4	(1.70E-01)	(1.05E-01)	(2.14E-01)	(2.08E-01)	(1.86E-01)	2.28E+00	2.65E+00
20578 Dup1 Stage 4	(1.61E-01)	(1.01E-01)	(3.43E-01)	(2.77E-01)	(1.91E-01)	3.65E+00	3.93E+00
20578 Dup2 Stage 4	(2.49E-01)	(1.61E-01)	(3.14E-01)	5.31E-01	(2.37E-01)	1.43E+01	1.49E+01
20578 Stage 5	(1.76E-01)	(1.17E-01)	9.12E-01	4.99E-01	(2.27E-01)	(1.03E-01)	(3.84E-01)
20578 Stage 1	(1.69E-01)	(1.19E-01)	5.83E-01	4.47E-01	(1.81E-01)	ND	(2.23E-01)
20578 Dup1 Stage 1	(1.74E-01)	(1.02E-01)	6.85E-01	5.72E-01	(2.45E-01)	(3.62E-02)	(3.28E-01)

(a) ICP-MS mass of choice

(b) Se results not reported for last replenishment due to QC failure.

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ICP-MS = Inductively coupled plasma mass spectrometer.

ND = Instrument returned a zero or negative response.

QC = Quality control.

**Table A.14.** Dissolved Anion Concentrations in Periodic Replenishment Double-Deionized Water Leachates from Tank C-108 Residual Waste Samples

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	μg/L					
20578 Stage 1	1.55E+05	(6.00E+03)	(8.45E+04)	(5.87E+04)	(1.71E+04)	1.51E+06
20578 Dup1 Stage 1	2.17E+05	(5.50E+03)	(6.18E+04)	(3.45E+04)	(1.48E+04)	2.10E+06
20578 Dup2 Stage 1	1.97E+05	(6.10E+03)	1.38E+05	1.12E+05	(2.01E+04)	1.93E+06
20578 Stage 2	ND	(5.50E+03)	(2.46E+04)	ND	(7.90E+03)	(5.31E+03)
20578 Dup1 Stage 2	ND	(5.00E+03)	(2.45E+04)	ND	(6.80E+03)	(6.85E+03)
20578 Dup2 Stage 2	(3.00E+02)	(5.20E+03)	(2.62E+04)	ND	(7.10E+03)	(1.71E+04)
20578 Stage 3	ND	(5.40E+03)	ND	ND	(7.00E+03)	(3.16E+03)
20578 Dup1 Stage 3	ND	(5.10E+03)	ND	ND	(6.80E+03)	(3.52E+03)
20578 Dup2 Stage 3	ND	(5.40E+03)	ND	ND	(8.60E+03)	(4.08E+03)
20578 Stage 4	ND	(5.10E+03)	ND	ND	(7.30E+03)	ND
20578 Dup1 Stage 4	ND	(5.90E+03)	ND	ND	(6.70E+03)	ND
20578 Dup2 Stage 4	ND	(5.30E+03)	ND	ND	(6.50E+03)	ND
20578 Stage 5	ND	(1.90E+03)	ND	(1.04E+04)	(1.31E+04)	(5.38E+03)
20578 Dup1 Stage 5	ND	(1.30E+03)	ND	(1.00E+04)	(1.29E+04)	(5.25E+03)
20578 Dup2 Stage 5	ND	(1.60E+03)	ND	(1.01E+04)	(1.30E+04)	(5.28E+03)

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

## A.2 Ca(OH)<sub>2</sub> Solution Leach Tests

### A.2.1 One-Month Single-Contact Ca(OH)<sub>2</sub> Solution Leach Tests

**Table A.15.** pH and Alkalinity Values for Single-Contact Ca(OH)<sub>2</sub> Solution Leachants

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		μg/L	μg/g waste
20578	12.65	8.15E+06	1.22E+06
20578 Dup1	12.66	1.16E+07	1.30E+06
20578 Dup2	12.68	8.25E+06	7.78E+05
20578 Average	12.66	9.32E+06	1.10E+06

Dup = Duplicate.

**Table A.16.** Dissolved  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations in Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578	3.67E+00	4.32E+02	6.23E-02	1.45E-04
20578 Dup1	4.83E+00	5.40E+02	8.22E-02	1.82E-04
20578 Dup2	4.99E+00	3.25E+02	8.48E-02	1.09E-04
20578 Average	4.50E+00	4.32E+02	7.64E-02	1.45E-04
Dup = Duplicate				

**Table A.17.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578	7.65E-04	8.57E-03	6.65E-02	1.20E+00
20578 Dup1	4.21E-04	5.14E-05	3.66E-02	7.20E-03
20578 Dup2	7.03E-04	3.26E-04	6.12E-02	4.57E-02
20578 Average	6.30E-04	2.98E-03	5.48E-02	4.18E-01
Dup = Duplicate				

**Table A.18.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Single-Contact  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578	ND	ND	(2.91E-03)	(0.0469)	ND	ND
20578 Dup1	ND	ND	ND	ND	ND	ND
20578 Dup2	ND	ND	(1.07E-04)	(0.0017)	ND	ND
20578 Average	ND	ND	(9.41E-04)	(0.0152)	ND	ND

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

**Table A.19.** Dissolved Metal Concentrations Determined by ICP-OES in Single-Contact Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

	Al	Bi	Ca	Cr	Fe	Ni	P
Sample Number	µg/L						
20578	1.69E+04	ND	1.35E+04	1.55E+02	(6.39E+01)	(3.12E+01)	(3.11E+02)
20578 Dup1	3.32E+04	ND	(1.06E+02)	2.17E+02	3.70E+02	(4.86E+01)	3.41E+04
20578 Dup2	2.12E+04	ND	9.71E+03	2.07E+02	(8.62E+01)	(7.23E+01)	(2.98E+02)
20578 Average	2.38E+04	ND	1.16E+04	1.93E+02	3.70E+02	(5.07E+01)	3.41E+04

	K	Si	Na	Sr	S	U
Sample Number	µg/L					
20578	(1.90E+03)	(4.00E+01)	9.24E+05	3.09E+02	4.25E+03	4.51E+02
20578 Dup1	(2.10E+03)	3.80E+03	1.44E+06	ND	6.89E+03	5.77E+02
20578 Dup2	2.22E+03	(4.84E+02)	9.63E+05	(1.10E+02)	6.31E+03	3.52E+02
20578 1 Month Avg	2.22E+03	3.80E+03	1.11E+06	3.09E+02	5.82E+03	4.60E+02

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

**Table A.20.** Dissolved Metal Concentrations Determined by ICP-MS in Single-Contact Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/L					
20578	1.46E+02	1.39E+02	(9.87E+00)	ND	(3.72E+01)	ND
20578 Dup1	2.47E+02	2.37E+02	(1.05E+02)	(4.88E+01)	(4.76E+01)	ND
20578 Dup2	2.38E+02	2.38E+02	(1.33E+01)	ND	(2.06E+01)	(9.57E-01)
20578 Avg	2.10E+02	2.05E+02	(4.26E+01)	(4.88E+01)	(3.52E+01)	(9.57E-01)

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
20578	(2.19E+01)	(3.71E+01)	(1.44E+01)	3.47E+01	1.35E+01	1.54E+01
20578 Dup1	5.50E+01	7.34E+01	(3.08E+01)	1.79E+02	8.31E+01	7.95E+01
20578 Dup2	(4.33E+01)	7.00E+01	(2.56E+01)	5.50E+01	2.49E+01	2.40E+01
20578 Avg	5.50E+01	7.17E+01	(2.36E+01)	8.94E+01	4.05E+01	3.96E+01

**Table A.20.** (contd)

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Sample Number	μg/L						
20578	(7.86E+00)	(8.28E+00)	ND	ND	ND	5.95E+01	6.64E+01
20578 Dup1	(9.26E+00)	(8.59E+00)	ND	ND	ND	ND	(2.49E+00)
20578 Dup2	(9.34E+00)	(9.02E+00)	ND	ND	ND	3.70E+01	4.24E+01
20578 Avg	(8.82E+00)	(8.63E+00)	ND	ND	ND	4.82E+01	5.44E+01

(a) ICP-MS mass of choice.

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ICP-MS = Inductively coupled plasma mass spectrometer.

ND = Instrument returned a zero or negative response.

**Table A.21.** Dissolved Anion Concentrations in Single-Contact Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
Sample Number	μg/L					
20578	(9.80E+03)	(6.20E+03)	1.06E+05	(7.97E+04)	(1.66E+04)	(3.29E+03)
20578 Dup1	9.08E+04	(6.70E+03)	1.38E+05	1.08E+05	(2.26E+04)	(1.05E+05)
20578 Dup2	(8.60E+03)	(6.70E+03)	1.39E+05	1.14E+05	(2.02E+04)	ND
20578 Avg	9.08E+04	(6.53E+03)	1.27E+05	1.11E+05	(1.98E+04)	(5.43E+04)

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

## A.2.2 Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leach Tests

**Table A.22.** pH and Alkalinity Values for Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachants

Contact Stage	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		µg/L	µg/g Dry Waste
20578 Stage 1	11.75	1.09E+07	1.45E+06
20578 Dup1 Stage 1	11.84	9.39E+06	1.27E+06
20578 Dup2 Stage 1	11.76	1.07E+07	1.39E+06
20578 Stage 2	11.84	1.13E+07	1.50E+06
20578 Dup1 Stage 2	11.86	8.15E+06	2.18E+06
20578 Dup2 Stage 2	12.73	1.57E+07	1.10E+06
20578 Stage 3	11.73	3.99E+05	5.30E+04
20578 Dup1 Stage 3	11.69	9.12E+06	1.42E+04
20578 Dup2 Stage 3	12.58	9.37E+06	2.01E+04
20578 Stage 4	11.50	8.87E+06	1.18E+06
20578 Dup1 Stage 4	11.60	8.78E+06	1.19E+06
20578 Dup2 Stage 4	11.57	8.99E+06	1.17E+06
20578 Stage 5	11.31	1.93E+06	2.57E+05
20578 Dup1 Stage 5	12.64	1.97E+06	3.51E+03
20578 Dup2 Stage 5	12.66	1.95E+06	3.47E+03

Dup = Duplicate.

**Table A.23.** Dissolved <sup>99</sup>Tc and <sup>238</sup>U Concentrations in Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	<sup>99</sup> Tc	<sup>238</sup> U	<sup>99</sup> Tc	<sup>238</sup> U
	µg/L		µCi/L	
20578 Stage 1	3.65E+00	1.87E+03	6.21E-02	6.28E-04
20578 Dup1 Stage 1	5.30E+00	3.16E+03	9.01E-02	1.06E-03
20578 Dup2 Stage 1	3.65E+00	5.44E+02	6.21E-02	1.83E-04
20578 Stage 2	9.13E-02	5.43E+01	1.55E-03	1.83E-05
20578 Dup1 Stage 2	1.35E-01	5.73E-01	2.30E-03	1.93E-07
20578 Dup2 Stage 2	3.42E-03	2.04E+01	5.81E-05	6.84E-06
20578 Stage 3	ND	8.15E+00	ND	2.74E-06
20578 Dup1 Stage 3	ND	1.91E+00	ND	6.41E-07
20578 Dup2 Stage 3	ND	1.05E+01	ND	3.53E-06
20578 Stage 4	ND	2.05E+01	ND	6.90E-06
20578 Dup1 Stage 4	4.38E-02	1.62E+01	7.45E-04	5.44E-06
20578 Dup2 Stage 4	7.19E-03	5.55E+00	1.22E-04	1.87E-06
20578 Stage 5	7.67E-02	2.11E+01	1.30E-03	7.09E-06
20578 Dup1 Stage 5	9.44E-02	2.16E+01	1.61E-03	7.27E-06
20578 Dup2 Stage 5	1.58E-01	4.91E+00	2.68E-03	1.65E-06

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

**Table A.24.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Periodic Replenishment  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578 Stage 1	(2.33E-04)	1.61E-04	(2.03E-02)	2.26E-02
20578 Dup1 Stage 1	(2.57E-04)	3.36E-04	(2.23E-02)	4.71E-02
20578 Dup2 Stage 1	ND	6.24E-05	ND	8.74E-03
20578 Stage 2	1.15E-03	9.21E-03	9.99E-02	1.29E+00
20578 Dup1 Stage 2	8.53E-04	1.82E-02	7.42E-02	2.55E+00
20578 Dup2 Stage 2	4.65E-04	3.33E-03	4.05E-01	4.66E-01
20578 Stage 3	ND	1.34E-03	ND	1.88E-01
20578 Dup1 Stage 3	ND	4.67E-03	ND	6.54E-01
20578 Dup2 Stage 3	2.36E-04	6.84E-04	2.05E-02	9.58E-02
20578 Stage 4	ND	7.79E-05	ND	1.09E-02
20578 Dup1 Stage 4	ND	7.71E-04	ND	1.08E-01
20578 Dup2 Stage 4	ND	4.04E-05	ND	5.65E-03
20578 Stage 5	ND	8.07E-05	ND	1.13E-02
20578 Dup1 Stage 5	ND	5.25E-04	ND	7.35E-02
20578 Dup2 Stage 5	ND	2.11E-05	ND	2.95E-03

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

**Table A.25.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Periodic Replenishment  $\text{Ca}(\text{OH})_2$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578 Stage 1	(3.17E-04)	(0.4460)	5.18E-02	0.8359	(7.34E-01)	(0.2159)
20578 Dup1 Stage 1	(3.55E-04)	(0.4994)	6.05E-02	0.9754	(1.91E-01)	(0.0562)
20578 Dup2 Stage 1	(2.10E-04)	(0.2953)	(4.26E-02)	(0.6869)	(6.74E-01)	(0.1984)
20578 Stage 2	(2.97E-05)	(0.0419)	ND	ND	ND	ND
20578 Dup1 Stage 2	(6.19E-05)	(0.0872)	(4.45E-03)	(0.0718)	ND	ND
20578 Dup2 Stage 2	(7.75E-05)	(0.1091)	(2.64E-03)	(0.0425)	ND	ND
20578 Stage 3	(6.44E-05)	(0.0908)	(7.41E-03)	(0.1195)	ND	ND
20578 Dup1 Stage 3	(6.85E-05)	(0.0964)	(8.93E-03)	(0.1440)	ND	ND
20578 Dup2 Stage 3	(1.68E-04)	(0.2360)	(5.84E-03)	(0.0942)	ND	ND
20578 Stage 4	(5.00E-05)	(0.0704)	(5.68E-03)	(0.0917)	ND	ND
20578 Dup1 Stage 4	(1.38E-04)	(0.1938)	(4.63E-03)	(0.0747)	ND	ND
20578 Dup2 Stage 4	(4.10E-05)	(0.0578)	(7.71E-03)	(0.1243)	ND	ND
20578 Stage 5	ND	ND	ND	ND	ND	ND
20578 Dup1 Stage 5	ND	ND	ND	ND	ND	ND
20578 Dup2 Stage 5	(4.35E-05)	(0.0613)	(5.21E-04)	(0.0084)	ND	ND

Values within parentheses were <EQL.  
Dup = Duplicate.  
EQL = Estimated quantification limit.  
ND = Instrument returned a zero or negative response.



**Table A.26.** Dissolved Metal Concentrations Determined by ICP-OES in Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	Al	Bi	Ca	Cr	Fe	Ni	P
	µg/L						
20578 Stage 1	2.08E+04	ND	(1.88E+01)	1.59E+02	2.65E+02	ND	3.32E+03
20578 Dup1 Stage 1	1.83E+04	ND	(4.40E+02)	2.41E+02	(1.71E+02)	(9.30E+00)	(4.72E+02)
20578 Dup2 Stage 1	1.05E+04	ND	(1.63E+02)	1.65E+02	3.60E+02	ND	3.10E+04
20578 Stage 2	2.25E+02	(4.94E+02)	7.08E+05	ND	(6.82E+01)	(2.83E+01)	3.19E+03
20578 Dup1 Stage 2	ND	(4.42E+02)	7.49E+05	ND	(8.54E+00)	(7.66E+00)	(5.49E+02)
20578 Dup2 Stage 2	3.97E+02	ND	1.77E+03	ND	(8.85E+01)	ND	3.02E+03
20578 Stage 3	ND	(4.74E+02)	8.49E+05	ND	(1.41E+01)	(1.58E+01)	(5.82E+02)
20578 Dup1 Stage 3	ND	(5.01E+02)	8.40E+05	ND	(7.95E+00)	(1.98E+01)	(5.70E+02)
20578 Dup2 Stage 3	ND	5.64E+02	8.58E+05	ND	ND	(1.26E+01)	(5.80E+02)
20578 Stage 4	ND	5.44E+02	8.68E+05	ND	(2.35E+00)	(1.31E+01)	(5.93E+02)
20578 Dup1 Stage 4	(1.95E+01)	5.97E+02	8.74E+05	ND	3.73E+02	(2.03E+01)	(5.86E+02)
20578 Dup2 Stage 4	(9.18E+00)	5.83E+02	8.79E+05	ND	(9.76E+00)	(1.39E+01)	(5.97E+02)
20578 Stage 5	ND	6.94E+02	8.70E+05	(2.43E+01)	(7.48E+00)	(4.25E+01)	(7.08E+02)
20578 Dup1 Stage 5	(2.52E+01)	6.32E+02	8.68E+05	(1.55E+01)	(1.16E+01)	(4.46E+01)	(6.46E+02)
20578 Dup2 Stage 5	ND	6.05E+02	8.86E+05	(1.29E+01)	(1.44E+01)	(4.55E+01)	(6.41E+02)

Contact Stage	K	Si	Na	Sr	S	U
	µg/L					
20578 Stage 1	(1.70E+03)	2.48E+03	1.19E+06	ND	5.24E+03	1.69E+03
20578 Dup1 Stage 1	2.29E+03	2.29E+03	1.10E+06	(1.89E+01)	7.44E+03	3.04E+03
20578 Dup2 Stage 1	(1.64E+03)	9.34E+02	1.35E+06	ND	6.40E+03	5.54E+02
20578 Stage 2	(5.88E+02)	ND	6.80E+04	5.72E+03	(1.06E+03)	(5.31E+01)
20578 Dup1 Stage 2	(6.45E+02)	ND	3.54E+04	6.96E+03	(1.24E+03)	ND
20578 Dup2 Stage 2	(2.60E+02)	1.44E+03	1.34E+04	1.72E+03	(3.75E+02)	(5.94E+01)
20578 Stage 3	(3.95E+02)	ND	5.11E+03	1.70E+03	(1.92E+03)	(3.24E+01)
20578 Dup1 Stage 3	(4.78E+02)	ND	5.58E+03	2.50E+03	(2.38E+03)	(3.16E+01)
20578 Dup2 Stage 3	(5.03E+02)	ND	1.26E+04	1.74E+03	(2.19E+03)	(2.21E+01)
20578 Stage 4	(4.67E+02)	ND	(1.24E+03)	1.30E+03	(2.43E+03)	(3.25E+01)
20578 Dup1 Stage 4	(4.22E+02)	ND	(1.26E+03)	1.50E+03	(2.54E+03)	(3.31E+01)
20578 Dup2 Stage 4	(5.02E+02)	ND	(1.29E+03)	1.25E+03	(2.39E+03)	(9.45E+00)
20578 Stage 5	(1.75E+02)	ND	(1.06E+03)	1.26E+03	(1.99E+03)	(4.25E+01)
20578 Dup1 Stage 5	(5.24E+02)	ND	(1.09E+03)	1.38E+03	(2.07E+03)	(5.33E+01)
20578 Dup2 Stage 5	(3.44E+02)	ND	(1.07E+03)	1.30E+03	(2.04E+03)	(3.29E+01)

Values within parentheses were <EQL.  
Dup = Duplicate.  
EQL = Estimated quantification limit.  
ND = Instrument returned a zero or negative response.

**Table A.27.** Dissolved Metal Concentrations Determined by ICP-MS in Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/L					
S1	1.44E+02	1.55E+02	(3.19E+01)	ND	(1.22E+00)	ND
S1DUP1	2.38E+02	2.53E+02	(8.61E+01)	(4.55E+01)	ND	ND
S1DUP2	1.58E+02	1.72E+02	(3.41E+01)	ND	(4.22E+00)	ND
S2	(1.40E+01)	(7.96E+00)	ND	ND	ND	ND
S2DUP1	(7.00E+00)	(3.01E+00)	ND	ND	ND	ND
S2DUP2	(1.58E+00)	ND	(1.09E+00)	ND	ND	ND
S3	(1.22E+01)	(1.21E+01)	ND	ND	ND	ND
S3DUP1	(2.55E+01)	(1.99E+01)	ND	ND	ND	ND
S3DUP2	(2.01E+01)	(1.50E+01)	ND	ND	ND	ND
S4	ND	ND	ND	ND	(2.23E+01)	ND
S4DUP1	ND	ND	ND	ND	(1.56E+01)	ND
S4DUP2	ND	ND	ND	ND	(2.29E+01)	(1.45E+01)
S5	(1.72E+01)	(2.61E+01)	ND	ND	ND	(b)
S5DUP1	(1.80E+01)	(1.71E+01)	ND	ND	ND	(b)
S5DUP2	(1.20E+01)	(1.49E+01)	ND	ND	ND	(b)

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
S1	(3.44E+01)	(5.30E+01)	(2.25E+01)	1.05E+02	5.12E+01	4.53E+01
S1DUP1	5.78E+01	8.45E+01	(3.36E+01)	1.19E+02	5.60E+01	4.89E+01
S1DUP2	(3.96E+01)	6.02E+01	(2.56E+01)	1.21E+02	5.71E+01	5.00E+01
S2	ND	ND	(2.56E+00)	(2.56E+00)	(4.97E+00)	3.07E+01
S2DUP1	ND	(1.00E+00)	(2.00E+00)	(2.41E+00)	(5.25E+00)	3.71E+01
S2DUP2	ND	(1.04E+00)	(2.53E+00)	(1.99E+00)	(1.59E+00)	(6.39E+00)
S3	ND	(9.48E-01)	(2.33E+00)	(2.09E+00)	(3.47E+00)	(4.09E+00)
S3DUP1	ND	(5.22E-01)	(2.37E+00)	(2.30E+00)	(3.75E+00)	(9.41E+00)
S3DUP2	ND	(9.99E-01)	(3.13E+00)	(2.05E+00)	(3.32E+00)	(5.52E+00)
S4	ND	ND	ND	ND	ND	ND
S4DUP1	ND	ND	(2.82E-01)	ND	ND	ND
S4DUP2	ND	ND	ND	ND	(1.43E+00)	(1.26E-01)
S5	ND	(2.18E+00)	(3.21E+00)	(2.79E+00)	(4.20E+00)	(5.51E+00)
S5DUP1	ND	ND	(2.69E+00)	(3.24E+00)	(3.99E+00)	(5.03E+00)
S5DUP2	(2.34E+00)	(4.54E+00)	(6.17E+00)	(2.05E+00)	(4.38E+00)	(3.02E+00)

**Table A.27. (contd)**

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Contact Stage	µg/L						
20578 Stage 1	(5.45E+00)	(3.46E+00)	(1.10E+01)	9.50E+00	(3.42E+00)	(2.21E+01)	2.66E+01
20578 Dup1 Stage 1	(6.72E+00)	(3.81E+00)	(8.66E+00)	8.42E+00	(3.39E+00)	6.96E+01	7.38E+01
20578 Dup2 Stage 1	(6.01E+00)	(3.06E+00)	1.15E+01	(6.81E+00)	(4.11E+00)	(5.88E+00)	(1.15E+01)
20578 Stage 2	(3.49E+00)	(2.12E+00)	(7.41E+00)	(7.21E+00)	(3.55E+00)	2.57E+02	2.63E+02
20578 Dup1 Stage 2	(3.31E+00)	(2.15E+00)	(7.99E+00)	(6.26E+00)	(3.29E+00)	1.60E+02	1.63E+02
20578 Dup2 Stage 2	(3.31E+00)	(1.99E+00)	(6.38E+00)	(5.55E+00)	(2.97E+00)	(9.98E-01)	(6.98E+00)
20578 Stage 3	(3.06E+00)	(1.96E+00)	(4.26E+00)	(4.89E+00)	(3.45E+00)	2.75E+02	2.76E+02
20578 Dup1 Stage 3	(3.17E+00)	(1.74E+00)	(3.09E+00)	(3.71E+00)	(2.74E+00)	1.75E+02	1.87E+02
20578 Dup2 Stage 3	(3.31E+00)	(1.75E+00)	(2.43E+00)	(3.13E+00)	(2.97E+00)	1.99E+02	2.03E+02
20578 Stage 4	(1.42E+01)	(1.34E+01)	ND	ND	ND	2.09E+02	2.18E+02
20578 Dup1 Stage 4	(1.12E+01)	(1.20E+01)	ND	ND	ND	1.46E+02	1.49E+02
20578 Dup2 Stage 4	(1.15E+01)	(1.17E+01)	ND	ND	ND	8.87E+01	9.22E+01
20578 Stage 5	(3.33E+00)	(2.07E+00)	1.34E+01	9.59E+00	(3.13E+00)	1.30E+02	1.38E+02
20578 Dup1 Stage 5	(3.53E+00)	(2.10E+00)	(1.06E+01)	8.28E+00	(3.12E+00)	1.19E+02	1.24E+02
20578 Dup2 Stage 5	(3.31E+00)	(2.10E+00)	1.18E+01	1.05E+01	(3.86E+00)	8.24E+01	8.96E+01

(a) ICP-MS mass of choice.

(b) Se results not reported for last replenishment due to QC failure.

Values within parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ICP-MS = Inductively coupled plasma mass spectrometer.

ND = Instrument returned a zero or negative response.

QC = Quality control.

**Table A.28.** Dissolved Anion Concentrations in Periodic Replenishment Ca(OH)<sub>2</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	µg/L					
20578 Stage 1	6.84E+04	(6.20E+03)	1.03E+05	(7.90E+04)	(1.91E+04)	(5.74E+03)
20578 Dup1 Stage 1	3.25E+04	(6.80E+03)	1.56E+05	1.28E+05	(2.44E+04)	(3.33E+03)
20578 Dup2 Stage 1	9.51E+04	(6.30E+03)	1.11E+05	(8.55E+04)	(2.22E+04)	(3.10E+04)
20578 Stage 2	ND	(5.30E+03)	(2.62E+04)	ND	(7.00E+03)	ND
20578 Dup1 Stage 2	ND	(5.70E+03)	(2.60E+04)	ND	(7.90E+03)	ND
20578 Dup2 Stage 2	ND	(5.10E+03)	(2.46E+04)	ND	(6.70E+03)	(4.76E+03)
20578 Stage 3	ND	(1.43E+04)	ND	ND	(7.70E+03)	ND
20578 Dup1 Stage 3	ND	(5.40E+03)	ND	ND	(7.10E+03)	ND
20578 Dup2 Stage 3	ND	(5.40E+03)	(2.47E+04)	ND	(7.30E+03)	ND
20578 Stage 4	ND	(5.60E+03)	ND	ND	(7.70E+03)	ND
20578 Dup1 Stage 4	ND	(5.50E+03)	ND	ND	(7.60E+03)	ND
20578 Dup2 Stage 4	ND	(6.40E+03)	ND	ND	(9.60E+03)	ND
20578 Stage 5	ND	(1.90E+03)	ND	(1.01E+04)	(1.34E+04)	(5.44E+03)
20578 Dup1 Stage 5	ND	(1.20E+03)	ND	ND	(1.31E+04)	ND
20578 Dup2 Stage 5	ND	(3.00E+03)	ND	(1.37E+04)	(1.66E+04)	(5.22E+03)

Values within parentheses were <EQL.

Dup = Duplicate.

EQL= Estimated quantification limit.

ND = Instrument returned a zero or negative response.

## A.3 CaCO<sub>3</sub> Solution Leach Tests

### A.3.1 One-Month Single-Contact CaCO<sub>3</sub> Solution Leach Tests

**Table A.29.** pH and Alkalinity Values for Single-Contact CaCO<sub>3</sub> Solution Leachants

Sample Number	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		(µg/L)	(µg/g waste)
20578	12.76	8.06E+06	9.34E+05
20578 Dup1	12.67	6.23E+06	9.05E+05
20578 Dup2	12.69	5.09E+06	7.53E+05
20578 Average	12.71	6.46E+06	8.64E+05

Dup = Duplicate.

**Table A.30.** Dissolved  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations in Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578	3.11E+00	3.98E+03	5.28E-02	1.34E-03
20578 Dup1	3.60E+00	4.09E+03	6.12E-02	1.37E-03
20578 Dup2	6.75E+00	6.01E+03	1.15E-01	2.02E-03
20578 Average	4.49E+00	4.70E+03	7.62E-02	1.58E-03
Dup = Duplicate.				

**Table A.31.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578	6.01E-04	1.91E-03	5.23E-02	2.68E-01
20578 Dup1	3.04E-04	2.73E-04	2.65E-02	3.82E-02
20578 Dup2	5.10E-04	8.14E-05	4.44E-02	1.14E-02
20578 Average	4.72E-04	7.56E-04	4.10E-02	1.06E-01
Dup = Duplicate.				

**Table A.32.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578	ND	ND	(2.72E-02)	(0.4388)	ND	ND
20578 Dup1	ND	ND	(1.64E-02)	(0.2644)	ND	ND
20578 Dup2	(1.91E-04)	(0.2696)	(4.07E-02)	(0.6570)	ND	ND
20578 Average	(1.91E-04)	(0.2696)	(2.81E-02)	(0.4534)	ND	ND

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

**Table A.33.** Dissolved Metal Concentrations Determined by ICP-OES in Single-Contact  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	Al	Bi	Ca	Cr	Fe	Ni	P
	$\mu\text{g/L}$						
20578	5.18E+03	ND	(4.27E+02)	1.23E+02	2.19E+02	(2.56E+01)	5.18E+05
20578 Dup1	6.32E+03	ND	(1.27E+02)	1.34E+02	1.92E+02	(1.19E+01)	3.40E+05
20578 Dup2	7.07E+03	ND	(3.59E+02)	2.91E+02	2.71E+02	3.47E+02	1.81E+05
20578 1 Month Avg	6.19E+03	ND	(3.05E+02)	1.82E+02	2.27E+02	3.47E+02	3.46E+05

**Table A.33.** (contd)

	K	Si	Na	Sr	S	U
Sample Number	µg/L					
20578	(1.26E+03)	3.22E+03	1.56E+06	(1.18E+01)	4.39E+03	3.94E+03
20578 Dup1	(1.44E+03)	3.54E+03	1.11E+06	(5.60E+00)	4.16E+03	4.11E+03
20578 Dup2	2.56E+03	1.67E+03	9.00E+05	(1.06E+01)	8.42E+03	6.26E+03
20578 1 Month Avg	2.56E+03	2.81E+03	1.19E+06	(9.33E+00)	5.66E+03	4.77E+03

Concentrations listed in parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ND = Instrument returned a zero or negative response.

**Table A.34.** Dissolved Metal Concentrations Determined by ICP-MS in Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste Samples

	<sup>53</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
Sample Number	µg/L					
20578	1.14E+02	1.22E+02	(3.72E+01)	ND	(4.69E+01)	ND
20578 Dup1	1.10E+02	1.04E+02	(4.11E+01)	ND	(4.39E+01)	ND
20578 Dup2	3.03E+02	3.33E+02	(4.39E+01)	(2.32E+00)	(1.99E+01)	ND
20578 Avg	1.76E+02	1.86E+02	(4.07E+01)	(2.32E+00)	(3.69E+01)	ND

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
20578	(2.34E+01)	(3.60E+01)	(1.44E+01)	9.93E+01	4.54E+01	4.40E+01
20578 Dup1	(3.21E+01)	(5.40E+01)	(1.98E+01)	1.24E+02	5.53E+01	4.96E+01
20578 Dup2	6.07E+01	9.30E+01	(3.43E+01)	2.18E+02	1.03E+02	9.71E+01
20578 Avg	6.07E+01	9.30E+01	(2.28E+01)	1.47E+02	6.80E+01	6.36E+01

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Sample Number	µg/L						
20578	(9.02E+00)	(8.29E+00)	ND	ND	ND	(2.31E+01)	2.99E+01
20578 Dup1	(8.19E+00)	(8.38E+00)	ND	ND	ND	(1.25E+01)	(1.74E+01)
20578 Dup2	(9.56E+00)	(9.37E+00)	ND	ND	ND	(2.49E+01)	3.09E+01
20578 1 Month Avg	(8.92E+00)	(8.68E+00)	ND	ND	ND	(2.01E+01)	3.04E+01

(a) ICP-MS mass of choice.

Concentrations listed in parentheses were <EQL.

Avg = Average.

Dup = Duplicate.

EQL = Estimated quantitation limit.

ICP-MS = Inductively coupled plasma mass spectrometer.

ND = Instrument returned a zero or negative response.

**Table A.35.** Dissolved Anion Concentrations in Single-Contact CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	μg/L					
20578	1.68E+02	(6.00E+03)	(8.91E+04)	(6.48E+04)	(1.66E+04)	1.63E+06
20578 Dup1	ND	(6.00E+03)	ND	ND	(7.10E+03)	(3.39E+03)
20578 Dup2	5.50E+01	(6.70E+03)	1.90E+05	1.59E+05	(2.59E+04)	5.62E+05
20578 Avg	1.11E+05	(6.23E+03)	1.90E+05	1.59E+05	(1.65E+04)	1.09E+06

Concentrations listed in parentheses were <EQL.  
 Avg = Average.  
 Dup = Duplicate.  
 EQL = Estimated quantitation limit.  
 ND = Instrument returned a zero or negative response.

### A.3.2 Periodic Replenishment CaCO<sub>3</sub> Solution Leach Tests

**Table A.36.** pH and Alkalinity Values for Periodic Replenishment CaCO<sub>3</sub> Solution Leachants

Contact Stage	pH	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint	Total Alkalinity (as CaCO <sub>3</sub> ) at pH 4.5 Endpoint
		μg/L	μg/g Dry Waste
20578 Stage 1	12.71	9.73E+06	1.21E+06
20578 Dup1 Stage 1	12.76	8.38E+06	9.97E+05
20578 Dup2 Stage 1	12.61	7.26E+06	1.01E+06
20578 Stage 2	7.63	3.50E+05	4.36E+04
20578 Dup1 Stage 2	8.20	2.35E+05	2.79E+04
20578 Dup2 Stage 2	11.77	1.73E+05	2.26E+04
20578 Stage 3	7.47	9.90E+06	1.23E+06
20578 Dup1 Stage 3	7.35	1.19E+05	1.23E+06
20578 Dup2 Stage 3	7.48	1.44E+05	1.22E+06
20578 Stage 4	7.12	1.15E+05	1.44E+04
20578 Dup1 Stage 4	7.99	8.65E+04	1.03E+04
20578 Dup2 Stage 4	7.72	1.32E+05	1.84E+04
20578 Stage 5	6.94	2.39E+04	2.97E+03
20578 Dup1 Stage 5	7.41	(2.14E+04)	(3.81E+01)
20578 Dup2 Stage 5	7.16	(2.14E+04)	(3.81E+01)

Concentrations listed in parentheses were <EQL.  
 Dup = Duplicate.  
 EQL = Estimated quantitation limit.

**Table A.37.** Dissolved  $^{99}\text{Tc}$  and  $^{238}\text{U}$  Concentrations in Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{99}\text{Tc}$	$^{238}\text{U}$	$^{99}\text{Tc}$	$^{238}\text{U}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578 Stage 1	3.28E+00	4.11E+03	5.57E-02	1.38E-03
20578 Dup1 Stage 1	3.99E+00	4.25E+03	6.79E-02	1.43E-03
20578 Dup2 Stage 1	3.12E+00	4.66E+03	5.31E-02	1.56E-03
20578 Stage 2	1.57E-01	5.82E+00	2.67E-03	1.96E-06
20578 Dup1 Stage 2	1.45E-01	1.08E+02	2.46E-03	3.64E-05
20578 Dup2 Stage 2	2.16E-01	6.76E+00	3.67E-03	2.27E-06
20578 Stage 3	5.60E-02	9.94E+00	9.52E-04	3.34E-06
20578 Dup1 Stage 3	1.29E-01	3.78E+01	2.20E-03	1.27E-05
20578 Dup2 Stage 3	ND	9.34E+01	ND	3.14E-05
20578 Stage 4	ND	8.67E+00	ND	2.91E-06
20578 Dup1 Stage 4	6.45E-02	2.43E+01	1.10E-03	8.16E-06
20578 Dup2 Stage 4	4.48E-03	6.22E+01	7.61E-05	2.09E-05
20578 Stage 5	3.89E-01	1.95E+01	6.61E-03	6.57E-06
20578 Dup1 Stage 5	2.83E-01	1.49E+01	4.81E-03	5.00E-06
20578 Dup2 Stage 5	2.97E-01	3.41E+01	5.05E-03	1.15E-05

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.

**Table A.38.** Dissolved  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$  Concentrations in Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{137}\text{Cs}$	$^{90}\text{Sr}$	$^{137}\text{Cs}$	$^{90}\text{Sr}$
	$\mu\text{g/L}$		$\mu\text{Ci/L}$	
20578 Stage 1	3.92E-04	1.33E-03	3.41E-02	1.86E-01
20578 Dup1 Stage 1	4.02E-04	3.64E-04	3.50E-02	5.09E-02
20578 Dup2 Stage 1	(2.31E-04)	4.16E-04	(2.01E-02)	5.83E-02
20578 Stage 2	ND	7.43E-03	ND	1.04E+00
20578 Dup1 Stage 2	4.58E-04	2.11E-03	3.98E-02	2.95E-01
20578 Dup2 Stage 2	1.30E-03	1.04E-01	1.13E-01	1.46E+01
20578 Stage 3	ND	5.49E-02	ND	7.68E+00
20578 Dup1 Stage 3	4.17E-04	2.15E-03	3.63E-02	3.01E-01
20578 Dup2 Stage 3	3.56E-04	2.12E-03	3.10E-02	2.97E-01
20578 Stage 4	ND	1.51E-02	ND	2.11E+00
20578 Dup1 Stage 4	ND	9.93E-04	ND	1.39E-01
20578 Dup2 Stage 4	2.12E-04	1.10E-03	1.84E-02	1.54E-01
20578 Stage 5	ND	5.06E-03	ND	7.09E-01
20578 Dup1 Stage 5	ND	6.01E-04	ND	8.41E-02
20578 Dup2 Stage 5	ND	4.38E-04	ND	6.13E-02

Dup = Duplicate.  
ND = Instrument returned a zero or negative response.



**Table A.39.** Dissolved  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ , and  $^{241}\text{Am}$  Concentrations in Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	$^{237}\text{Np}$		$^{239}\text{Pu}$		$^{241}\text{Am}$	
	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$	$\mu\text{Ci/L}$	$\mu\text{g/L}$
20578 Stage 1	5.17E-04	0.7281	8.41E-02	1.3559	(9.67E-01)	(0.2843)
20578 Dup1 Stage 1	(3.69E-04)	(0.5195)	7.77E-02	1.2525	(6.70E-01)	(0.1970)
20578 Dup2 Stage 1	4.72E-04	0.6643	7.68E-02	1.2383	(4.51E-01)	(0.1327)
20578 Stage 2	ND	ND	ND	ND	ND	ND
20578 Dup1 Stage 2	ND	ND	(1.85E-03)	(0.0298)	ND	ND
20578 Dup2 Stage 2	(2.91E-05)	(0.0410)	ND	ND	ND	ND
20578 Stage 3	ND	ND	(7.75E-05)	(0.0013)	ND	ND
20578 Dup1 Stage 3	ND	ND	ND	ND	ND	ND
20578 Dup2 Stage 3	ND	ND	(2.01E-03)	(0.0324)	ND	ND
20578 Stage 4	ND	ND	ND	ND	ND	ND
20578 Dup1 Stage 4	(3.14E-05)	(0.0442)	(2.81E-03)	(0.0453)	ND	ND
20578 Dup2 Stage 4	(1.08E-05)	(0.0152)	(1.15E-04)	(0.0019)	ND	ND
20578 Stage 5	ND	ND	ND	ND	ND	ND
20578 Dup1 Stage 5	ND	ND	(3.98E-03)	(0.0642)	ND	ND
20578 Dup2 Stage 5	(1.16E-04)	(0.1636)	(1.10E-02)	(0.1775)	ND	ND

Values within parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

**Table A.40.** Dissolved Metal Concentrations Determined by ICP-OES in Periodic Replenishment  $\text{CaCO}_3$  Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	Al	Bi	Ca	Cr	Fe	Ni	P
	$\mu\text{g/L}$						
20578 Stage 1	4.76E+03	ND	(2.24E+02)	1.42E+02	4.39E+02	(9.89E+00)	5.38E+05
20578 Dup1 Stage 1	5.41E+03	ND	(9.81E+01)	1.77E+02	1.08E+03	ND	4.87E+05
20578 Dup2 Stage 1	3.36E+03	ND	(1.26E+02)	1.19E+02	3.33E+02	ND	4.08E+05
20578 Stage 2	3.58E+02	ND	2.64E+03	ND	ND	ND	3.29E+03
20578 Dup1 Stage 2	1.07E+03	ND	1.01E+03	ND	(6.33E+01)	(2.19E+01)	5.46E+03
20578 Dup2 Stage 2	5.37E+02	5.45E+02	6.22E+05	ND	(1.33E+02)	(1.07E+01)	(5.90E+02)
20578 Stage 3	1.66E+02	ND	5.67E+03	ND	(5.24E+01)	ND	(5.58E+02)
20578 Dup1 Stage 3	1.66E+02	(3.92E+00)	4.95E+03	ND	(6.65E+00)	(2.14E+00)	(8.18E+02)
20578 Dup2 Stage 3	4.78E+02	ND	4.99E+03	ND	(5.17E+01)	(4.97E-01)	(9.03E+02)
20578 Stage 4	(9.74E+01)	ND	5.89E+03	ND	(2.09E+01)	ND	(3.59E+02)
20578 Dup1 Stage 4	1.74E+02	ND	6.01E+03	ND	(9.56E+00)	ND	(5.41E+02)
20578 Dup2 Stage 4	2.30E+02	(4.00E+01)	6.03E+03	ND	(4.09E+01)	(6.67E-02)	(6.28E+02)
20578 Stage 5	(4.30E+01)	ND	5.48E+03	(4.27E+00)	(2.03E+00)	(4.86E+00)	(2.18E+02)
20578 Dup1 Stage 5	(6.33E+01)	ND	6.07E+03	(6.22E+00)	ND	(9.23E+00)	(3.55E+02)
20578 Dup2 Stage 5	(1.05E+02)	ND	5.83E+03	(1.59E+00)	2.98E+00	(4.67E+00)	(3.62E+02)

**Table A.40.** (contd)

Contact Stage	K	Si	Na	Sr	S	U
	µg/L					
20578 Stage 1	(1.54E+03)	1.59E+03	1.61E+06	(1.45E+01)	4.76E+03	3.97E+03
20578 Dup1 Stage 1	(1.71E+03)	1.54E+03	1.56E+06	(3.00E+00)	5.66E+03	4.07E+03
20578 Dup2 Stage 1	(1.41E+03)	1.47E+03	1.28E+06	(2.49E+00)	3.86E+03	4.58E+03
20578 Stage 2	(1.33E+02)	1.40E+03	1.19E+04	2.98E+03	(2.33E+02)	(4.08E+01)
20578 Dup1 Stage 2	(2.42E+02)	1.67E+03	2.32E+04	8.95E+02	(2.75E+02)	(1.61E+02)
20578 Dup2 Stage 2	(6.22E+02)	ND	1.37E+05	6.24E+03	(1.03E+03)	(2.31E+01)
20578 Stage 3	(1.70E+02)	1.14E+03	(1.31E+03)	1.31E+03	(1.35E+02)	(4.84E+01)
20578 Dup1 Stage 3	(2.43E+02)	1.10E+03	2.74E+03	1.43E+03	(1.12E+02)	(7.01E+01)
20578 Dup2 Stage 3	(9.06E+01)	1.37E+03	2.60E+03	1.85E+03	(2.88E+02)	(1.27E+02)
20578 Stage 4	(2.07E+02)	1.15E+03	(5.74E+02)	4.29E+02	(1.32E+02)	(3.31E+01)
20578 Dup1 Stage 4	(2.84E+02)	1.11E+03	(5.75E+02)	7.15E+02	(1.58E+02)	(6.32E+01)
20578 Dup2 Stage 4	(6.56E+01)	1.28E+03	(7.32E+02)	1.08E+03	(1.44E+02)	(1.10E+02)
20578 Stage 5	(1.37E+02)	9.53E+02	(2.92E+02)	1.68E+02	ND	(6.16E+01)
20578 Dup1 Stage 5	(8.40E+01)	1.02E+03	(2.68E+02)	3.44E+02	ND	(4.15E+01)
20578 Dup2 Stage 5	(1.37E+02)	1.14E+03	(4.40E+02)	4.52E+02	ND	(6.85E+01)

Values within parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

**Table A.41.** Dissolved Metal Concentrations Determined by ICP-MS in Periodic Replenishment CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Sample Number	<sup>52</sup> Cr <sup>(a)</sup>	<sup>53</sup> Cr	<sup>63</sup> Cu	<sup>65</sup> Cu <sup>(a)</sup>	<sup>75</sup> As	<sup>82</sup> Se
	µg/L					
S1	1.19E+02	1.25E+02	(4.82E+01)	ND	(6.73E+00)	ND
S1DUP1	1.51E+02	1.59E+02	(4.73E+01)	ND	(1.83E+01)	ND
S1DUP2	1.12E+02	1.17E+02	(3.79E+01)	ND	(7.53E+00)	ND
S2	ND	(1.53E-01)	ND	ND	ND	ND
S2DUP1	(1.09E-01)	(5.48E-02)	ND	ND	ND	ND
S2DUP2	(2.73E+00)	ND	(3.24E+00)	ND	ND	ND
S3	ND	ND	(9.70E-01)	ND	ND	ND
S3DUP1	ND	ND	ND	ND	ND	ND
S3DUP2	(2.20E+00)	(3.57E+00)	ND	ND	ND	ND
S4	ND	ND	ND	ND	ND	ND
S4DUP1	(3.07E-01)	ND	ND	ND	ND	ND
S4DUP2	(5.38E+00)	(2.92E+00)	ND	ND	ND	ND
S5	ND	ND	ND	ND	ND	ND
S5DUP1	ND	ND	ND	ND	ND	ND
S5DUP2	ND	ND	ND	ND	ND	ND

**Table A.41. (contd)**

	<sup>95</sup> Mo <sup>(a)</sup>	<sup>97</sup> Mo	<sup>98</sup> Mo	<sup>101</sup> Ru <sup>(a)</sup>	<sup>102</sup> Ru	<sup>104</sup> Ru
Sample Number	µg/L					
S1	(3.68E+01)	(5.31E+01)	(2.53E+01)	1.15E+02	5.37E+01	4.67E+01
S1DUP1	(4.09E+01)	6.41E+01	(2.84E+01)	1.33E+02	6.31E+01	5.64E+01
S1DUP2	(3.40E+01)	(5.19E+01)	(2.29E+01)	1.05E+02	4.76E+01	3.97E+01
S2	(4.90E-01)	(4.06E+00)	(4.60E+00)	(2.85E+00)	(2.25E+00)	1.40E+01
S2DUP1	ND	(1.22E+00)	(4.19E+00)	(3.62E+00)	(1.76E+00)	(2.33E+00)
S2DUP2	(2.93E+00)	(6.12E+00)	(6.57E+00)	(2.26E+00)	(5.04E+00)	3.26E+01
S3	ND	ND	(1.68E+00)	(1.78E+00)	(1.70E+00)	(3.56E+00)
S3DUP1	(2.80E+00)	(4.33E+00)	(6.83E+00)	(2.05E+00)	(1.62E+00)	(3.86E+00)
S3DUP2	ND	(6.60E-01)	(2.91E+00)	(2.12E+00)	(1.53E+00)	(6.47E+00)
S4	(5.59E-01)	(3.02E+00)	(4.74E+00)	(1.61E+00)	(1.14E+00)	ND
S4DUP1	ND	ND	(1.26E+00)	(1.95E+00)	(1.11E+00)	(2.53E-01)
S4DUP2	ND	ND	(1.10E+00)	(1.82E+00)	(1.06E+00)	(1.59E+00)
S5	(1.33E+00)	(4.09E+00)	(4.54E+00)	(2.35E+00)	(1.00E+00)	ND
S5DUP1	ND	(3.53E+00)	(2.80E+00)	(2.43E+00)	(9.63E-01)	ND
S5DUP2	ND	(2.72E+00)	(4.29E+00)	(1.85E+00)	(1.15E+00)	ND

	<sup>107</sup> Ag <sup>(a)</sup>	<sup>109</sup> Ag	<sup>111</sup> Cd <sup>(a)</sup>	<sup>114</sup> Cd	<sup>121</sup> Sb	<sup>206</sup> Pb	<sup>208</sup> Pb <sup>(a)</sup>
Contact Stage	µg/L						
20578 Stage 1	(6.87E+00)	(4.54E+00)	1.25E+01	9.67E+00	(3.75E+00)	(1.71E+01)	2.45E+01
20578 Dup1 Stage 1	(6.20E+00)	(3.51E+00)	1.32E+01	1.03E+01	(4.59E+00)	3.77E+01	4.35E+01
20578 Dup2 Stage 1	(5.52E+00)	(3.07E+00)	1.50E+01	1.04E+01	(4.34E+00)	(7.43E-02)	(6.08E+00)
20578 Stage 2	(3.20E+00)	(2.05E+00)	(8.92E+00)	(7.19E+00)	(2.89E+00)	(8.84E-01)	(6.61E+00)
20578 Dup1 Stage 2	(3.49E+00)	(2.24E+00)	(8.00E+00)	(7.60E+00)	(3.58E+00)	(1.64E+01)	(2.23E+01)
20578 Dup2 Stage 2	(3.61E+00)	(2.16E+00)	(1.07E+01)	8.03E+00	(3.47E+00)	1.69E+02	1.79E+02
20578 Stage 3	(3.26E+00)	(1.80E+00)	(6.14E+00)	(6.47E+00)	(3.05E+00)	(4.29E+00)	(8.70E+00)
20578 Dup1 Stage 3	(3.24E+00)	(2.10E+00)	(8.20E+00)	(6.89E+00)	(3.38E+00)	(8.55E+00)	(1.47E+01)
20578 Dup2 Stage 3	(3.48E+00)	(1.96E+00)	(6.81E+00)	(3.95E+00)	(2.59E+00)	(4.34E+00)	(1.03E+01)
20578 Stage 4	(3.09E+00)	(1.79E+00)	(1.72E+00)	(2.28E+00)	(3.15E+00)	(6.62E+00)	(1.11E+01)
20578 Dup1 Stage 4	(2.97E+00)	(1.76E+00)	(1.28E+00)	(2.58E+00)	(2.71E+00)	ND	(4.74E+00)
20578 Dup2 Stage 4	(3.31E+00)	(1.96E+00)	(1.64E+00)	(3.05E+00)	(2.42E+00)	(7.01E+00)	(1.31E+01)
20578 Stage 5	(3.39E+00)	(2.08E+00)	1.23E+01	8.39E+00	(3.03E+00)	ND	(7.21E-01)
20578 Dup1 Stage 5	(3.24E+00)	(1.80E+00)	(1.10E+01)	8.13E+00	(3.02E+00)	ND	(1.46E+00)
20578 Dup2 Stage 5	(3.27E+00)	(1.97E+00)	1.30E+01	1.08E+01	(3.34E+00)	(1.71E+01)	(9.87E-01)

(a) ICP-MS mass of choice.  
Dup = Duplicate.  
EQL = Estimated quantitation limit.  
ICP-MS = Inductively coupled plasma mass spectrometer.  
ND = Instrument returned a zero or negative response.  
QC = Quality control.

**Table A.42.** Dissolved Anion Concentrations in Periodic Replenishment CaCO<sub>3</sub> Solution Leachates from Tank C-108 Residual Waste Samples

Contact Stage	F	Cl	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	PO <sub>4</sub>
	μg/L					
20578 Stage 1	1.74E+05	(5.80E+03)	(9.87E+04)	(7.44E+04)	(1.78E+04)	1.70E+06
20578 Dup1 Stage 1	1.61E+05	(6.10E+03)	1.22E+05	(9.37E+04)	(1.96E+04)	1.56E+06
20578 Dup2 Stage 1	1.33E+05	(5.90E+03)	(9.42E+04)	(6.75E+04)	(1.58E+04)	1.29E+06
20578 Stage 2	ND	(5.00E+03)	(2.47E+04)	ND	(7.30E+03)	(5.41E+03)
20578 Dup1 Stage 2	ND	(6.00E+03)	(2.50E+04)	ND	(6.90E+03)	(6.85E+03)
20578 Dup2 Stage 2	ND	(5.60E+03)	(2.90E+04)	(9.00E+02)	(6.90E+03)	ND
20578 Stage 3	ND	(6.20E+03)	ND	ND	(7.10E+03)	(3.03E+03)
20578 Dup1 Stage 3	ND	(5.00E+03)	ND	ND	(6.60E+03)	(3.33E+03)
20578 Dup2 Stage 3	ND	(5.40E+03)	ND	ND	(1.29E+04)	(3.29E+03)
20578 Stage 4	ND	(5.80E+03)	ND	ND	(8.90E+03)	(3.10E+03)
20578 Dup1 Stage 4	1.12E+05	(6.10E+03)	1.00E+05	(7.88E+04)	(1.64E+04)	1.09E+06
20578 Dup2 Stage 4	ND	(5.30E+03)	ND	ND	(6.50E+03)	(3.59E+03)
20578 Stage 5	ND	(1.10E+03)	ND	(9.90E+03)	(1.23E+04)	(5.38E+03)
20578 Dup1 Stage 5	ND	(1.10E+03)	ND	(9.70E+03)	(1.22E+04)	(5.57E+03)
20578 Dup2 Stage 5	ND	(1.50E+03)	ND	(1.00E+04)	(1.27E+04)	(5.35E+03)

Values within parentheses were <EQL.

Dup = Duplicate.

EQL = Estimated quantification limit.

ND = Instrument returned a zero or negative response.

## **Appendix B**

### **X-Ray Diffraction Patterns for Unleached and Leached Samples of Residual Waste from Tank C-108**

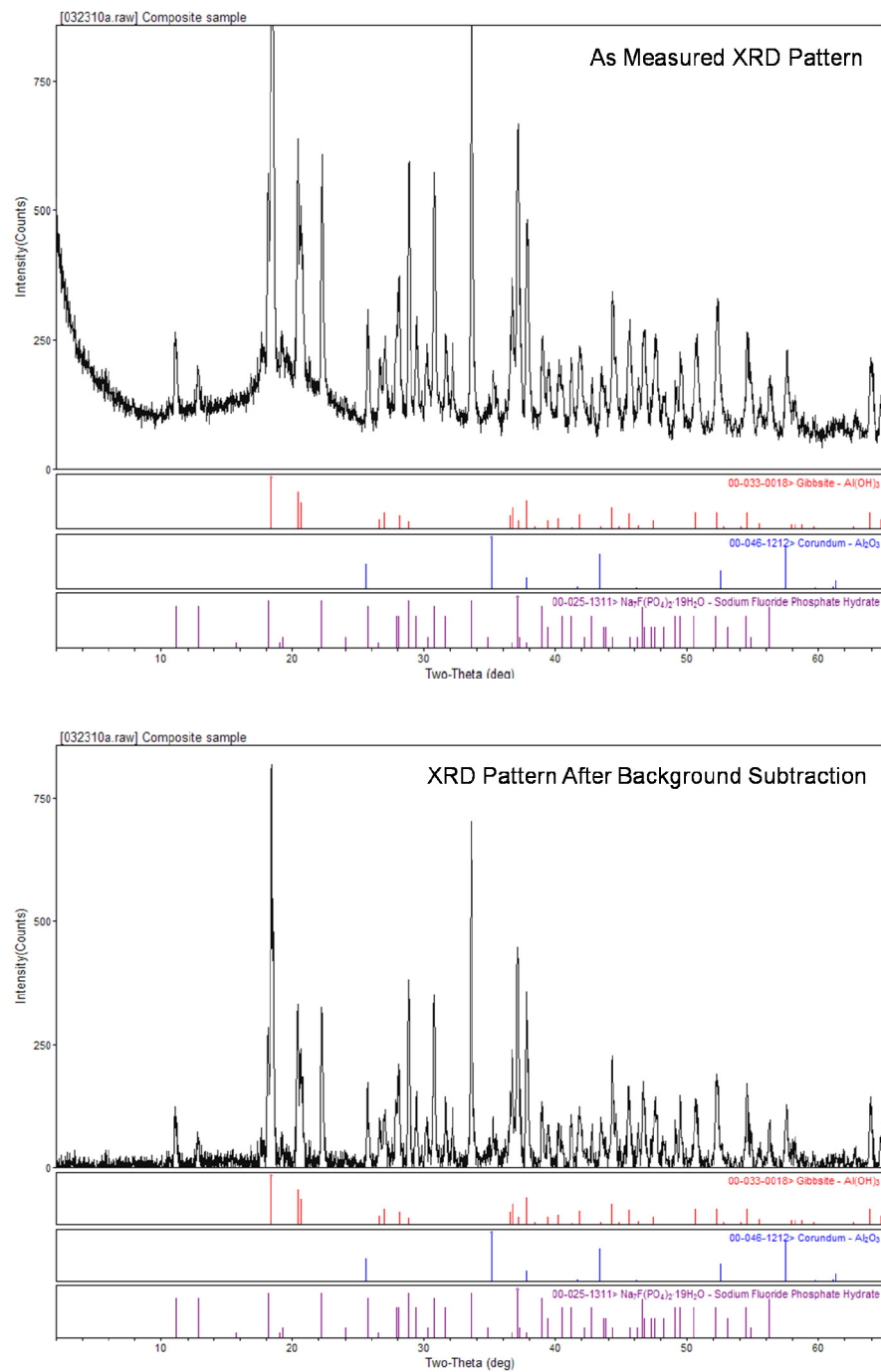
## Appendix B

### X-Ray Diffraction Patterns for Unleached and Leached Samples of Residual Waste from Tank C-108

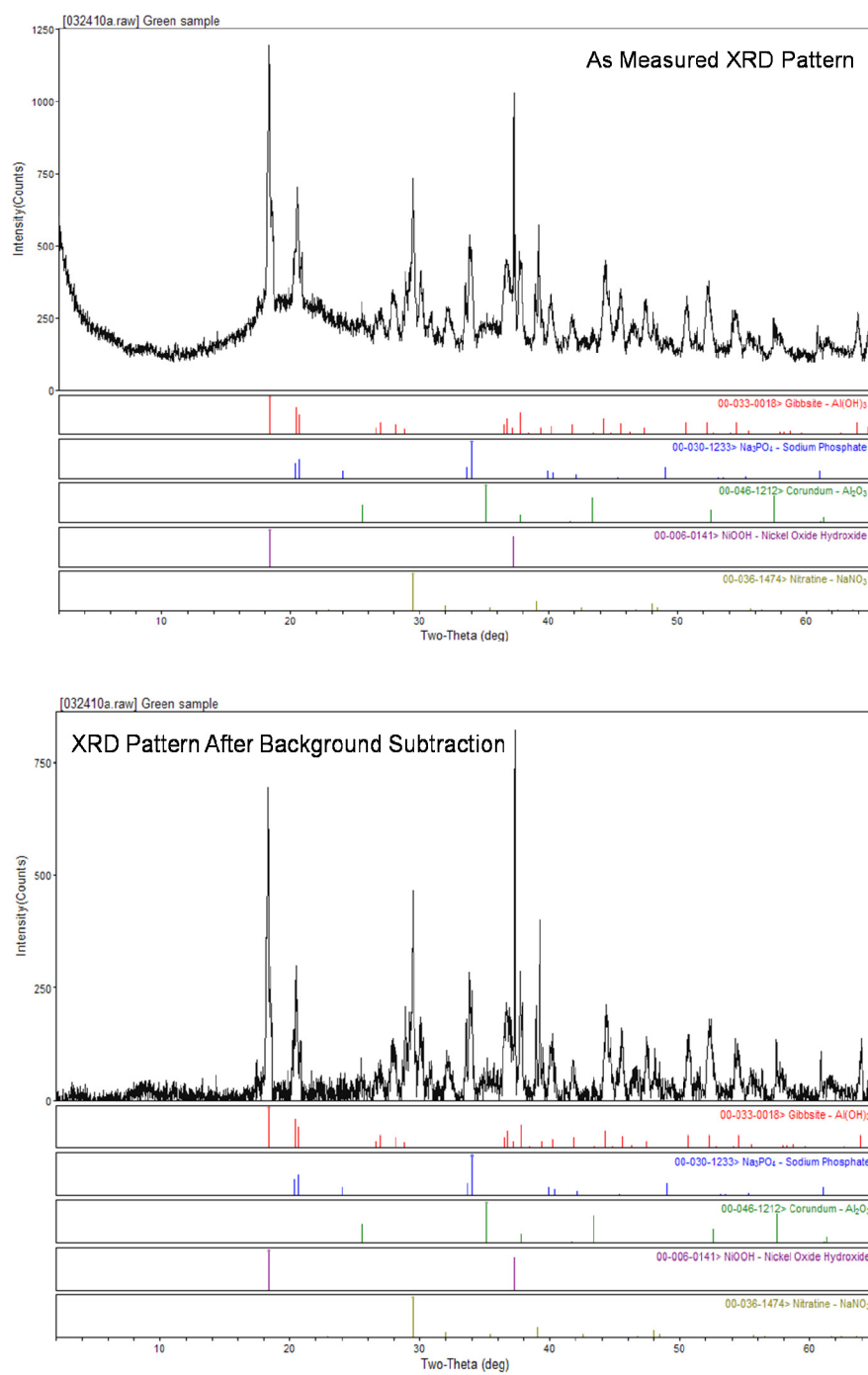
This appendix presents the as-measured and background subtracted x-ray powder diffraction (XRD) patterns for the unleached and leached samples of residual waste from tank C-108. The samples for which XRD patterns were measured include the following unleached and leached tank C-103 residual waste samples:

- Composite subsample – A mixture of all phases visible in the jar sample of as-received residual waste.
- Green nodule subsample – Handpicked particles of green colored solid(s) in the jar sample of as-received residual waste.
- Black nodule subsample – Handpicked particles of black colored solid(s) in the jar sample of as-received residual waste.
- Crystal nodule subsample – Handpicked particles of crystalline-appearing phase in the jar sample of as-received residual waste. This solid consists of rounded, dense-looking, tan grains that appear to consist primarily of a single phase and can be as much as a couple centimeters in largest dimension. In the jar sample, these grains usually have some light tan, fine grain material adhering to their surfaces.
- One month dionized (DI) water leached sample – Solid material remaining from the 1-month, single-contact DI water leach test.
- One month  $\text{Ca}(\text{OH})_2$  leached sample – Solid material remaining from the 1-month, single-contact  $\text{Ca}(\text{OH})_2$ -solution leach test.
- One month  $\text{CaCO}_3$  leached sample – Solid material remaining from the 1-month, single contact  $\text{CaCO}_3$ -solution leach test.

In Figures B.1 through B.7, the top XRD patterns show the as-measured pattern and the bottom patterns are those corrected for background subtraction. The instrumentation and procedures used for measuring, subtracting background, and interpreting the XRD patterns for these materials are described in Section 2.3 of the main report. The vertical axis in each of the following patterns represents the intensity in counts per second (cps) of the XRD peaks. The horizontal axis is in terms of degrees  $2\theta$  based on  $\text{Cu}_{K\alpha}$  radiation ( $\lambda=1.5406 \text{ \AA}$ ), and is related to  $d$  spacing according to the Bragg law (Cullity 1956). The XRD patterns show, for comparison purposes, the schematic database (PDF) pattern for corundum (used as a  $2\theta$  internal standard) and any other phases thought to be present in the sample mount. The height of each line in the schematic PDF patterns represents the relative intensity of an XRD peak (i.e., the most intense [the highest] peak has a relative intensity  $[I/I_0]$  of 100%).

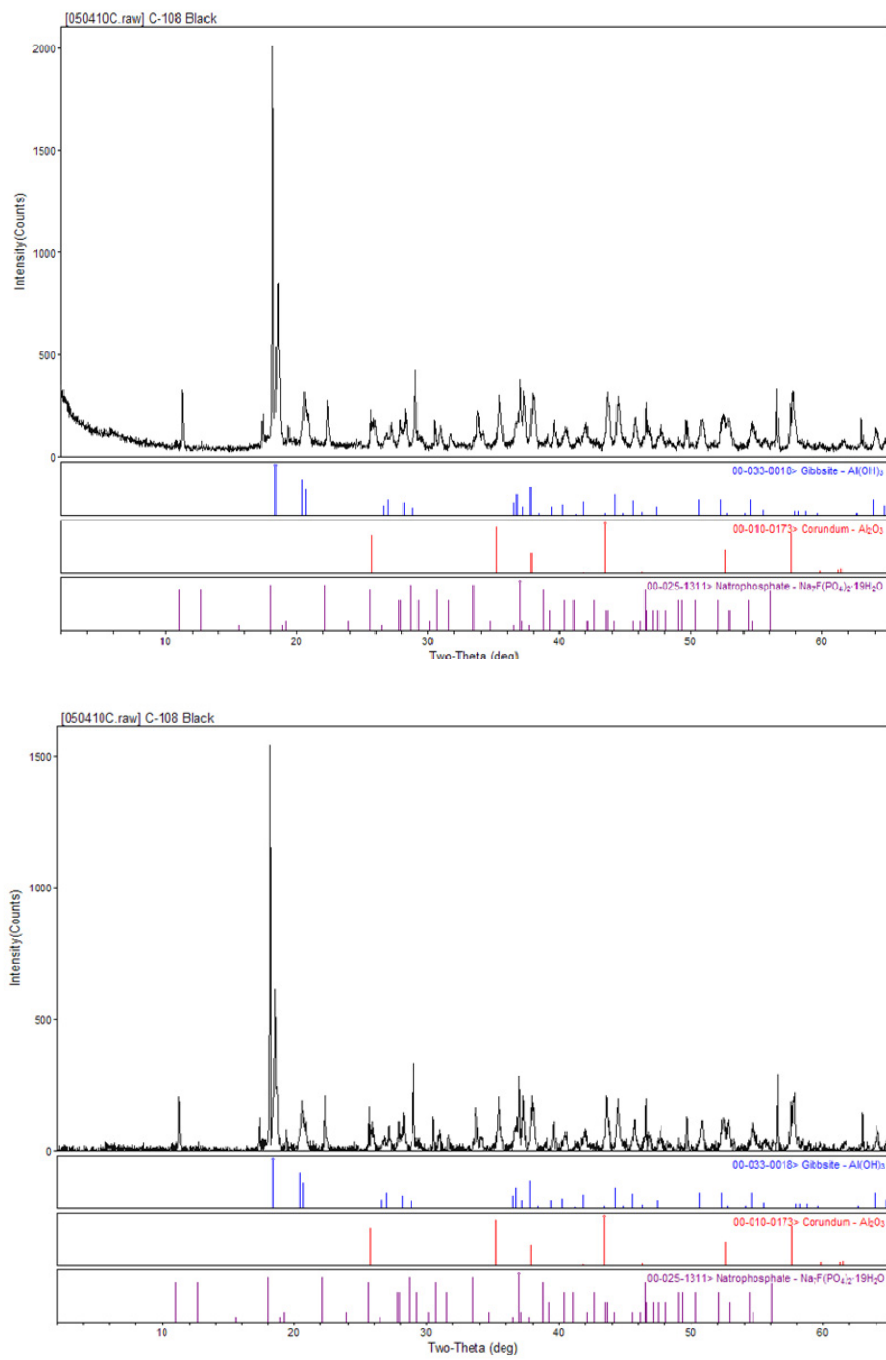


**Figure B.1.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for Composite Subsample from the As-Received Tank C-108 Residual Waste (Sample 20578)

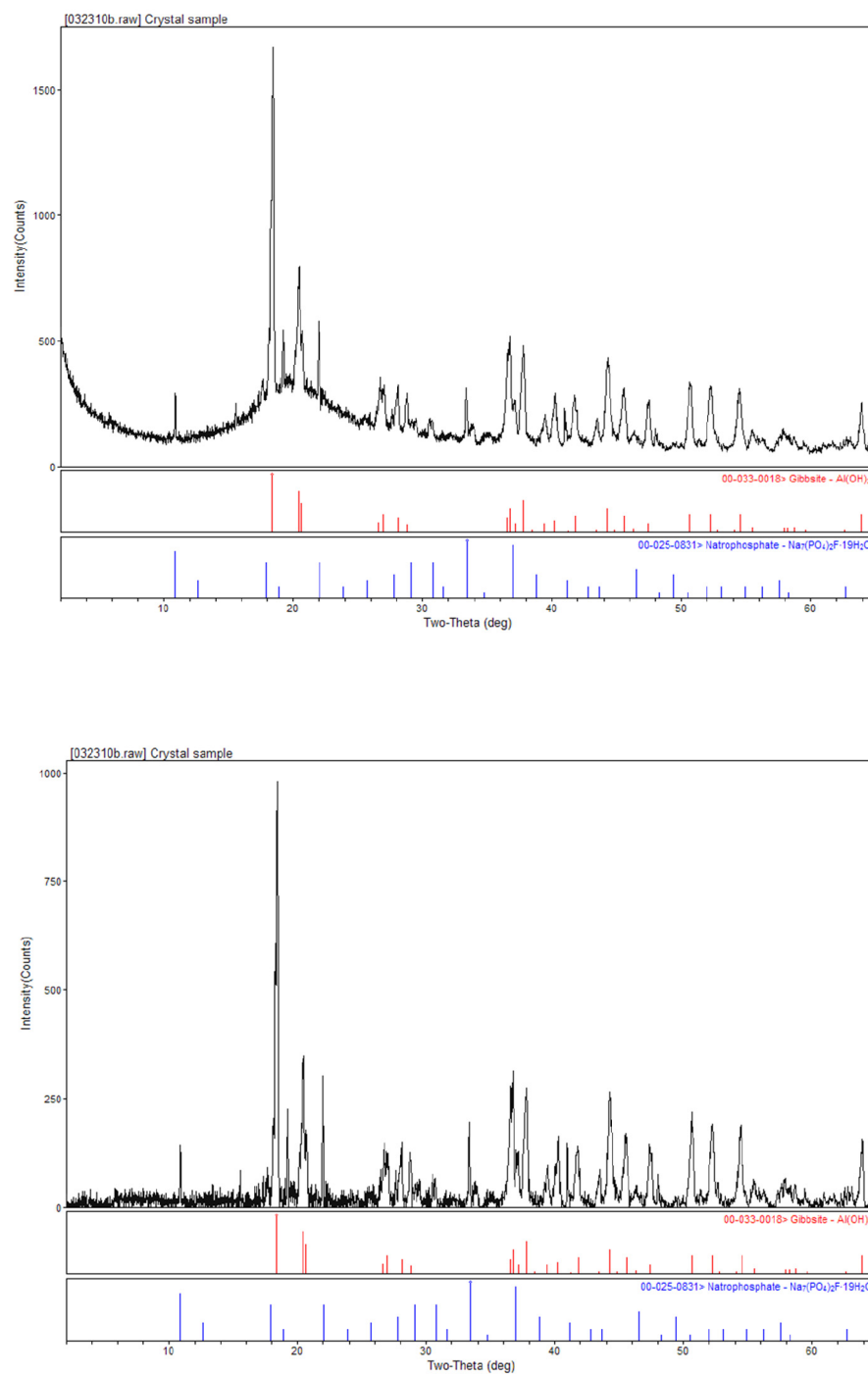


**Figure B.2.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for Green Nodule Subsample from the As-Received Tank C-108 Residual Waste (Sample 20578)

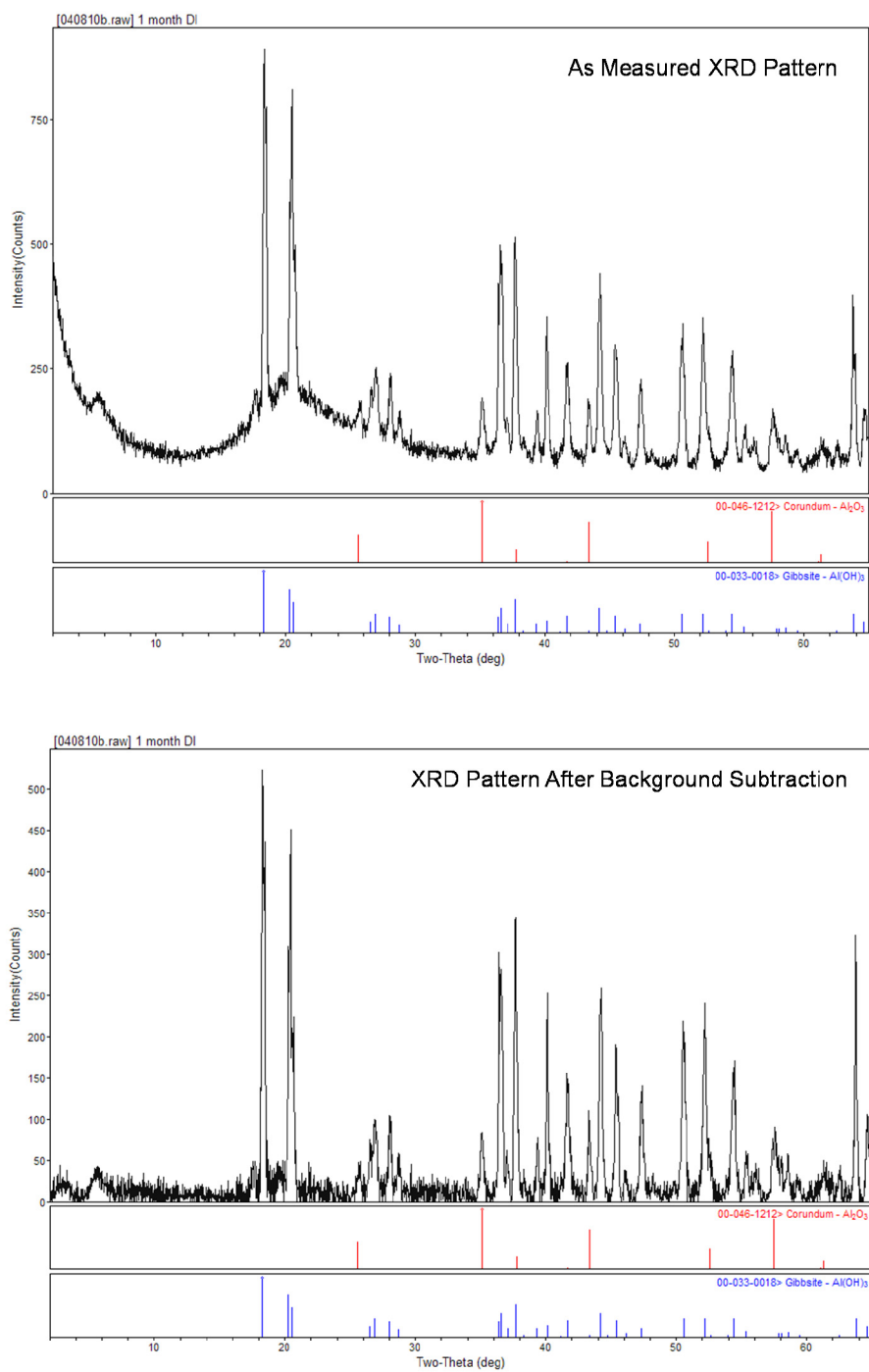




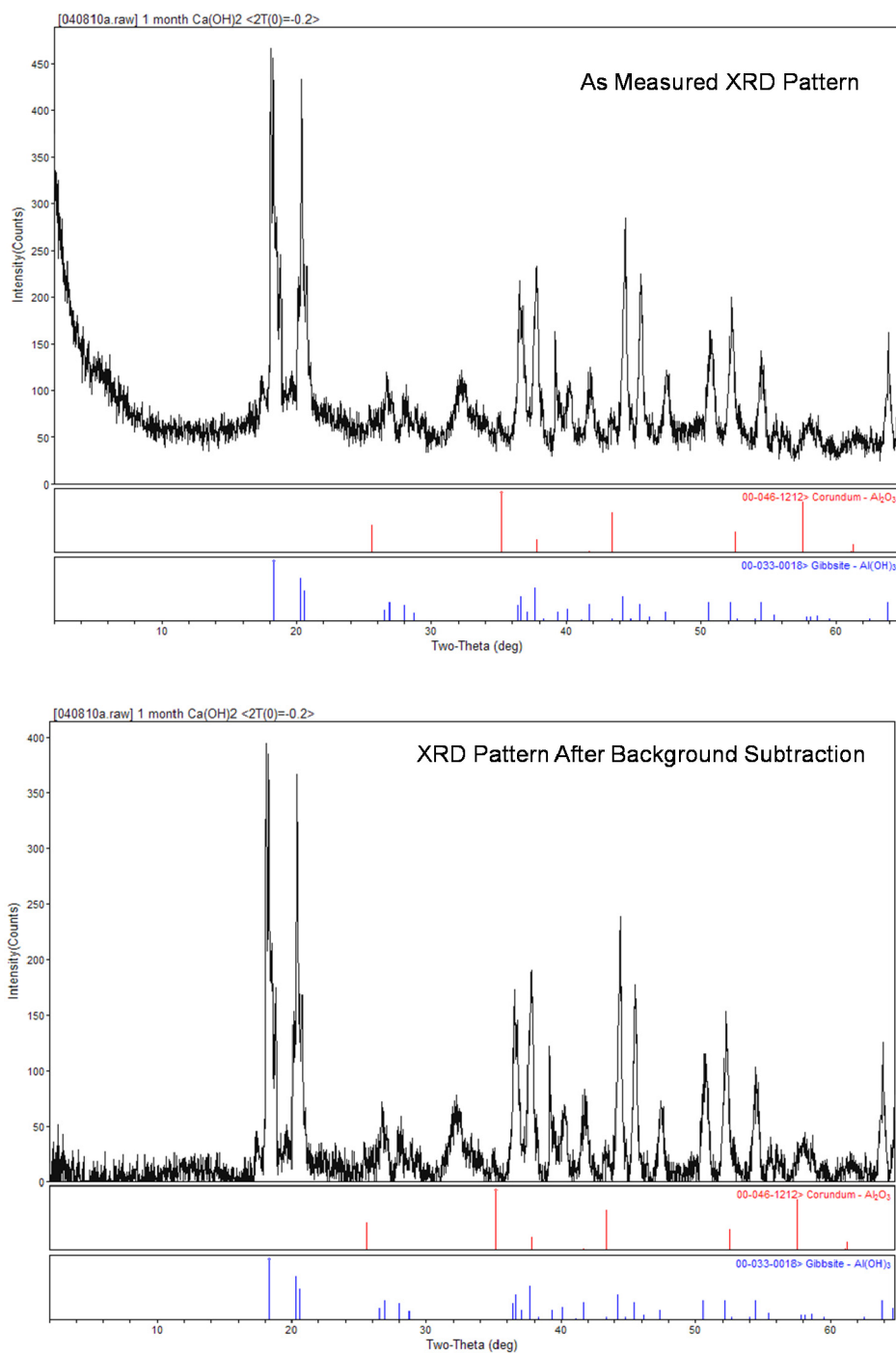
**Figure B.3.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for Black Nodule Subsample from the As-Received Tank C-108 Residual Waste (Sample 20578)



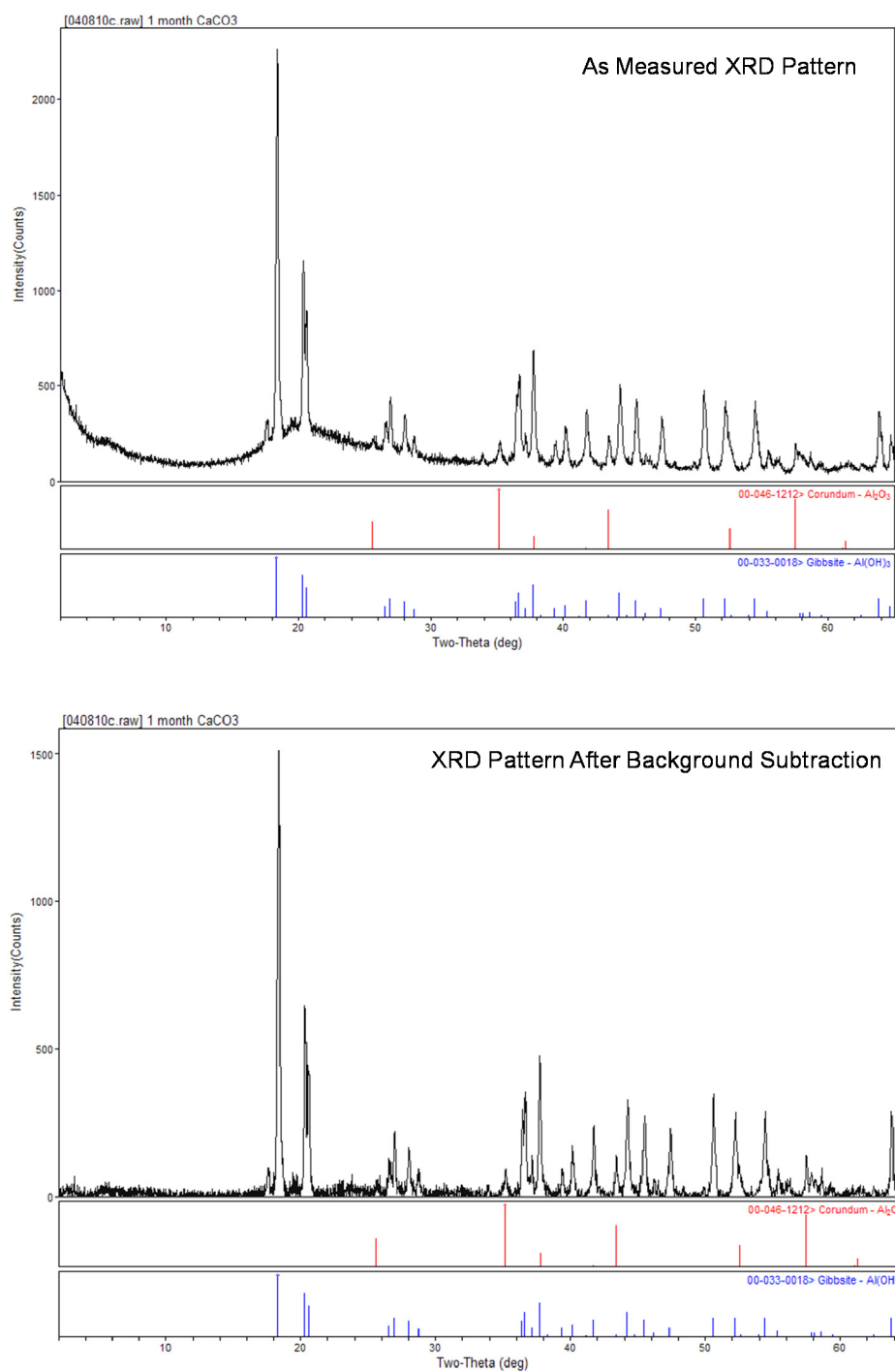
**Figure B.4.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for Crystal Nodule Subsample from the As-Received Tank C-108 Residual Waste (Sample 20578)



**Figure B.5.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for 1-Month DI Water Leached Tank C-108 Residual Waste (Sample 20578)



**Figure B.6.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for 1-Month Ca(OH)<sub>2</sub> Leached Tank C-108 Residual Waste (Sample 20578)



**Figure B.7.** As-Measured (top) and Background Subtracted (bottom) XRD Patterns for 1-Month CaCO<sub>3</sub> Leached Tank C-108 Residual Waste (Sample 20578)

## References

Cullity BD. 1967. *Elements of X-Ray Diffraction*. Addison-Wesley Publishing Company, Inc., Reading, Massachusetts.

Krupka KM, WJ Deutsch, MJ Lindberg, KJ Cantrell, NJ Hess, HT Schaef, and BW Arey. 2004. *Hanford Tanks 241-AY-102 and 241-BX-101: Sludge Composition and Contaminant Release Data*. PNNL-14614, Pacific Northwest National Laboratory, Richland, Washington.

## **Appendix C**

### **Scanning Electron Microscope Micrographs and Energy Dispersive Spectroscopy Results for Composite Subsample from Unleached Residual Waste from Tank C-108**

## **Appendix C**

### **Scanning Electron Microscope Micrographs and Energy Dispersive Spectroscopy Results for Composite Subsample from Unleached Residual Waste from Tank C-108**

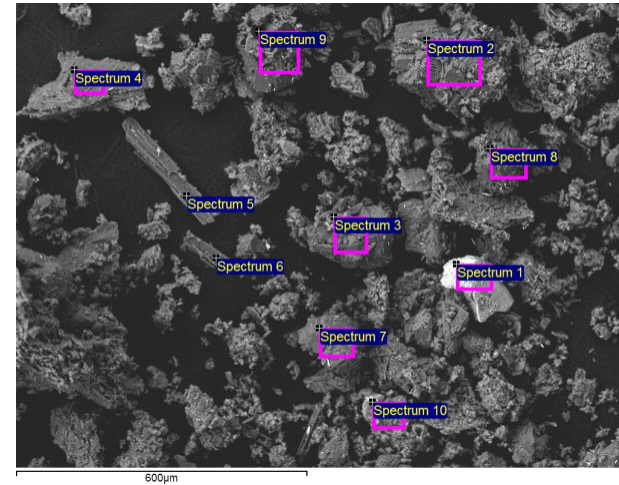
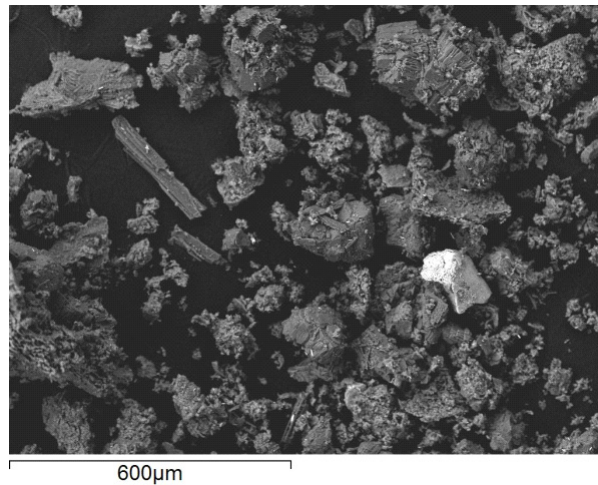
This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the composite subsample from tank C-108 unleached residual waste, and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the composite subsample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by x-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.



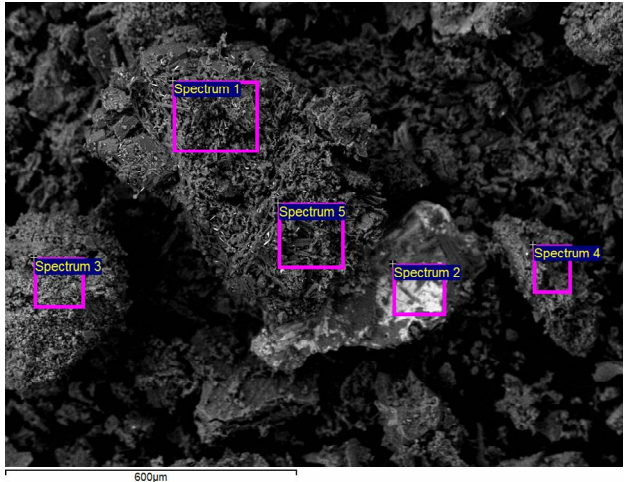
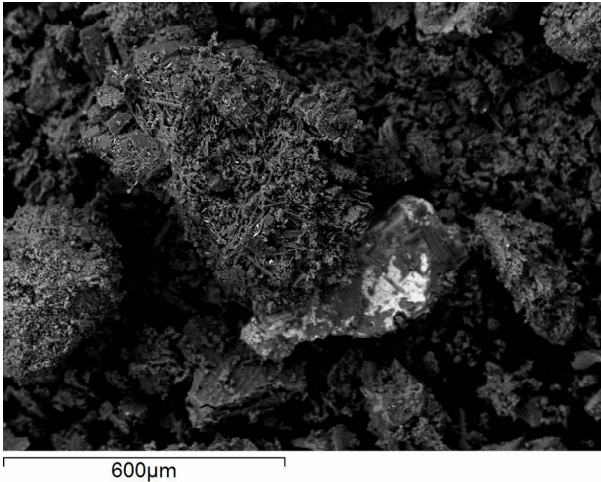
## Backscattered Electron (BSE) Micrograph of Particles Site 1 in Composite Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
at.% normalized to 100%														
1	0.7	15.7			66.6	7.2	1.1			0.1				Sr 8.5, S 0.1
2	17.9	8.2			72.8	1.1					0.1			
3	13.9	17.2			68.2	0.7								S 0.1
4	0.1	29.9			56.8	0.2								S 0.3, N 12.7
5		42.7			56.9	0.1								S 0.1
6	4.4	28.1			62.8	4.7								
7	17.9	4.9			76.6	0.5				0.1	0.1			
8	11.1	20.3			67.5	0.8				0.1	0.0			S 0.2
9	10.4	20.7			62.9	2.8	3.2			0.1				
10	0.9	30.7			51.6	11.5	5.3							

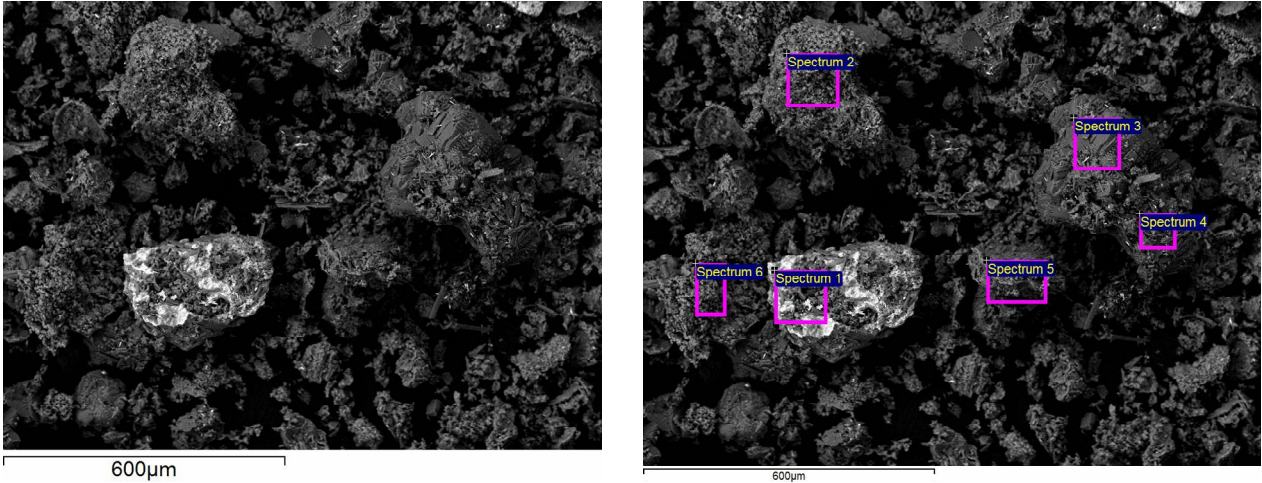
**Backscattered Electron (BSE) Micrograph of Particles at Site 2 in Composite Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron in SEM Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	10.4	23.4			65.2	0.7		0.1				0.2		S 0.2
2	1.6	21.1	0.2		67.4	1.5		1.8			2.0	0.3	1.0	Ti 1.9, Mg 1.2, S 0.1
3	1.4	28.3		0.5	54.8	9.3	5.7							
4	0.1	32.2			51.9	7.6	8.1							
5	2.7	34.9			60.3	1.7					0.1			S 0.3

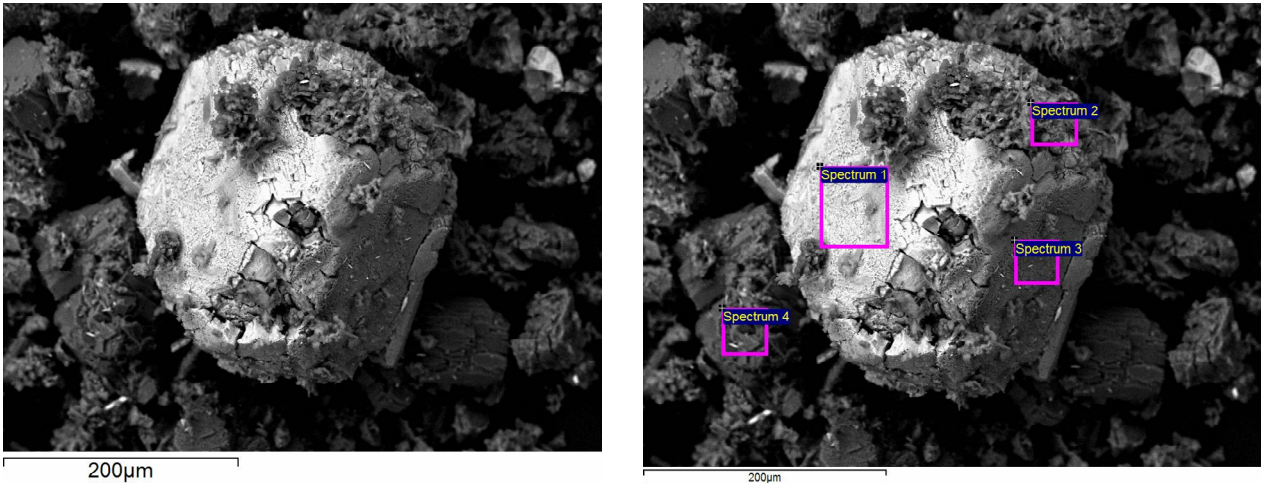
**Backscattered Electron (BSE) Micrograph of Particles at Site 3 in Composite Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	5.2	25.7			59.7	2.2		0.4			6.3	0.3		S 0.2
2	0.3	32.2			51.3	10.2	6.2							
3	17.1	11.8			70.2	0.8						0.1		
4	2.0	36.0			60.6	0.9		0.1			0.1	0.1		S 0.2
5		32.2			52.8	7.8	7.1							
6	0.2	33.1			52.2	7.0	7.5							S 0.1

Backscattered Electron (BSE) Micrograph of Particles at Site 4 in Composite Subsample of C-108 Residual Waste

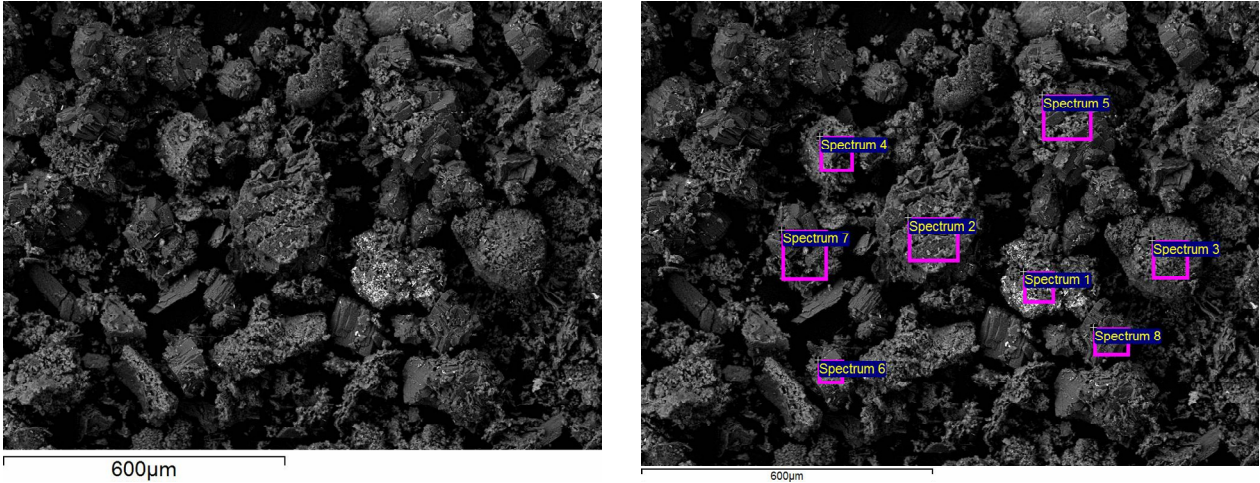


Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	4.4	27.7			63.3	4.3								Sr 0.3
2	0.3	25.9		0.1	58.7	7.5	4.7							Sr 2.9
3	1.0	18.6			66.6	7.4						0.2		Sr 6.3
4	5.5	26.0			64.0	4.6								



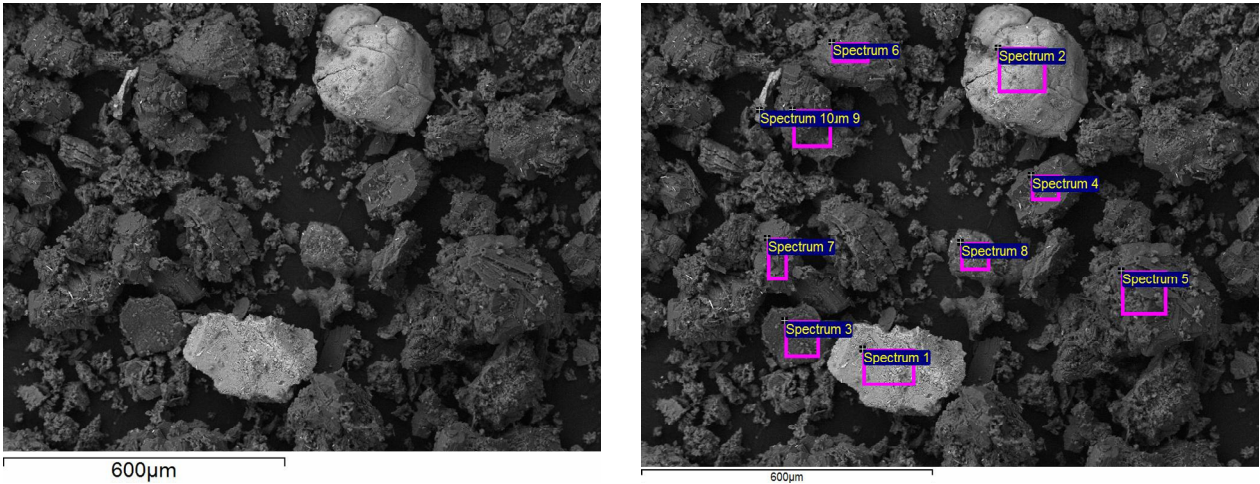
**Backscattered Electron (BSE) Micrograph of Particles at Site 5 in Composite Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron in SEM Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	10.4	17.4			69.5	1.5		0.9				0.4		
2	0.3	31.9			52.8	10.4	4.7							
3	0.5	32.0			55.5	7.7	4.4							
4	0.3	32.5			51.8	8.7	6.7							
5	13.4	16.0			64.8	3.3	2.5							
6	19.1	6.1			74.5	0.4								
7	16.7	9.6			71.9	1.2		0.1			0.2	0.3		
8	18.3	8.1			72.9	0.6								S 0.1

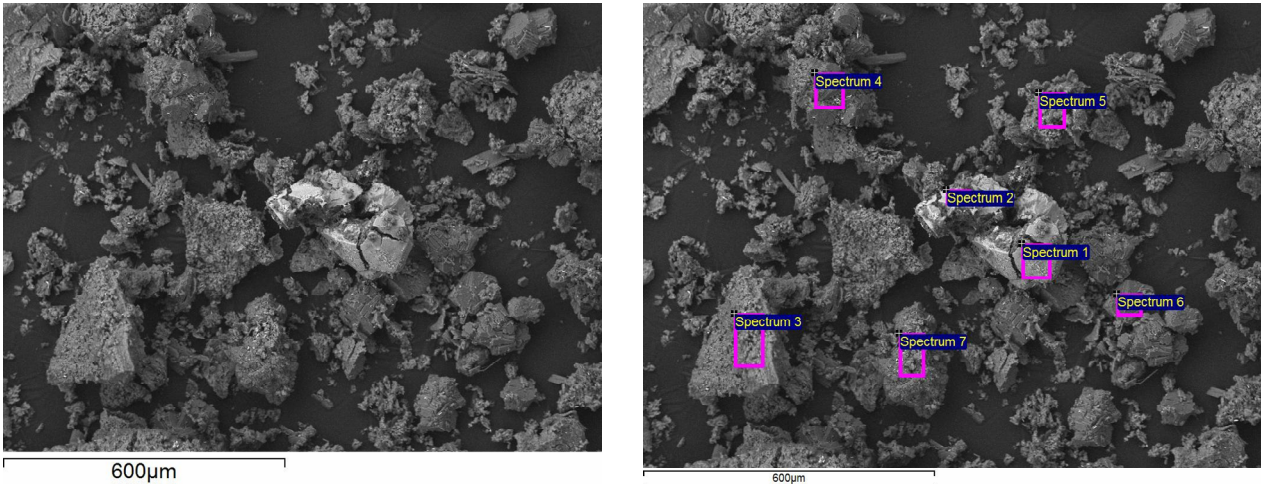
Backscattered Electron (BSE) Micrograph of Particles at Site 6 in Composite Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	4.8	21.4			65.3	4.6					0.6	0.7		Sr 2.7
2		17.2			63.0	10.3								Sr 9.6
3	0.6	32.0			51.4	11.3	4.8							
4	2.6	30.6			52.1	7.0	7.2					0.5		
5	5.5	32.8			60.4	1.1								S 0.1
6	0.2	33.1			51.7	9.1	5.8							
7	16.9	12.0			69.9	1.1					0.1			
8	2.3	29.6			61.6	3.0	1.4	0.1				1.6		S 0.4
9	6.2	25.0			61.6	4.9	2.3							
10	6.2	25.8			63.7	4.3								

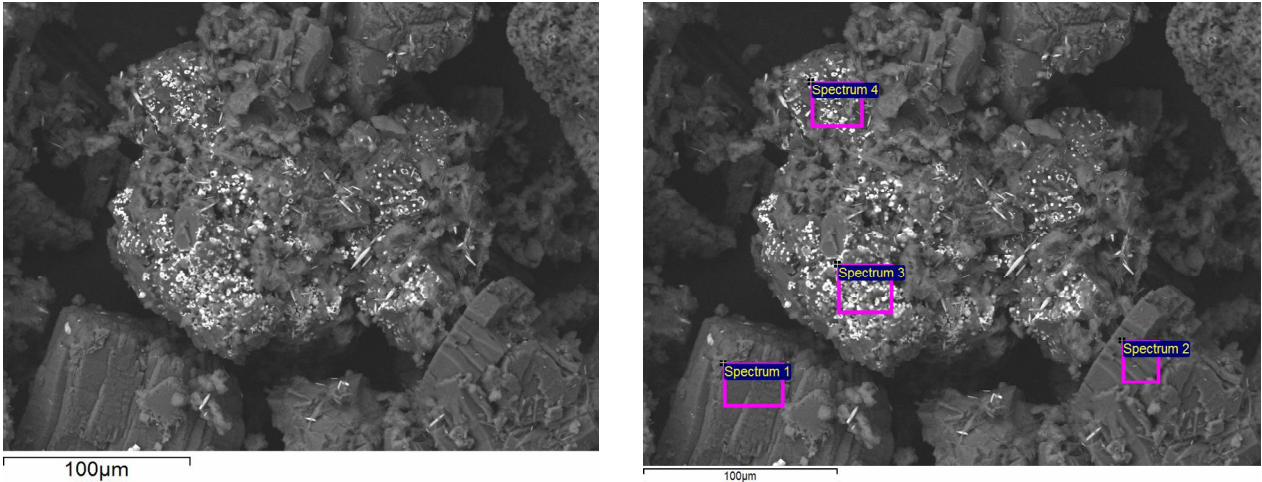
**Backscattered Electron (BSE) Micrograph of Particles at Site 7 in Composite Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.7	17.9			64.0	8.8								Sr 8.6
2	0.4	17.6			67.4	7.8								Sr 6.9
3	0.8	32.2			51.3	8.9	6.7							
4	14.2	13.8			70.1	1.8					0.1	0.1		
5		33.8			52.0	8.0	6.2							
6	17.8	6.9			74.6	0.7								
7	13.8	15.9	0.0		67.6	2.4					0.1	0.1		S 0.2

**Backscattered Electron (BSE) Micrograph of Particles at Site 8 in Composite Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	23.9	3.8			71.9	0.4								
2	20.3	1.3			78.1	0.3								
3	13.4	11.0	0.4		72.2	1.3		1.7						
4	16.1	11.9			69.5	1.6		1.0						



## **Appendix D**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Green Nodule Subsample from Unleached Residual Waste from Tank C-108**

## Appendix D

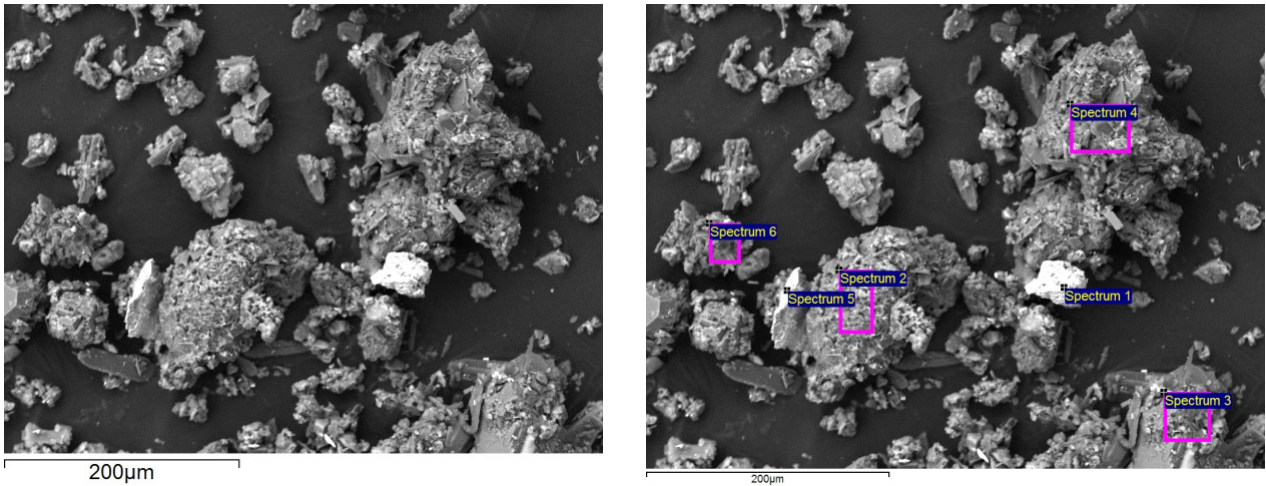
### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Green Nodule Subsample from Unleached Residual Waste from Tank C-108**

This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the green nodule subsample from tank C-108 unleached residual waste, and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in green nodule subsample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.

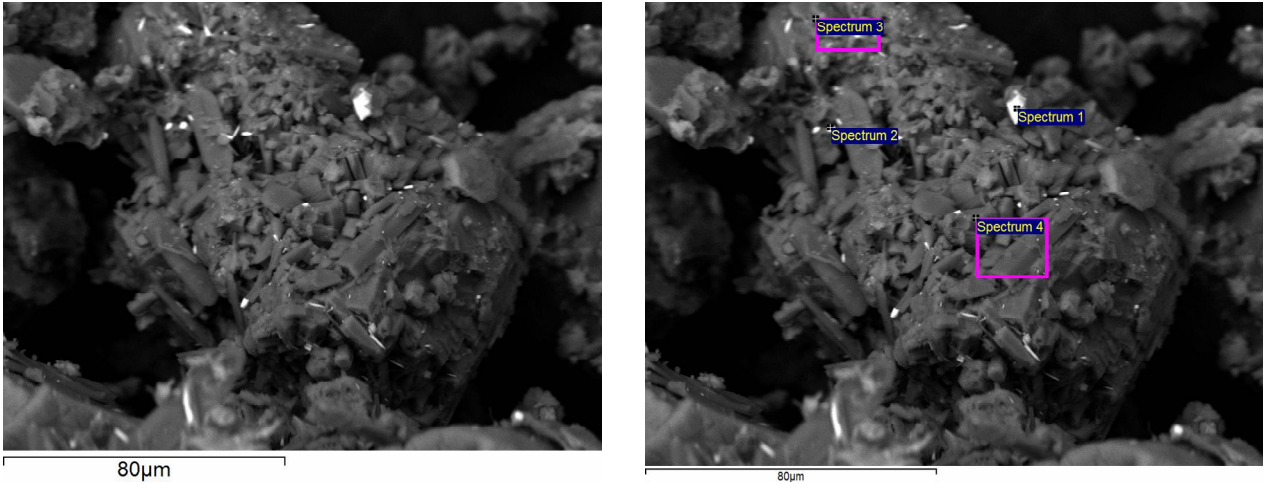
Backscattered Electron (BSE) Micrograph of Particles at Site 1 in Green Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.1	12.2	0.2		52.8	3.4					26.5	0.4		Pb 1.0, K 0.2, Cl 0.2
2	5.8	28.7		0.3	56.2	2.8		0.1			0.3	5.8		
3	16.3	6.5			76.0	0.6		0.0			0.1	0.3		Sr 0.1, S 0.1
4	11.9	16.5			68.9	1.6					0.1	0.9		S 0.1
5	1.9	8.9			59.2	0.6					29.1	0.3	0.1	
6	5.6	25.4		0.1	64.8	2.1		0.0			0.1	1.7		S 0.1

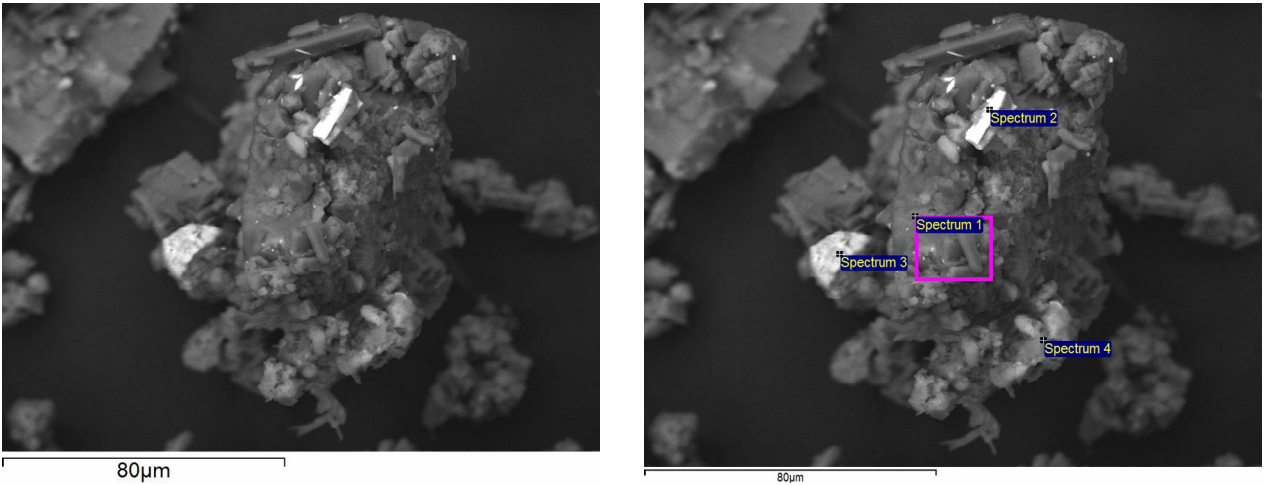
**Backscattered Electron (BSE) Micrograph of Particles at Site 2 in Green Nodule Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	2.6	15.1		0.5	74.4	1.8		3.7				1.8		S 0.1
2	2.2	20.1	0.1		68.1	1.7	2.2	4.8				0.5		Cu 0.4
3	13.8	15.9			68.8	0.6		0.4			0.2	0.4		
4	8.7	21.4			69.5	0.3						0.1		

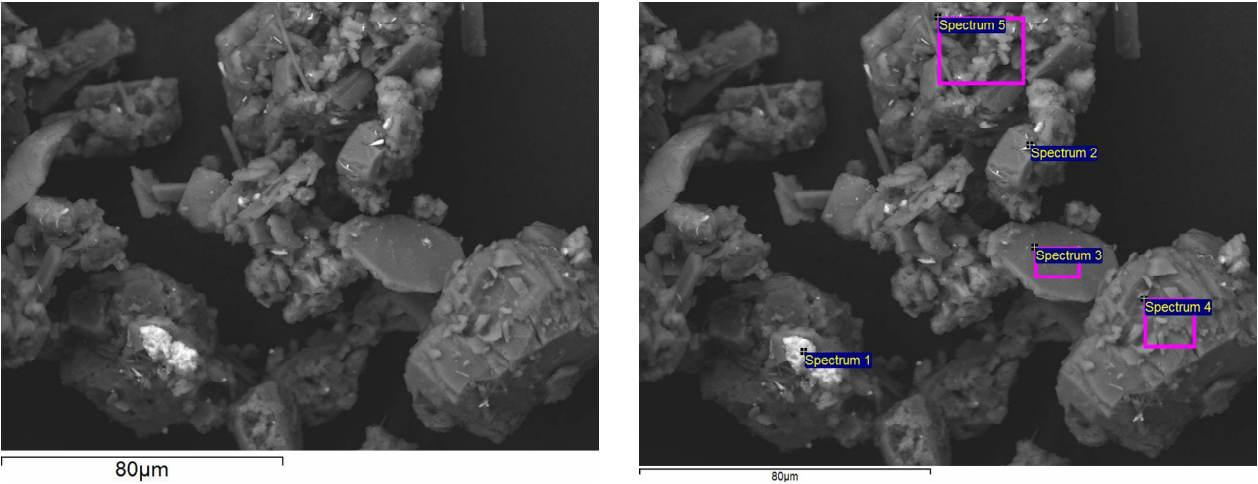
**Backscattered Electron (BSE) Micrograph of Particles at Site 3 in Green Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	17.6	9.0		0.1	71.1	0.8					0.1	1.4		
2	1.9	15.9			70.8	0.7		9.9				0.8		
3	4.3	18.6			31.9	1.1					41.8	1.0		Cu 0.7
4	5.6	22.5			53.0	7.0					0.2	11.2		K 0.1

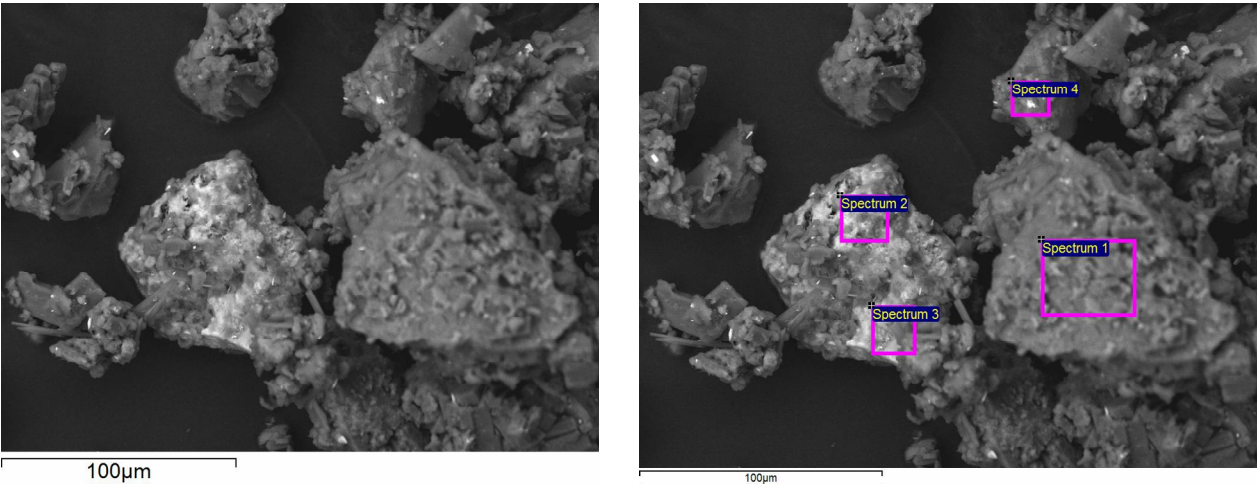
**Backscattered Electron (BSE) Micrograph of Particles at Site 4 in Green Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	2.3	11.5			74.8	1.4		6.8				3.1		
2	31.8	4.4			60.5	1.5		0.5				1.3		
3	24.2	2.2			73.0	0.4						0.2		
4	23.7	2.2			73.9	0.2								
5	5.5	23.1		0.2	67.0	1.8		0.1			0.1	2.1		S 0.2

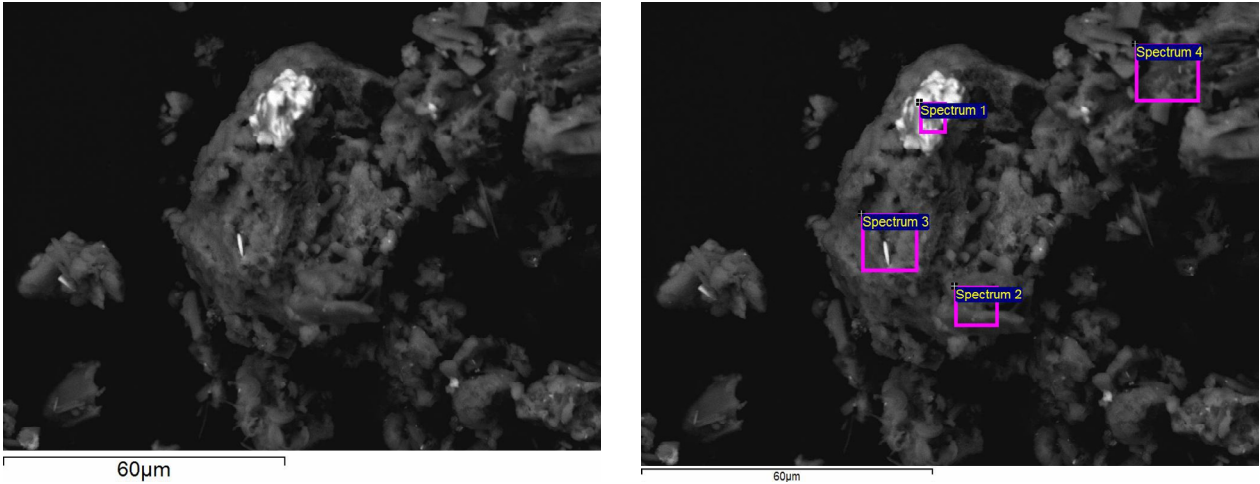
**Backscattered Electron (BSE) Micrograph of Particles at Site 5 in Green Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	5.7	23.9			57.3	7.3					0.3	4.9		S 0.7
2	2.2	24.3	0.4	0.1	56.3	2.3					13.3	0.5		Pb 0.5, Zn 0.2, K 0.1
3	3.9	18.7			36.0	0.8					33.2	7.5		Pb 0.1
4	1.6	32.3			48.8	10.2	5.7	0.2			0.2	1.1		

Backscattered Electron (BSE) Micrograph of Particles at Site 6 in Green Nodule Subsample of C-108 Residual Waste

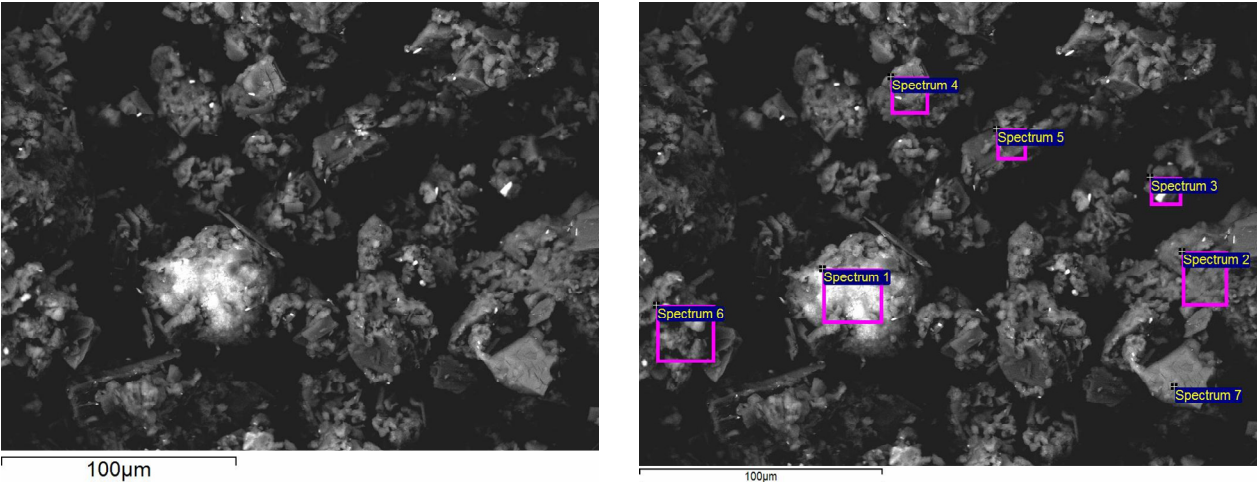


Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.2	20.3			71.5	0.8		3.9				0.3		
2	2.3	29.4		0.1	64.3	1.0		0.0			0.1	2.0		K 0.1, S 0.7
3	4.1	24.5			48.9	2.9		0.2			0.4	18.4		K 0.3, S 0.2, Cl 0.2
4	2.6	31.0			62.4	1.1		0.1				2.7		S 0.1



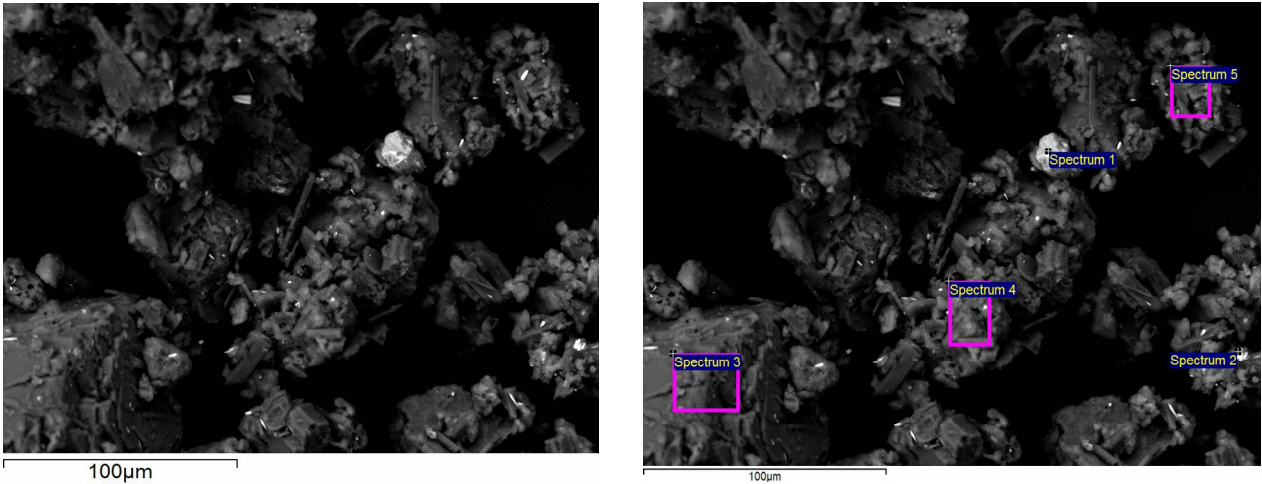
Backscattered Electron (BSE) Micrograph of Particles at Site 7 in Green Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	4.0	17.1			70.7	1.5		2.5			0.1	4.1		S 0.1
2	4.3	25.4			55.9	1.7					0.6	12.0		
3	2.4	29.6			58.5	3.4	3.6	0.4			0.1	1.8		S 0.3
4	1.0	34.0			48.3	9.1	5.9				0.2	0.7		S 0.8
5	2.3	38.5			52.2	0.9						6.1		
6	6.6	22.0		0.1	64.2	2.4					0.3	3.9		K 0.1, S 0.5
7		33.6			43.5	19.2	3.7							

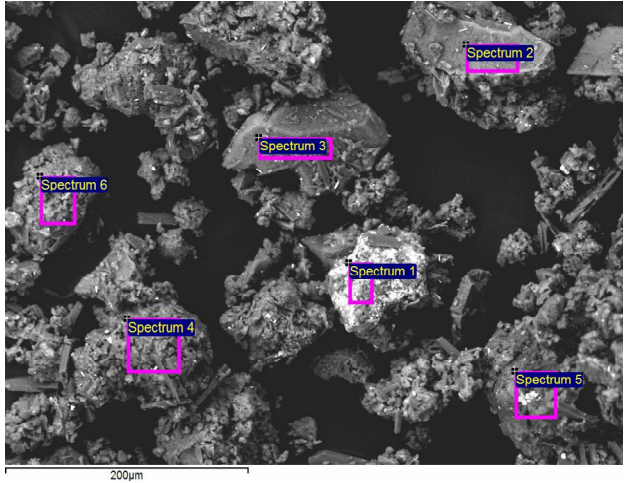
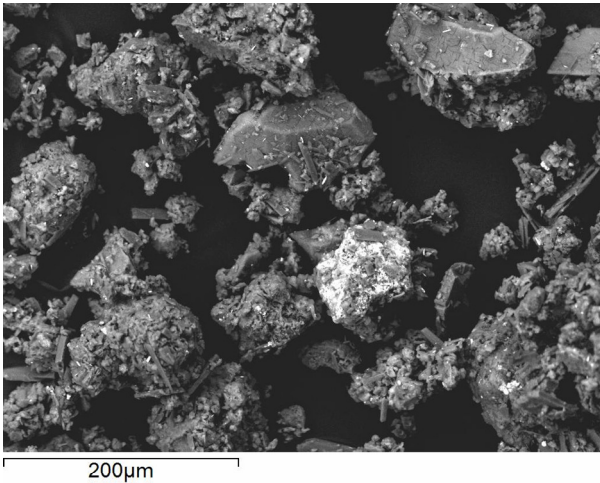
Backscattered Electron (BSE) Micrograph of Particles at Site 8 in Green Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.4	14.2			75.5	1.6		2.8				2.4		S 0.1
2	1.4	22.5			72.0	0.6		3.2				0.4		
3	19.3	6.6			73.3	0.4						0.4		
4	5.8	25.6	0.2		65.3	1.4						1.6		S 0.1
5	4.3	26.6			62.1	2.7		0.1			0.1	4.0		

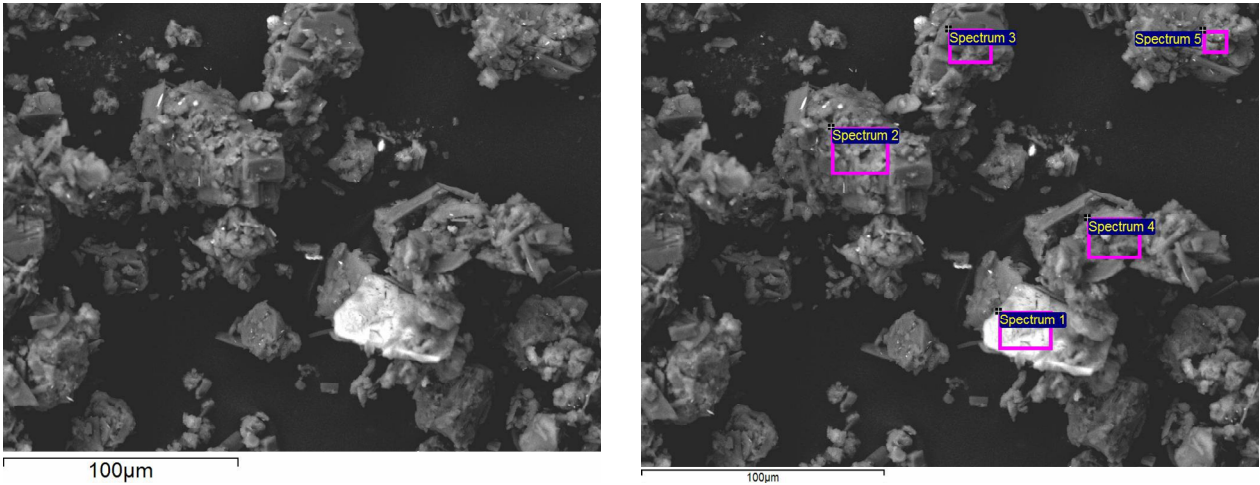
Backscattered Electron (BSE) Micrograph of Particles at Site 9 in Green Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	6.1	13.8	2.1	0.1	65.6	1.8					7.7	0.6		Pb 0.5, Mg 1.6, K 0.1
2	1.1	31.3			46.8	16.2	3.9	0.2				0.4		
3	0.4	32.4			52.7	10.6	3.6				0.1	0.2		
4	13.5	14.6			69.9	0.9		0.0				1.1		S 0.1
5	10.2	20.2			59.9	1.0		0.3			3.5	5.0		
6	4.5	24.9			61.6	2.0					0.2	5.7		K 0.2, S 0.9, Cl 0.1

Backscattered Electron (BSE) Micrograph of Particles at Site 10 in Green Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.8	11.3			63.4	13.5						0.3		Sr 10.7
2	10.8	18.9			66.5	1.6					0.1	1.7		S 0.4, Cl 0.1
3	18.8	4.7			75.3	0.7						0.6		
4	8.4	22.3			61.4	3.7					0.8	2.1		S 1.4
5	2.8	28.6		0.1	58.1	4.5	3.2	0.1			0.1	2.5		S 0.2

## **Appendix E**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Black Nodule Subsample from Unleached Residual Waste from Tank C-108**

## **Appendix E**

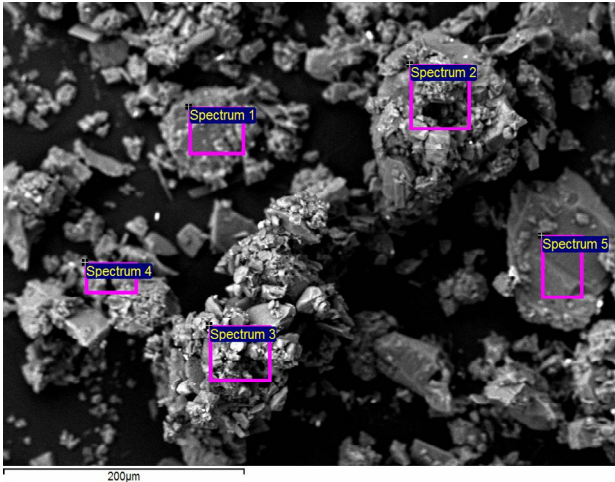
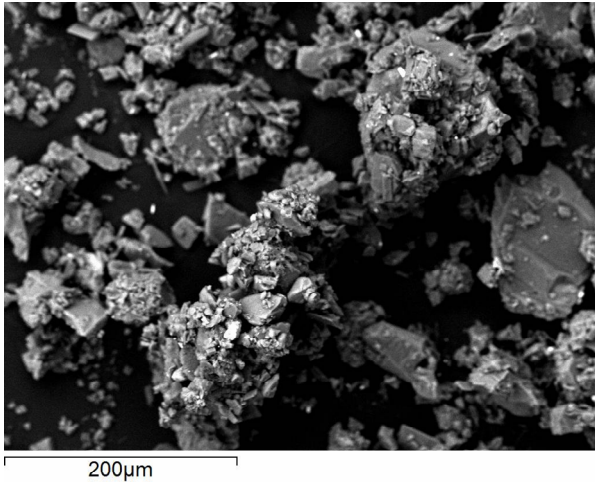
### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Black Nodule Subsample from Unleached Residual Waste from Tank C-108**

This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the black nodule subsample from tank C-108 unleached residual waste and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the black nodule subsample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.

**Backscattered Electron (BSE) Micrograph of Particles at Site 1 in Black Nodule Subsample of C-108 Residual Waste**

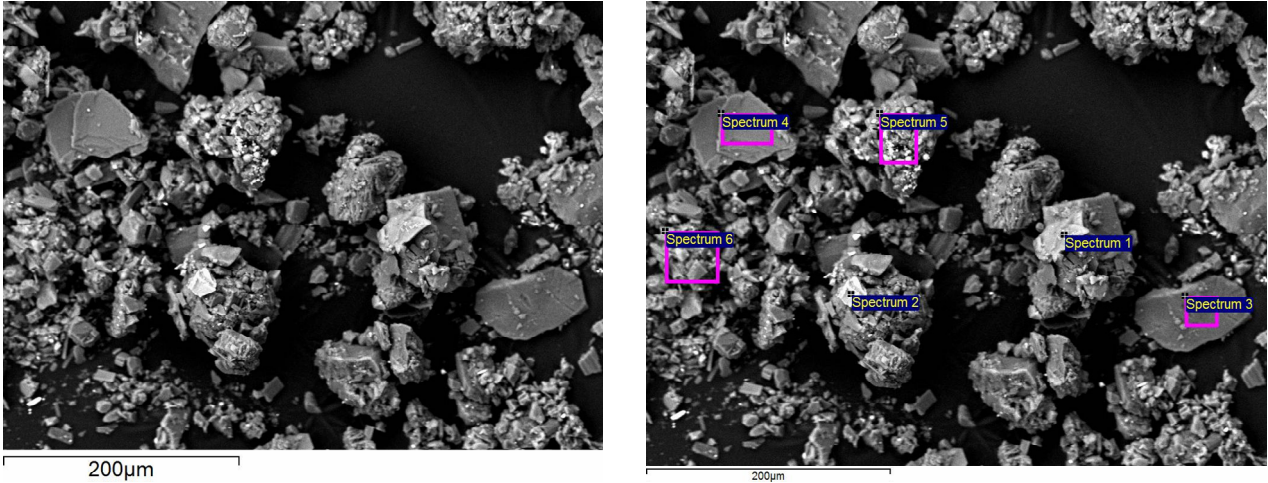


**Element Compositions Derived from EDS Analyses of Particles Identified in Electron in SEM Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	15.3	11.8			71.6	1.2		0.0			0.1			
2	10.9	16.7			66.6	3.2	2.4	0.1			0.1	0.1		S 0.1
3	9.4	17.1	0.1	0.2	68.2	2.6					2.1	0.1	0.1	S 0.1
4	4.3	24.4	0.1		64.0	4.1					2.8	0.1		Pb 0.1, Cu 0.1, S 0.1
5	0.2	23.5			65.5	5.6	5.2				0.0			



**Backscattered Electron (BSE) Micrograph of Particles at Site 2 in Black Nodule Subsample of C-108 Residual Waste**

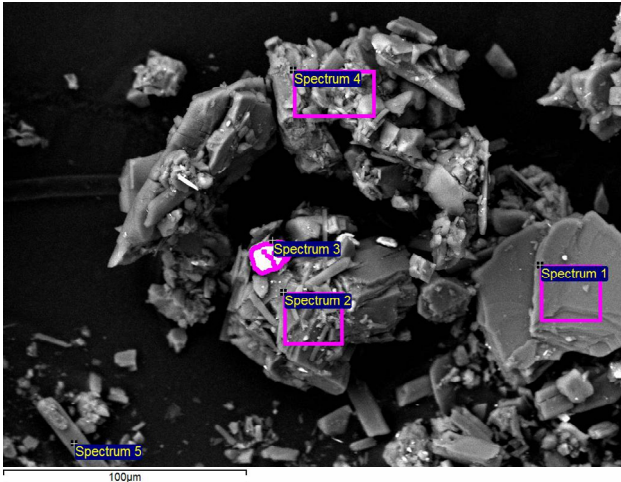
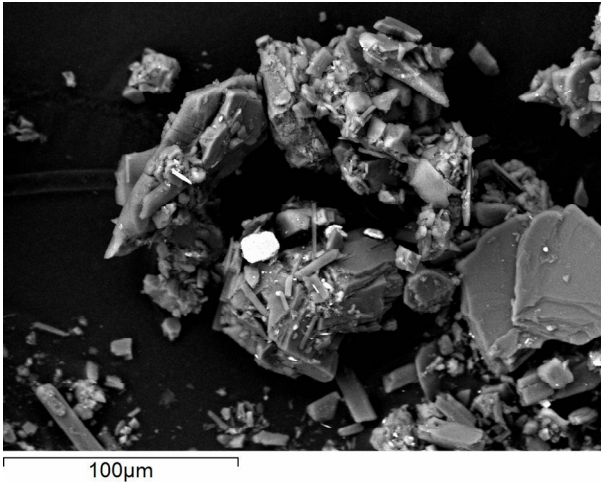


**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.3	22.9	0.2		57.2	14.6	1.7				0.1			
2	6.3	1.5	14.4	25.4	49.2								0.2	Ti 0.3, Mg 1.8, K 0.3, S 0.6
3	0.4	20.6			69.0	6.0	4.0							
4	23.6	1.1			75.2	0.2								
5	11.5	9.7	2.0	1.6	72.9	1.4					0.4	0.1		Mg 0.2, S 0.2
6	14.0	12.2			68.8	1.7	2.9				0.1	0.1	0.1	



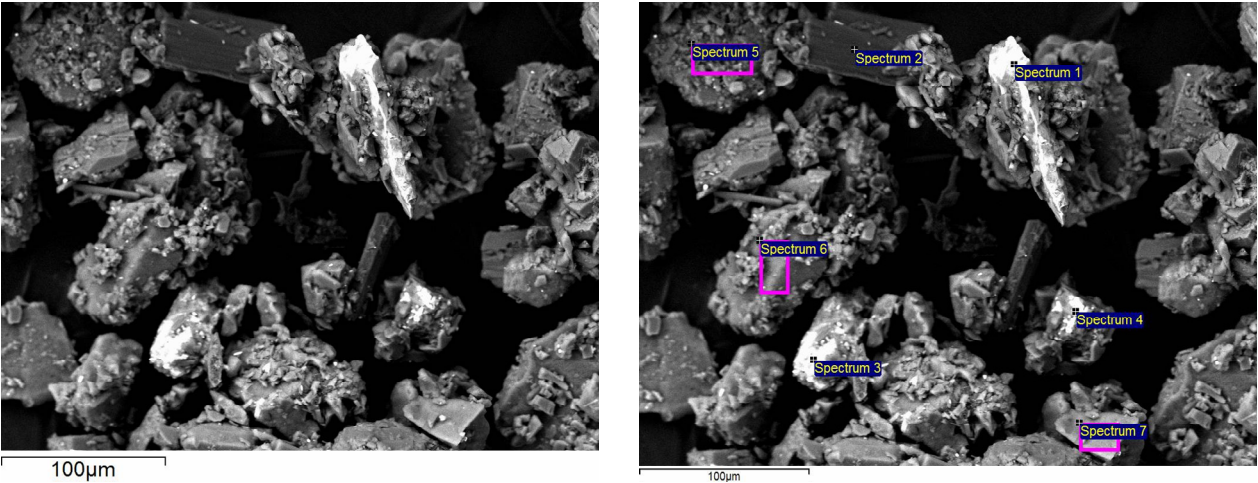
**Backscattered Electron (BSE) Micrograph of Particles at Site 3 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	23.9	1.1			74.9	0.2								
2	17.1	10.0			72.0	0.6					0.4			
3	2.9	10.9	0.2		65.5	2.2					16.7	0.1	0.2	Pb 1.1, Zn 0.2, K 0.1
4	9.5	18.6			68.3	2.0	1.2				0.1	0.1		S 0.1
5	4.0	27.5			63.8	2.0	2.1				0.3			S 0.3

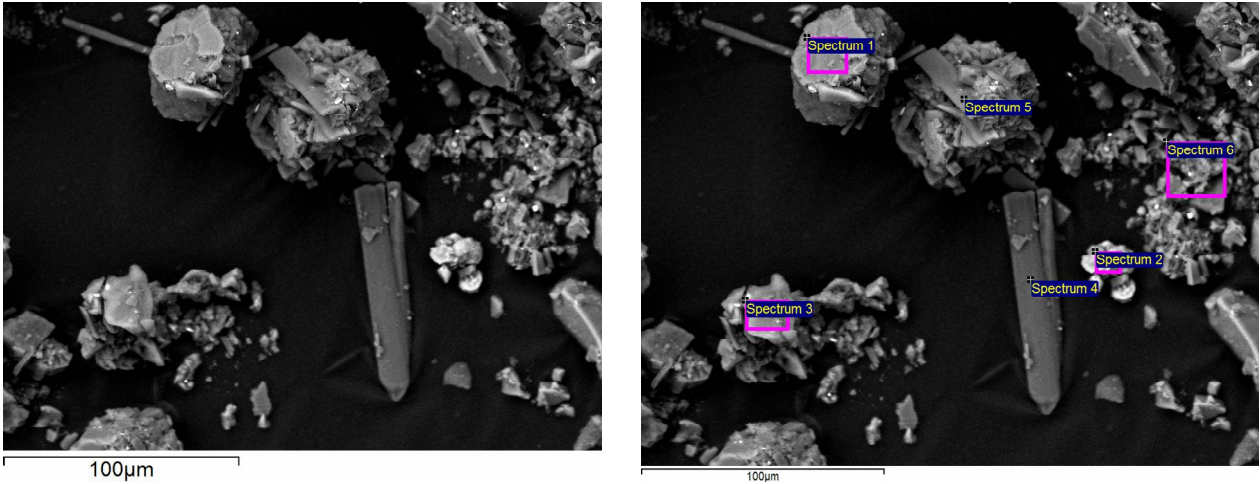
**Backscattered Electron (BSE) Micrograph of Particles at Site 4 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	4.2	8.2			58.9	1.0					27.4		0.1	Cl 0.1
2		37.3			62.7									
3	1.8	13.6			57.9	1.2					25.2	0.1	0.2	Pb 0.1, S 0.1
4	16.2	8.5	0.2		67.1	3.3	3.2	1.3			0.1	0.1		Cu 0.1
5	16.1	10.9	0.1		71.2	1.4					0.1	0.1	0.1	
6	10.1	14.3			69.2	5.3					1.0			
7	1.2	28.4	0.3	0.1	57.2	6.2	6.7				0.0			

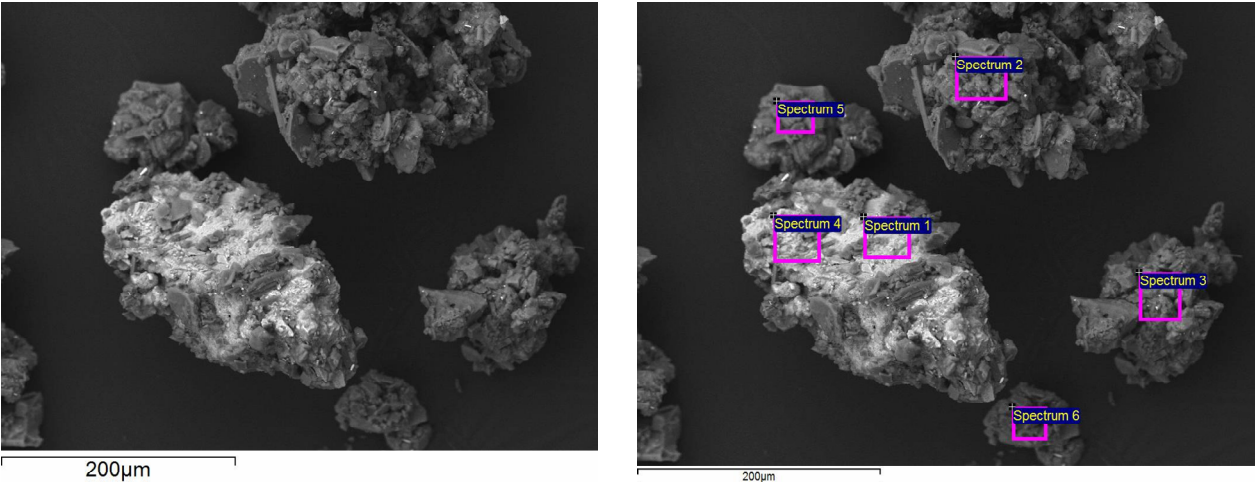
**Backscattered Electron (BSE) Micrograph of Particles at Site 5 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	22.0	1.8			76.0	0.3								
2	9.7	14.7	8.0	0.0	64.6	1.2		0.1			1.6		0.1	S 0.1
3	2.0	30.0			53.2	6.3	8.4							
4		35.2			63.5									S 1.4
5	11.3	21.8			62.9	1.7					1.2	1.0		
6	7.3	22.4			65.5	2.9	1.8	0.0						S 0.2

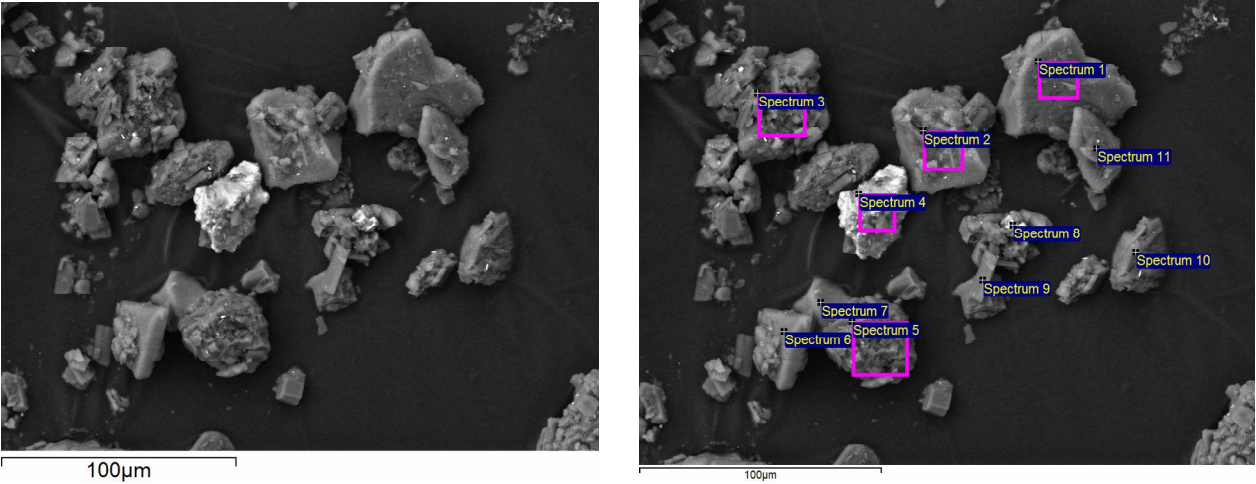
**Backscattered Electron (BSE) Micrograph of Particles at Site 6 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.9	13.6	0.2		58.1	0.9					23.1		0.1	Cl 0.1
2	11.9	16.0			66.6	3.1	2.0				0.2			S 0.1
3	11.0	16.6			65.6	3.5	3.0				0.2			
4	6.9	11.9			35.2	0.4					44.9		0.3	S 0.4
5	9.8	15.2			68.5	1.8					2.9	1.8		
6	13.9	12.7			69.7	3.1					0.4			S 0.2

**Backscattered Electron (BSE) Micrograph of Particles at Site 7 in Black Nodule Subsample of C-108 Residual Waste**



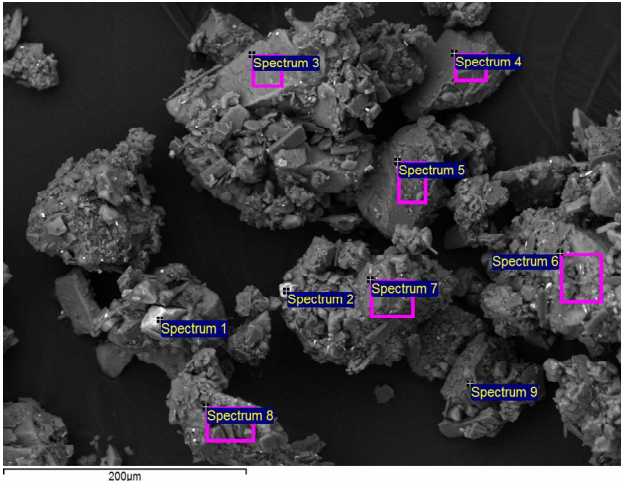
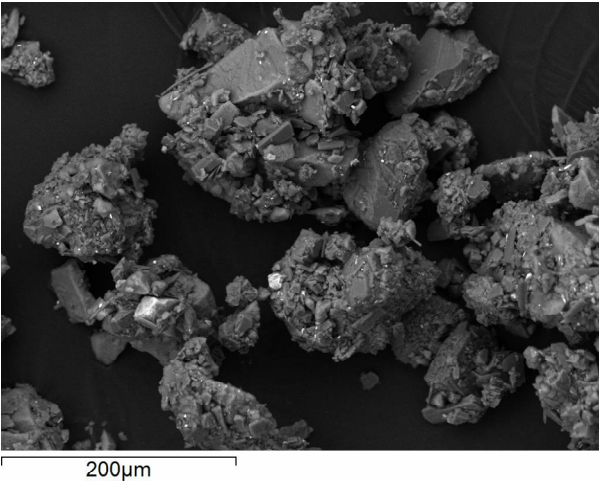
E.8

**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	2.2	31.5			48.2	12.1	6.0							
2	6.0	26.4			55.4	10.4	1.7				0.2			
3	9.7	21.0			65.7	2.5	1.0				0.1	0.1		
4	4.1	18.4	0.1		64.6	1.4					11.3		0.1	Cl 0.1
5	16.5	9.2			69.7	1.6	2.9				0.1		0.1	
6	3.0	23.1			54.1	19.7								
7	0.3	32.3			45.7	15.8	5.9							
8	2.2	15.6			58.1	1.0					23.1			
9	18.2	9.4			71.2	0.5		0.1			0.1	0.1	0.4	S 0.1
10	4.1	35.3			58.2	1.7					0.2	0.6		
11	2.3	30.7			50.8	9.5	6.5							S 0.2



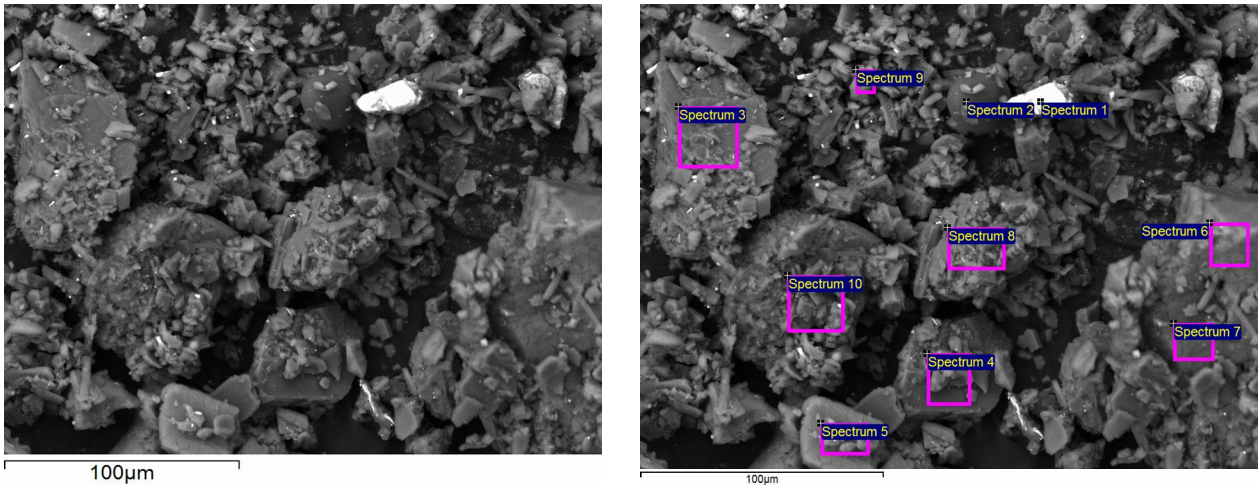
**Backscattered Electron (BSE) Micrograph of Particles at Site 8 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.6	9.4			62.6	13.1								Sr 14.2
2	5.0	10.8			39.1	2.1					42.3			Pb 0.1, Cu 0.2, K 0.3, Cl 0.2
3	10.2	16.7			67.8	5.3								
4	19.6	1.5			78.6	0.3								
5	20.6	8.6			69.7	1.1								
6	6.9	25.6	0.1		56.9	6.1	4.3	0.0			0.1			S 0.1
7	15.5	14.2		0.0	69.0	1.2					0.1			S 0.1
8	7.4	24.8			62.8	4.9					0.1			
9	8.6	21.1			67.9	2.4								

**Backscattered Electron (BSE) Micrograph of Particles at Site 9 in Black Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.3	18.6		0.4	68.0	3.0		5.4			3.2			Pb 0.2
2	2.3	14.3		0.4	64.9	1.2								Zn 2.1, Mg 5.6, K 1.0, S 2.1, Cl 6.1
3	18.9	4.8			75.8	0.5								
4	16.7	10.0			69.6	1.5	2.2							
5	3.9	30.0			51.2	8.6	6.2				0.0			
6	1.3	33.4			48.2	11.5	5.5				0.1			
7	0.6	34.8			46.0	11.6	7.1							
8	18.2	8.9			71.8	0.9					0.2	0.1		
9	7.2	25.6			55.6	6.2	5.2				0.1	0.1		
10	13.5	20.4	0.2		64.3	1.6								

## **Appendix F**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Crystal Nodule Subsample from Unleached Residual Waste from Tank C-108**



## **Appendix F**

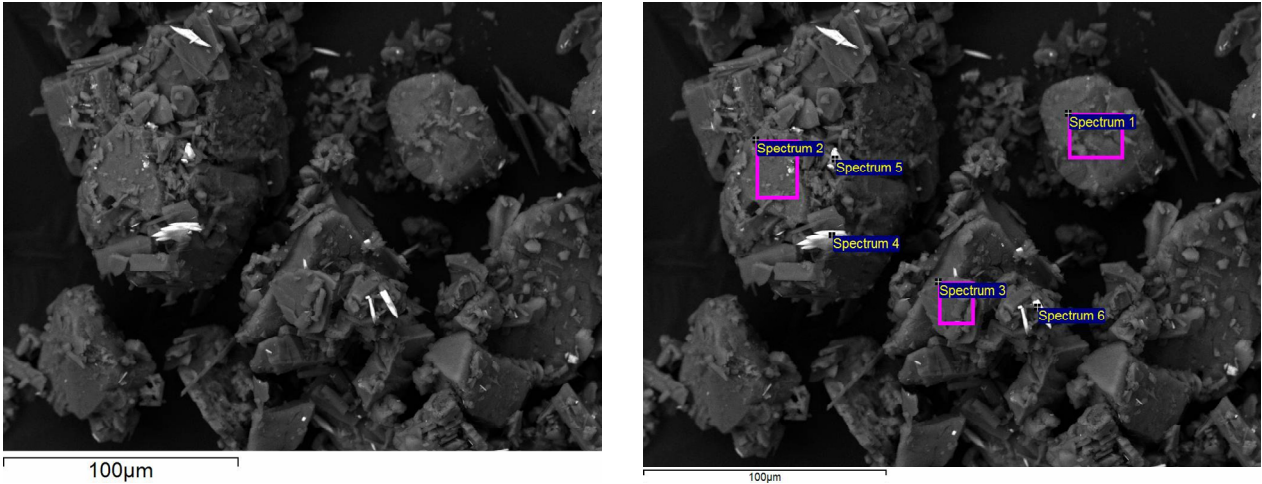
### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Crystal Nodule Subsample from Unleached Residual Waste from Tank C-108**

This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the crystal nodule subsample from tank C-108 unleached residual waste, and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the crystal nodule subsample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.

Backscattered Electron (BSE) Micrograph of Particles at Site 1 in Crystal Nodule Subsample of C-108 Residual Waste

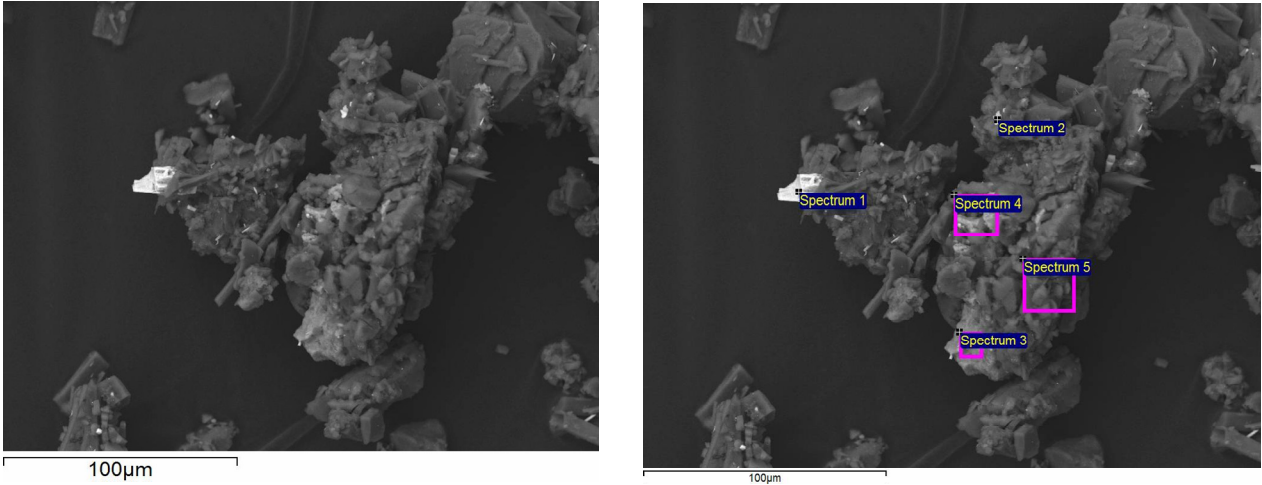


F.2

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.2	27.5			57.8	9.2	4.3							
2	22.9	6.3			70.1	0.8								
3	16.1	14.4			67.9	1.4								S 0.2
4	0.8	17.3			74.2	0.4		7.3						
5	6.9	13.8			75.3	0.3		3.7						
6	7.5	16.3			72.5	0.2		3.4						

Backscattered Electron (BSE) Micrograph of Particles at Site 2 in Crystal Nodule Subsample of C-108 Residual Waste

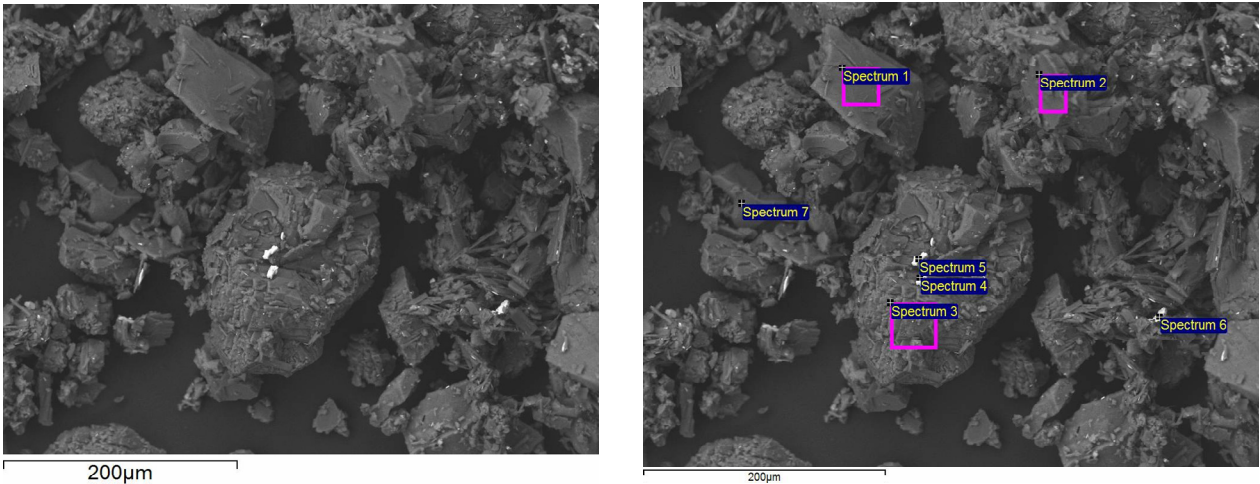


F.3

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	6.6	8.1			70.5	0.6		14.2						
2	5.1	19.3			69.0	2.2	3.0	1.4						
3	6.8	12.3			41.1	1.0					38.5	0.4		
4	3.8	21.4			51.9	3.7		0.1			18.5	0.4		Pb 0.2
5	7.2	20.8			64.2	5.0	2.8							

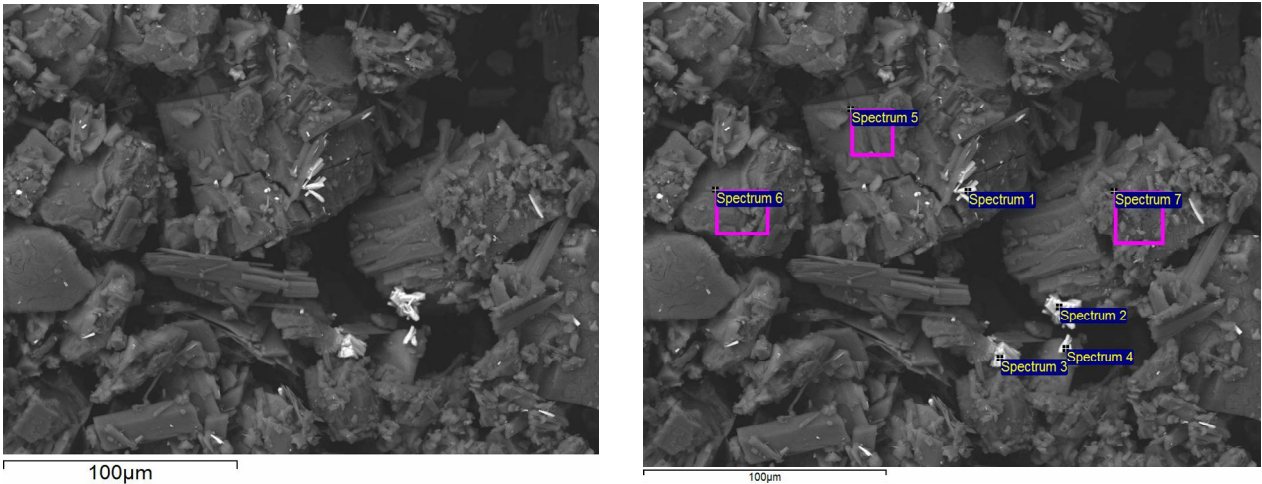
Backscattered Electron (BSE) Micrograph of Particles at Site 3 in Crystal Nodule Subsample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.3	30.8			52.8	6.5	9.6							
2	18.5	5.7			75.4	0.5								
3	22.7	3.8			72.9	0.7								
4	1.6	18.8			76.2	0.2		3.4						
5	7.7	8.9			79.1			4.3						
6	8.0	13.0		1.0	71.8	1.0		4.2				0.9		
7	10.3	9.2			74.2	6.3								

Backscattered Electron (BSE) Micrograph of Particles at Site 4 in Crystal Nodule Subsample of C-108 Residual Waste

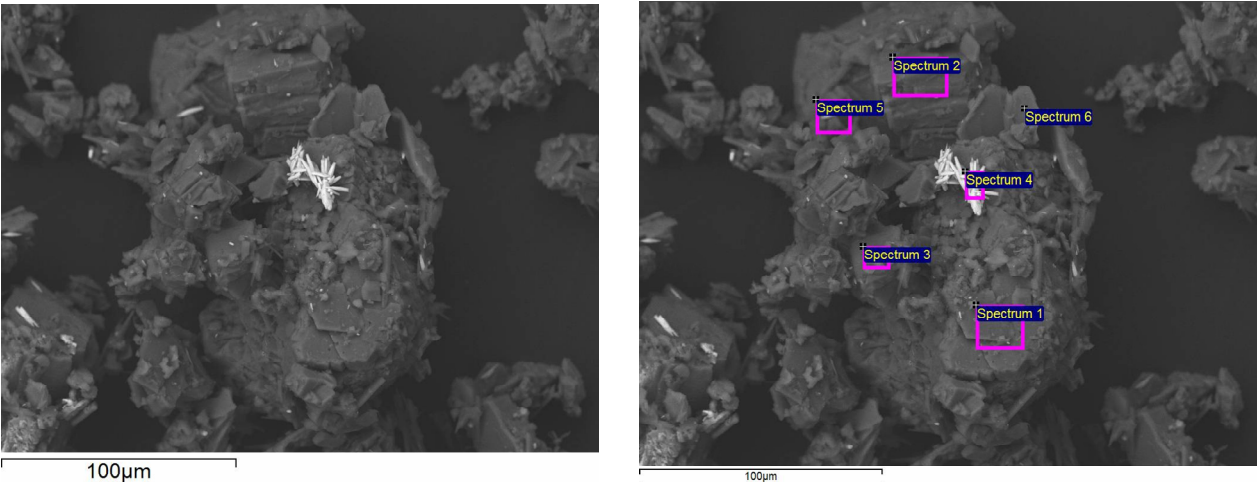


F.5

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.7	16.7			74.2	0.3		5.1						
2	5.7	11.2			72.7	1.2		9.2						
3	0.9	20.9			74.2	0.3		3.7						
4	2.6	22.8			67.2	1.1		6.4						
5	24.9	6.3			67.8	1.0								
6	20.6	5.9	0.2		72.7	0.7								
7	8.0	20.7			61.1	8.2	1.9							S 0.1

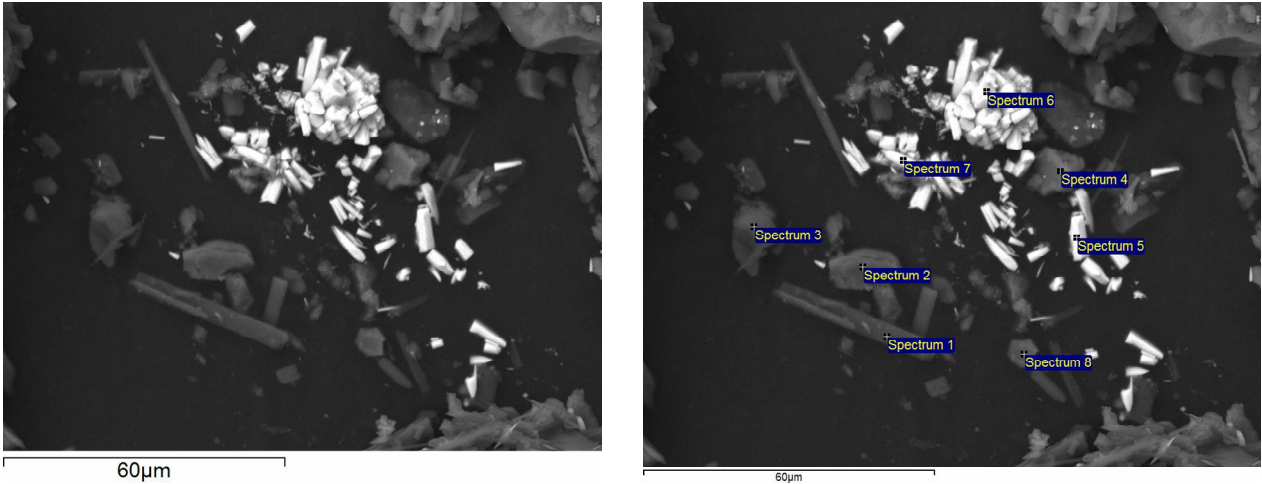
**Backscattered Electron (BSE) Micrograph of Particles at Site 5 in Crystal Nodule Subsample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	22.9	3.6			72.9	0.6								
2	21.6	2.5			75.6	0.3								
3	25.9	4.0			69.5	0.4		0.1			0.1			
4	3.0	19.7			73.6	0.3		3.5						
5	6.2	20.8			70.8	1.5		0.8						
6	29.8	3.2			66.5	0.5								

Backscattered Electron (BSE) Micrograph of Particles at Site 6 in Crystal Nodule Subsample of C-108 Residual Waste



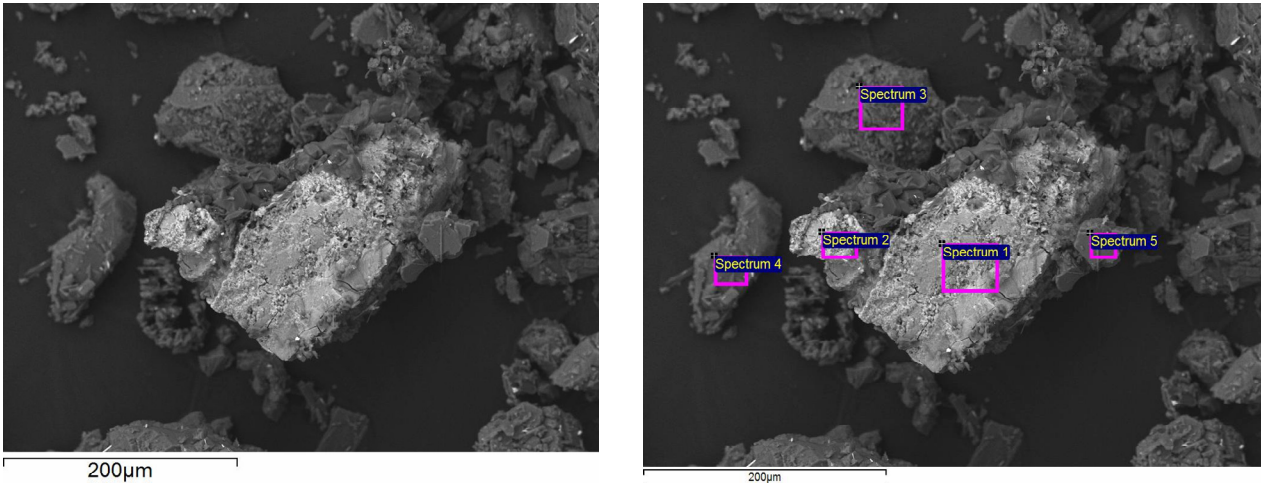
F.7

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	11.0	22.3			63.3	2.0								S 1.3
2	26.3	0.8			72.8	0.2								
3	2.1	34.6			58.6	2.2								S 2.5
4	22.7	1.4			75.6	0.3		0.1						
5	0.6	20.8			68.2	0.3		10.1						
6	0.8	17.7			76.6	0.2		4.7						
7	4.9	13.8			74.8	0.4		6.2						
8	25.3	2.1			72.1	0.6								



Backscattered Electron (BSE) Micrograph of Particles at Site 7 in Crystal Nodule Subsample of C-108 Residual Waste

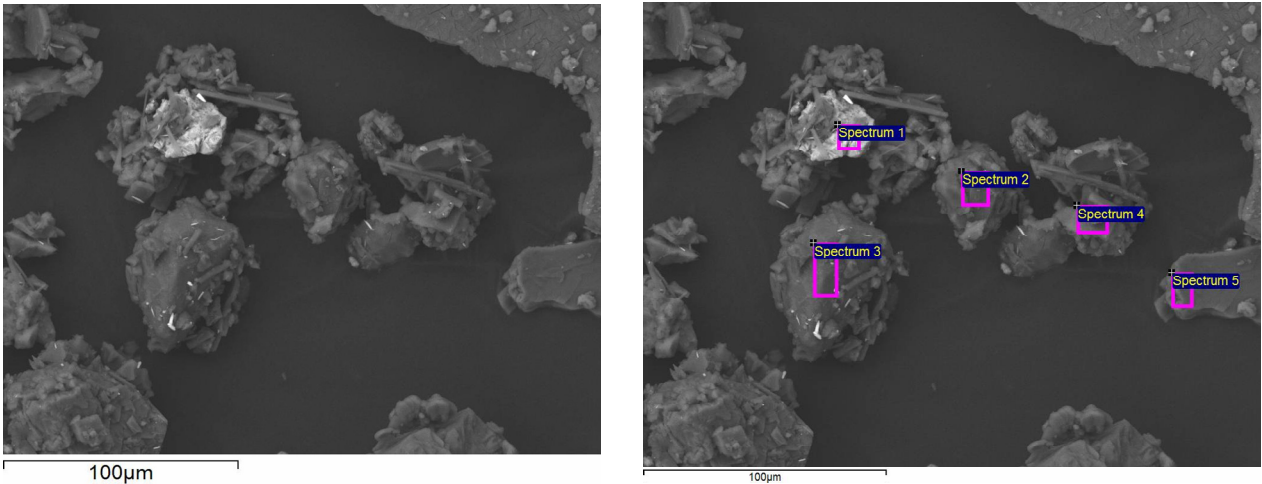


Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.9	10.4	0.3		62.8	2.8					19.2			Pb 0.6
2	3.5	13.6			58.3	3.2					20.6			Pb 0.8
3	16.9	9.4			71.9	1.7					0.1			
4	0.5	24.4			61.6	10.4	3.2							
5	25.9	8.7			64.4	0.5					0.6			



Backscattered Electron (BSE) Micrograph of Particles at Site 8 in Crystal Nodule Subsample of C-108 Residual Waste



F.9

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.8	14.7			62.2	3.0				0.1	17.3		0.1	Pb 0.7
2	19.6	8.1			71.9	0.4								
3	0.5	27.9			60.2	6.7	4.6				0.1			
4	7.9	25.5			63.6	2.8								S 0.2
5	15.6	9.9			72.8	1.7								

## **Appendix G**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Deionized Water Leached Residual Waste from Tank C-108**

## Appendix G

### Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for Deionized Water Leached Residual Waste from Tank C-108

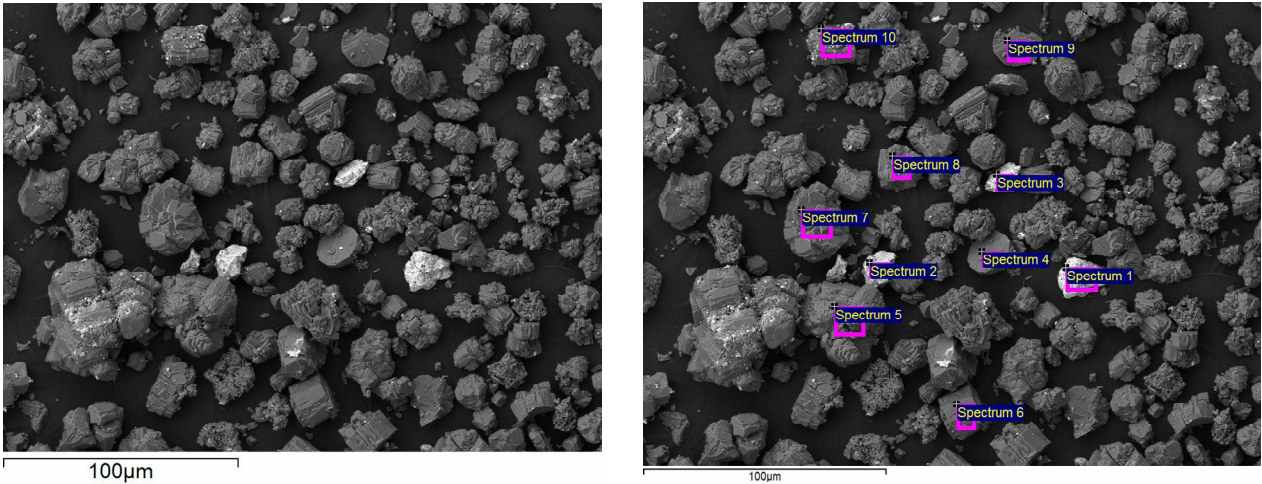
This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the sample of tank C-108 DI water leached residual waste and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the DI water leached sample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.

Compositions were not determined for particles analyzed by EDS at sites of interest 6 through 14. Iron oxide/hydroxide particles at these sites were analyzed by EDS at 30 or 35 keV (instead of 20 keV) to check for the  $K\alpha$  x-ray emission lines for Tc at 18.37 and 18.25 keV. Analyses completed at 20 keV of Fe oxide/hydroxide particles at sites 1 through 5 suggested the possible presence of Tc based on the existence of a shoulder on the high energy side of the Pb peak at ~2.3 keV. However, the EDS analyses completed at 30 and 35 keV of Fe oxide/hydroxide particles at sites of interest 6 through 14 did not detect the Tc  $K\alpha$  x-ray emission lines. Thus, presence of Tc could not be verified based on SEM/EDS analyses, but it may be present at less than the minimum detection limit of EDS.

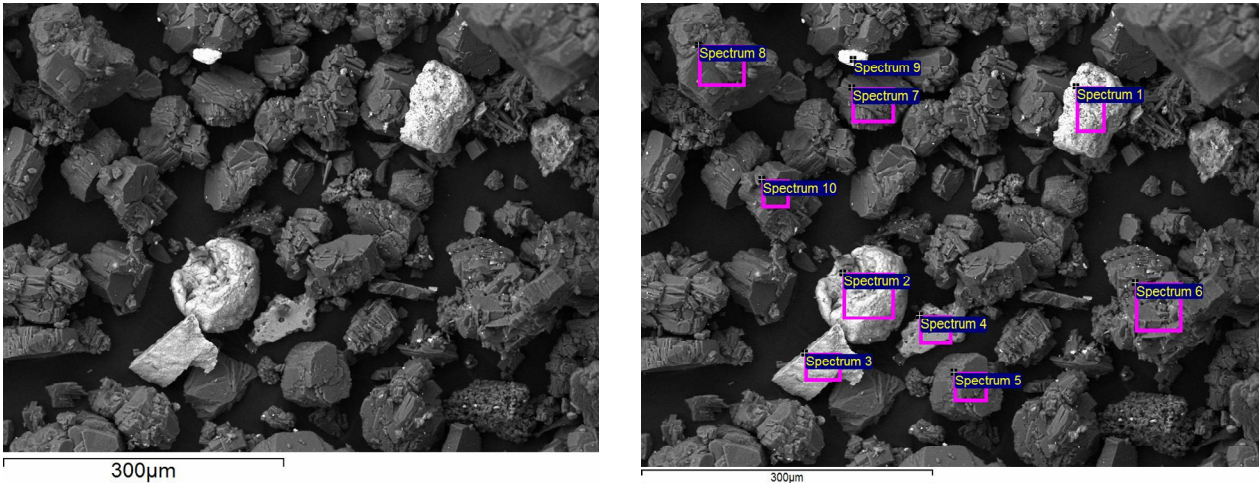
Backscattered Electron (BSE) Micrograph of Particles at Site 1 in DI Water Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	4.3	5.9	0.4	0.1	61.9	2.2					24.2		0.2	Pb 0.9
2	4.5	3.1	0.4		61.8	0.6					28.8	0.1	0.2	Pb 0.3, Zn 0.2
3	4.7	3.2	1.8		60.2	2.1					24.7			Pb 1.1, Zn 0.3, Mg 1.9
4	23.5	0.9	0.2		75.2	0.2								
5	21.9	0.3			77.5	0.2					0.2			
6	25.6	0.6			73.8									
7	20.3	0.5			78.9	0.2					0.1			
8	22.4	0.4			77.1	0.1					0.0			
9	22.8	0.5			76.7									
10	18.0	3.9	2.0		75.6	0.3		0.1			0.1		0.0	

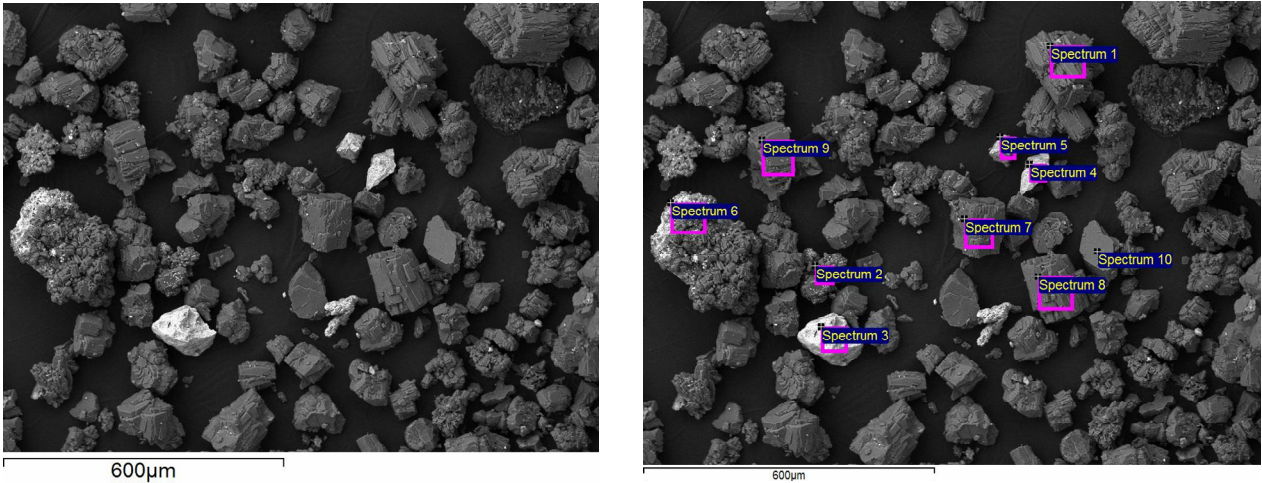
Backscattered Electron (BSE) Micrograph of Particles at Site 2 in DI Water Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	15.2				42.4						41.2			Cu 1.1
2	20.1				78.9						1.1			
3	15.5	0.9			53.1						30.2	0.3		
4	11.1	0.8			32.0	0.4					54.0			Pb 0.2, Cu 0.7, Zn 1.0
5	21.5	0.4			77.9	0.2								
6	21.5	1.4		0.1	75.9	0.4					0.2	0.5		
7	21.6	0.4		0.0	77.6	0.2						0.1		
8	20.7	0.7			78.4	0.2								
9	12.6				84.6									Cd 2.1, K 0.8
10	20.9	0.6			78.5									

Backscattered Electron (BSE) Micrograph of Particles at Site 3 in DI Water Leached Sample of C-108 Residual Waste

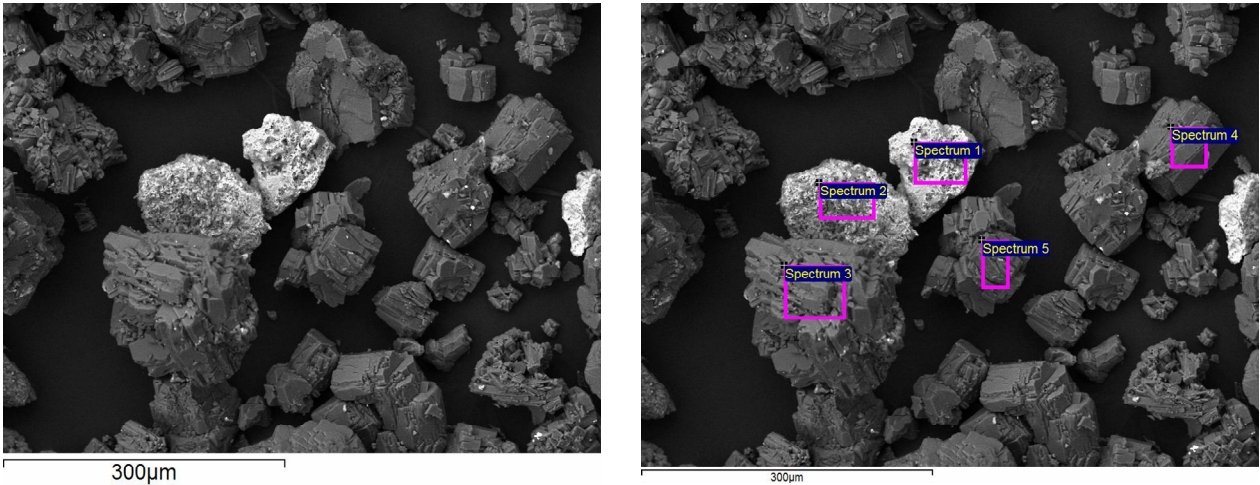


Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	19.7	0.5	0.1	0.0	79.3	0.2					0.0	0.1		
2	20.6	0.5			78.3	0.2		0.2			0.2	0.1		
3	2.5	5.2	0.4	0.1	69.6	2.3	0.3	0.1			18.5			Pb 0.8, Zn 0.2
4	3.0	2.9	0.4		66.7	0.7	0.3				25.2		0.1	Pb 0.2, Zn 0.5
5	4.1	1.9	0.2		65.4	0.2					27.9		0.2	Pb 0.1, Zn 0.2
6	16.7	5.4	2.9		73.1	0.4		0.7			0.6	0.1		
7	21.5	0.4			78.0	0.1								
8	22.5	0.5			76.8	0.2					0.1			
9	21.3	0.6			78.0	0.2					0.0			
10	24.1	0.3			75.7									



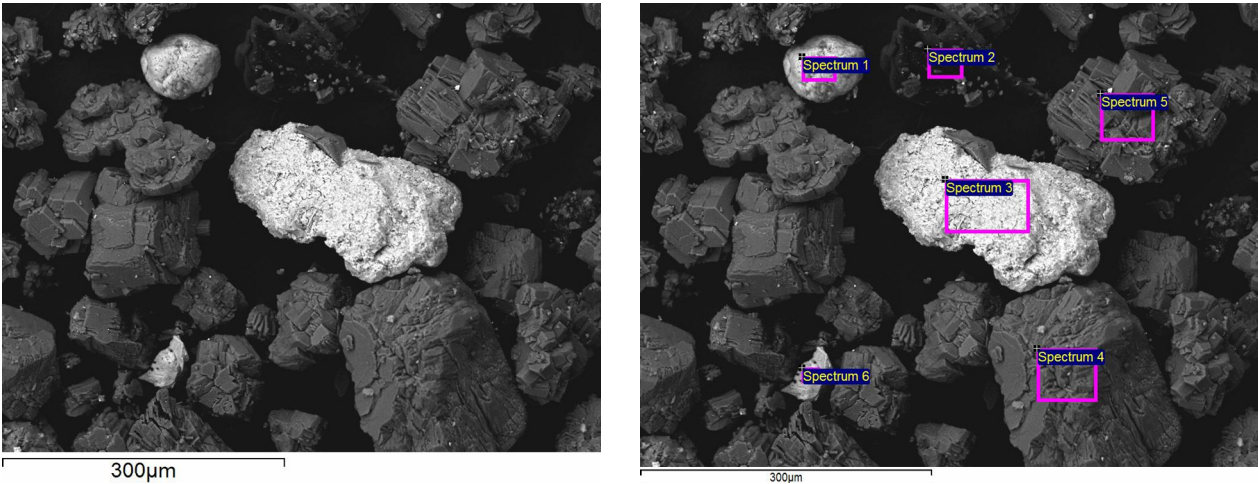
Backscattered Electron (BSE) Micrograph of Particles at Site 4 in DI Water Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.7	5.1	1.4	0.2	64.0	1.7		0.1		0.1	21.0	0.5		Pb 0.6
2	8.1	9.1	0.9	0.5	64.1	3.3		0.2	0.2		9.5	2.6		Pb 0.5
3	23.8	0.4			75.6						0.2			
4	22.9	0.5			76.3	0.2					0.1			
5	17.8	0.7			81.4						0.1			

**Backscattered Electron (BSE) Micrograph of Particles at Site 5 in DI Water Leached Sample of C-108 Residual Waste**

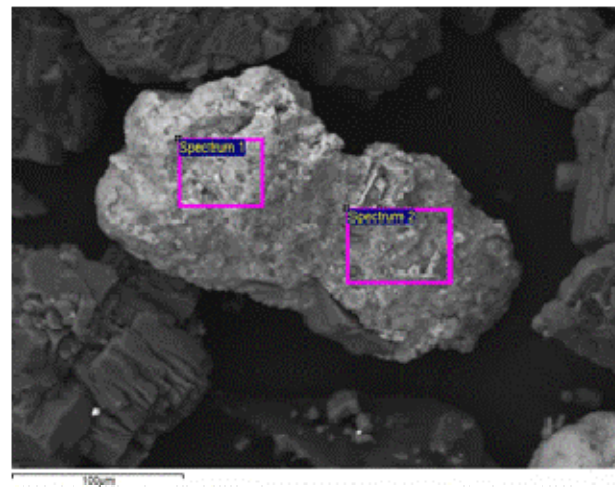
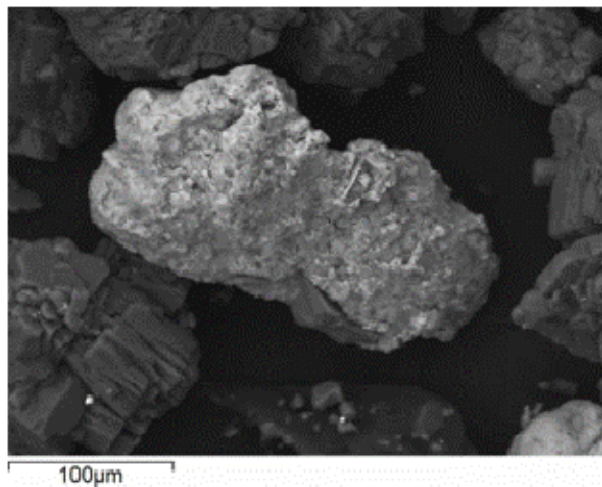


**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

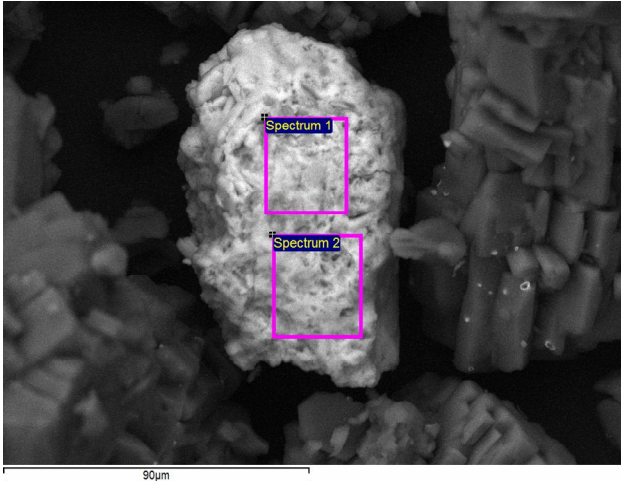
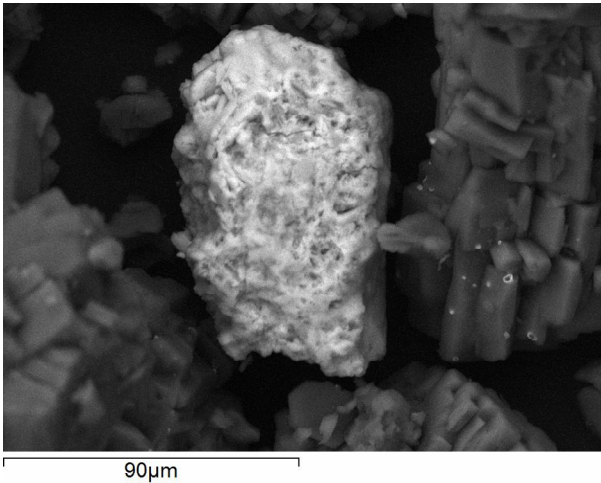
Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.1	10.6		0.5	62.9	12.3					0.1			Sr 12.4
2	16.2	1.9	5.3		72.3	1.1					3.2			
3	2.6	6.6	1.4	0.2	64.0	2.4		0.2			19.7	0.3	0.2	Pb 1.0, Ti 0.7, Mg 0.8
4	22.5	0.3			76.9	0.2					0.1			
5	20.7	0.7			78.2	0.2					0.1	0.1		
6	13.6	0.4			51.6	0.4		0.2			32.8		0.4	Pb 0.1, Cu 0.3, Zn 0.4



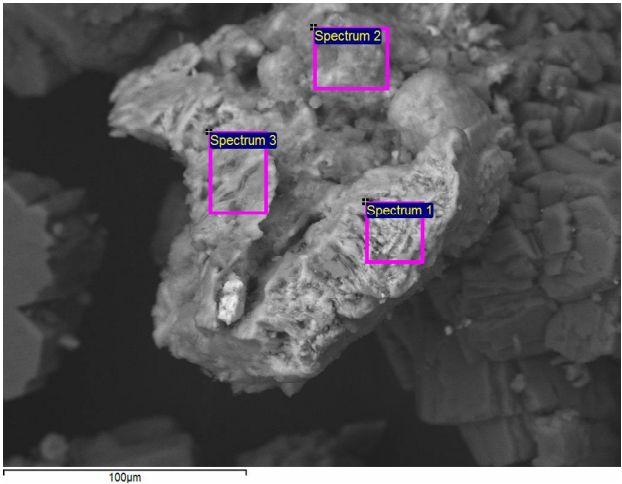
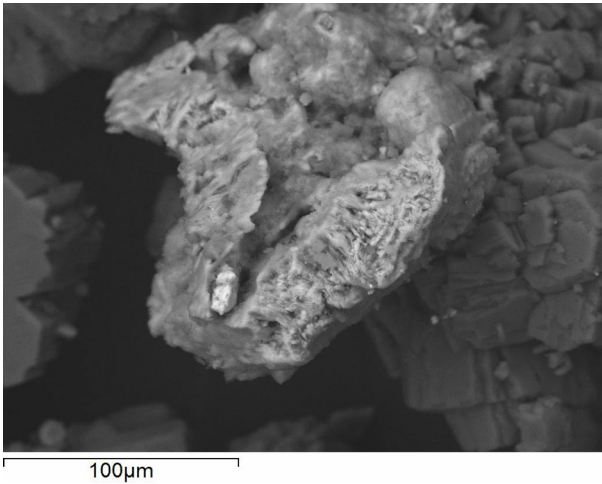
**Backscattered Electron (BSE) Micrograph of Particles at Site 6 in DI Water Leached Sample of C-108 Residual Waste**



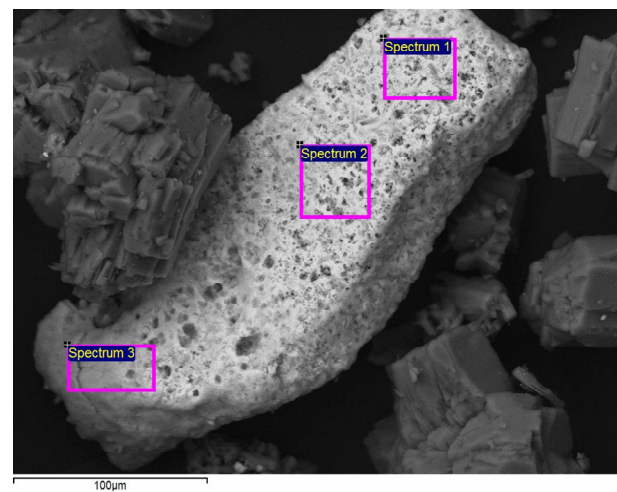
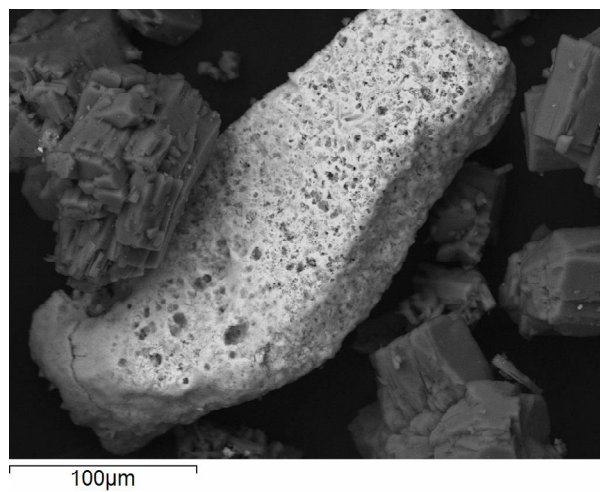
**Backscattered Electron (BSE) Micrograph of Particles at Site 7 in DI Water Leached Sample of C-108 Residual Waste**



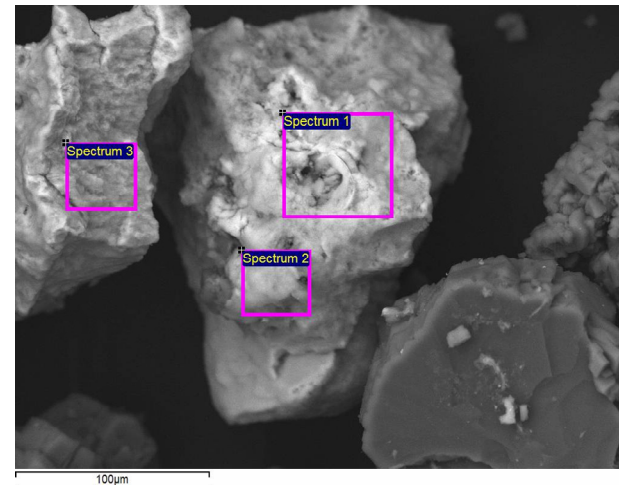
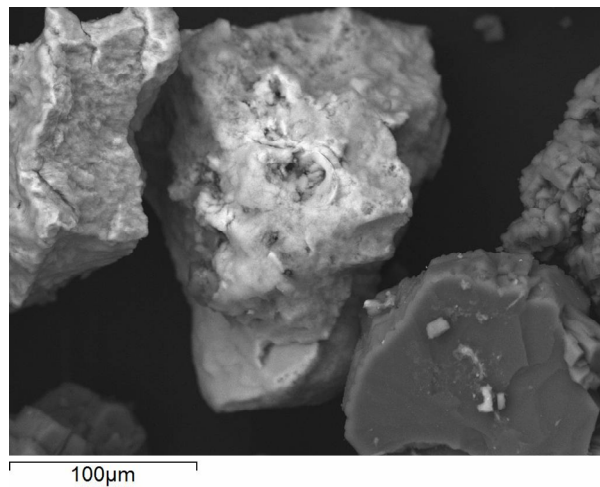
**Backscattered Electron (BSE) Micrograph of Particles at Site 8 in DI Water Leached Sample of C-108 Residual Waste**



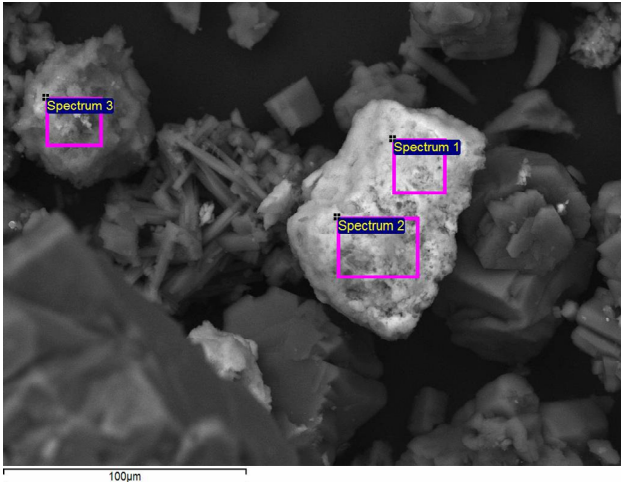
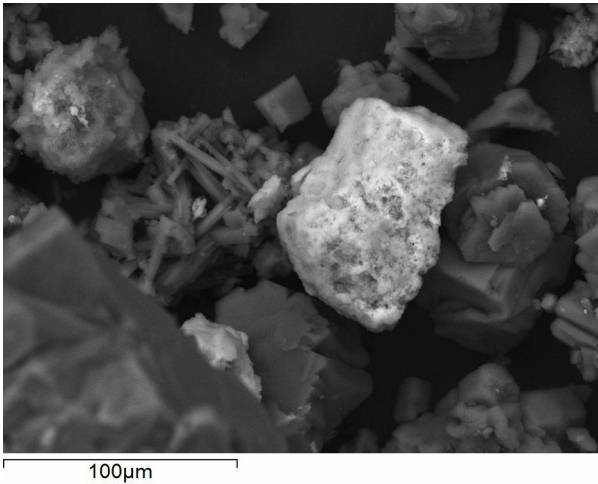
**Backscattered Electron (BSE) Micrograph of Particles at Site 9 in DI Water Leached Sample of C-108 Residual Waste**



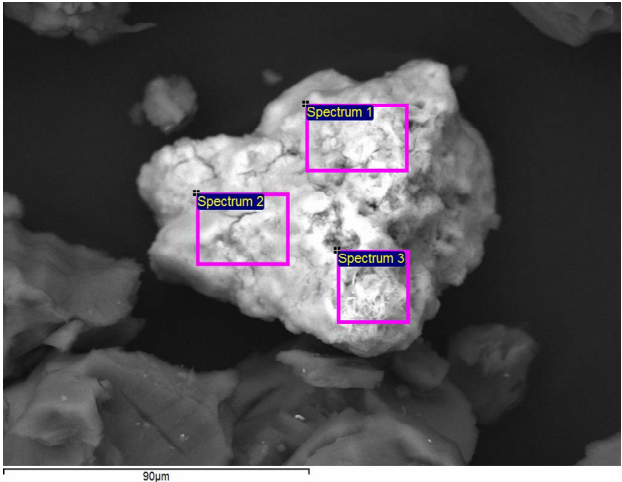
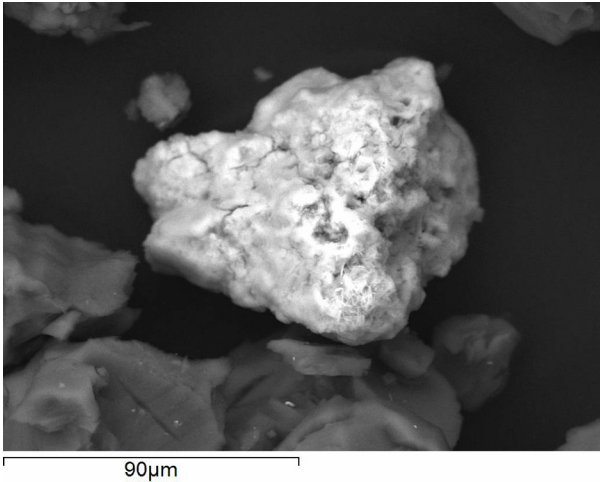
**Backscattered Electron (BSE) Micrograph of Particles at Site 10 in DI Water Leached Sample of C-108 Residual Waste**



**Backscattered Electron (BSE) Micrograph of Particles at Site 11 in DI Water Leached Sample of C-108 Residual Waste**

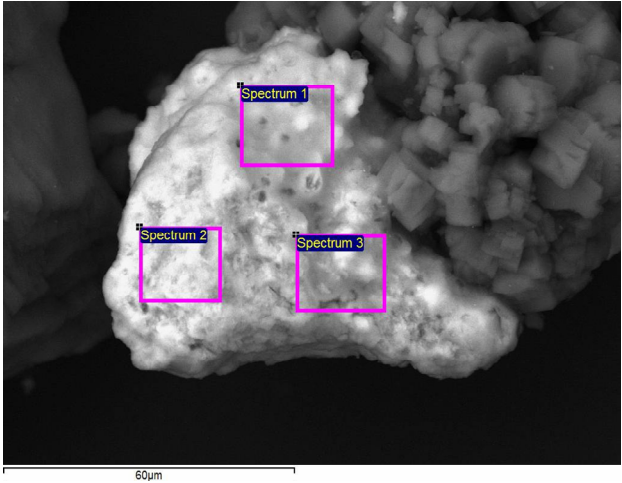
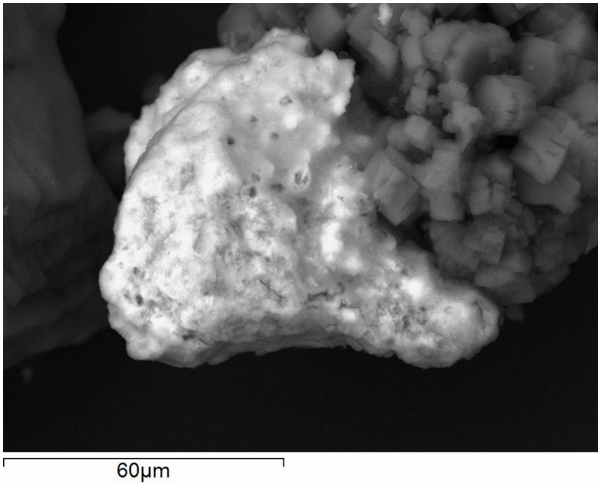


**Backscattered Electron (BSE) Micrograph of Particles at Site 12 in DI Water Leached Sample of C-108 Residual Waste**



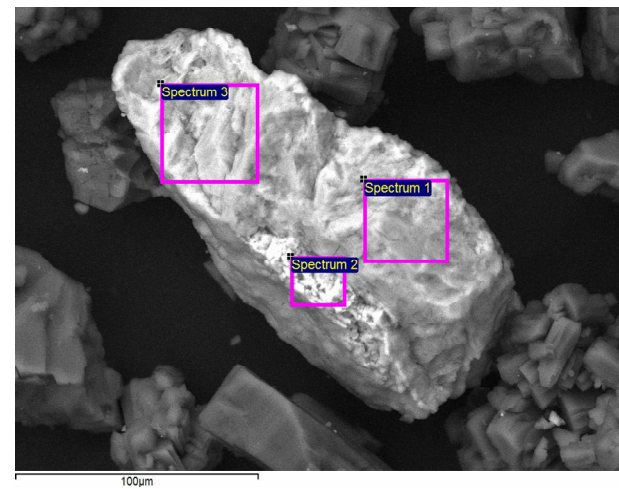
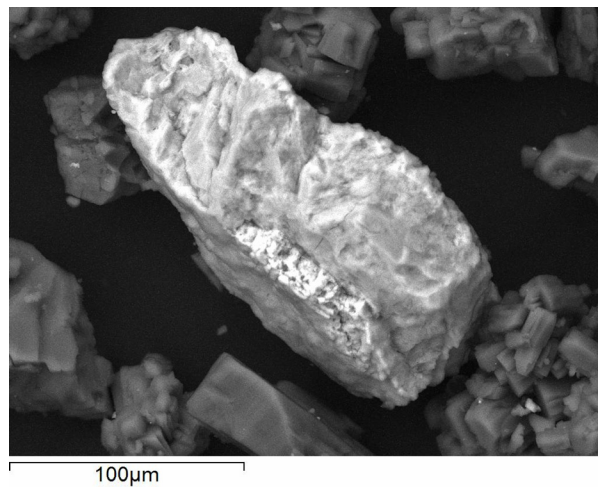


**Backscattered Electron (BSE) Micrograph of Particles at Site 13 in DI Water Leached Sample of C-108 Residual Waste**





**Backscattered Electron (BSE) Micrograph of Particles at Site 14 in DI Water Leached Sample of C-108 Residual Waste**



## **Appendix H**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for $\text{Ca}(\text{OH})_2$ Leached Residual Waste from Tank C-108**

## Appendix H

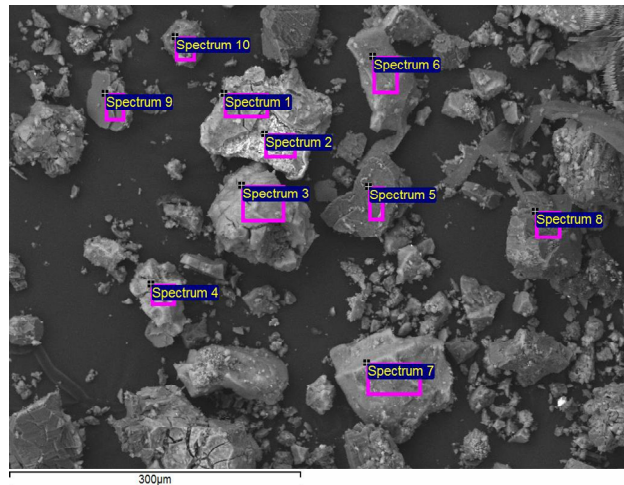
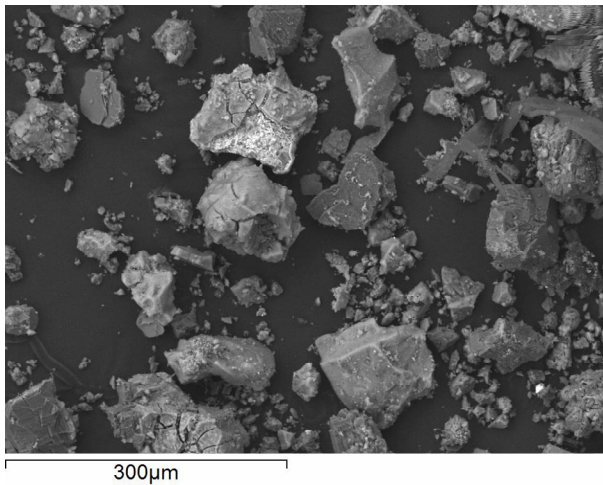
### Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for $\text{Ca}(\text{OH})_2$ Leached Residual Waste from Tank C-108

This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the sample of tank C-108  $\text{Ca}(\text{OH})_2$  leached residual waste, and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the  $\text{Ca}(\text{OH})_2$  leached sample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.

**Backscattered Electron (BSE) Micrograph of Particles at Site 1 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste**

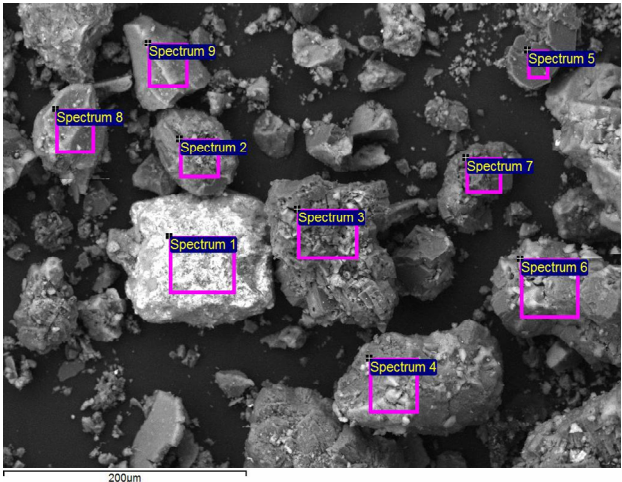
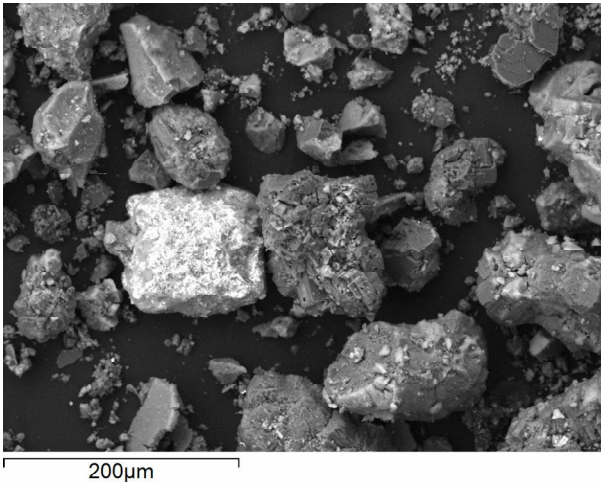


H.2

**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.3	3.4		14.4	67.2	10.1	3.5	0.1			1.0			Pb 0.1
2	1.0	4.0	1.3	4.4	69.2	4.0		0.3			14.2	0.2	0.1	Pb 1.0, Mg 0.3
3	0.4	0.4	0.2	13.9	70.9	6.9	7.3							S 0.1
4	1.7			27.0	67.0	4.3								
5	16.5	1.0	0.4	10.8	60.7	7.9	2.8							
6	0.2	0.4	0.2	13.6	72.0	6.8	6.8							
7	1.2	0.6	0.2	22.6	63.9	7.8	3.7							
8	22.3	0.7		0.9	75.6	0.6								
9	19.7	0.5	0.3	4.5	69.8	2.7	2.6							
10	16.8	0.5	0.3	6.2	70.1	3.4	2.7							

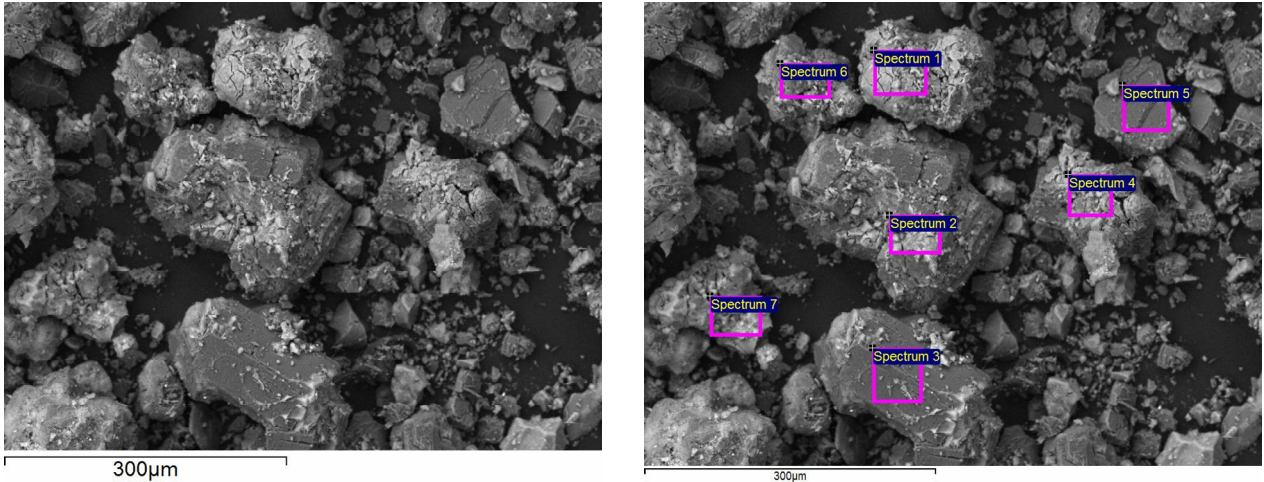
**Backscattered Electron (BSE) Micrograph of Particles at Site 2 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.5	1.1	0.9	10.0	63.3	4.6		0.7			17.5			Pb 0.5
2	15.8	0.4		8.7	66.9	4.8	3.3							
3	15.8	0.8		4.8	75.8	2.8	0.0							Cd 0.1
4	12.1	0.5		7.1	72.2	4.0	4.1							
5	23.8			0.3	75.9									
6	14.5			10.5	72.6	2.5								
7	22.2	0.8		2.4	73.4	1.3								
8	0.2	0.8	0.2	13.6	71.9	6.6	6.8							
9	0.4	0.6	0.1	13.2	72.6	6.4	6.7							

Backscattered Electron (BSE) Micrograph of Particles at Site 3 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste



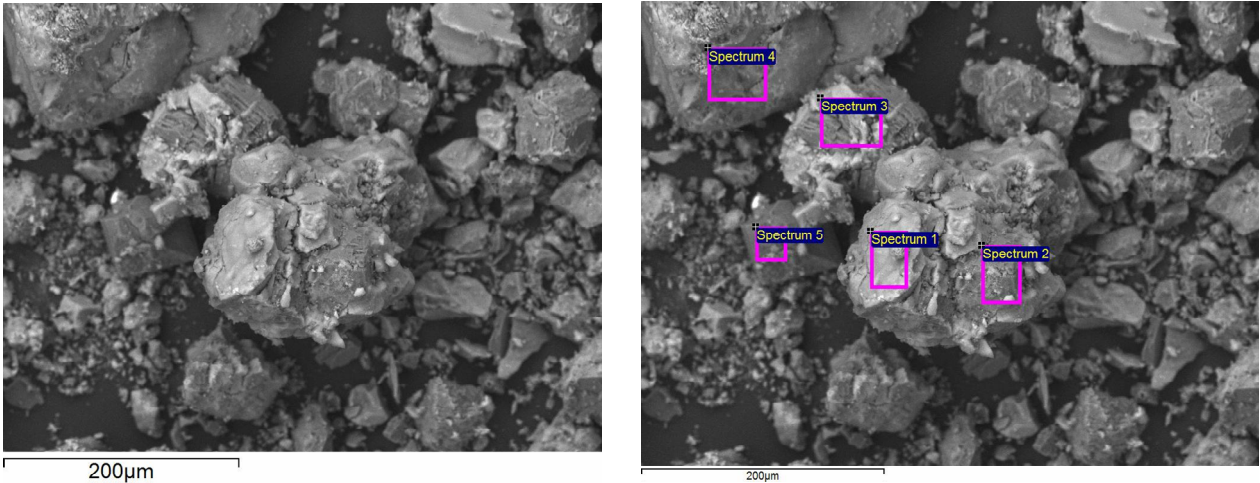
H.4

Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	9.5	2.1		8.7	71.8	5.7	2.3							
2	7.9	1.4		10.5	71.4	5.6	3.3				0.1			
3	19.6	0.3		1.1	78.1	0.8								
4	7.7	1.1		11.6	69.6	6.7	3.5							
5	20.4	0.6		0.6	77.9	0.5								
6	11.3	1.5		7.5	74.6	5.0	0.0				0.2			
7	5.3	0.5		11.8	72.5	5.5	4.4							



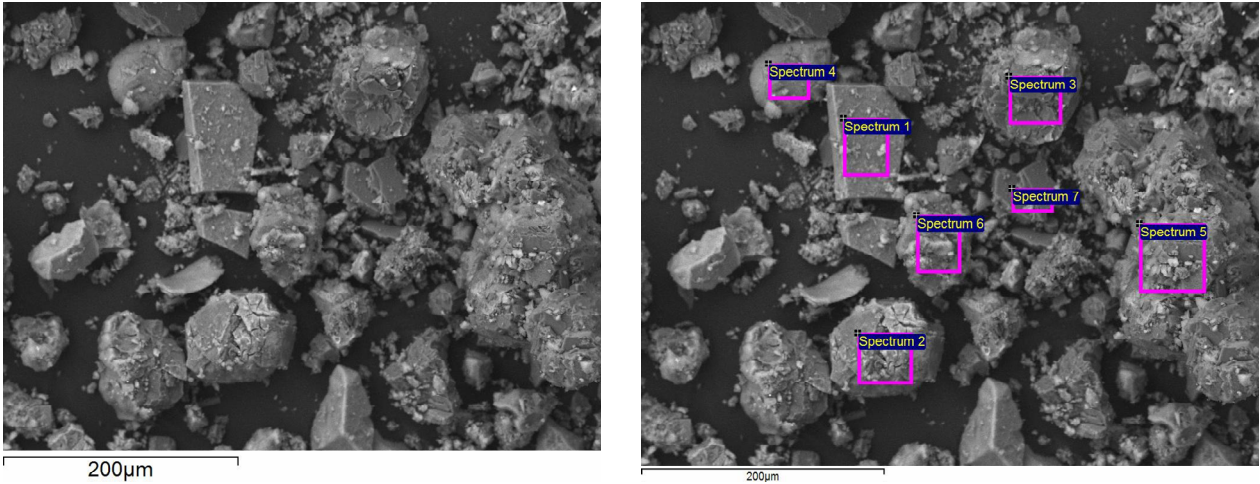
Backscattered Electron (BSE) Micrograph of Particles at Site 4 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.2	0.4		25.5	64.9	5.7	2.3							
2	15.9	0.3		2.9	79.1	1.8								
3	5.7	0.3		13.1	72.8	5.3	2.8							
4	0.6	0.6	0.2	19.9	68.8	6.2	3.7							
5	10.8		0.4	30.4	54.5	4.0								

Backscattered Electron (BSE) Micrograph of Particles at Site 5 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste

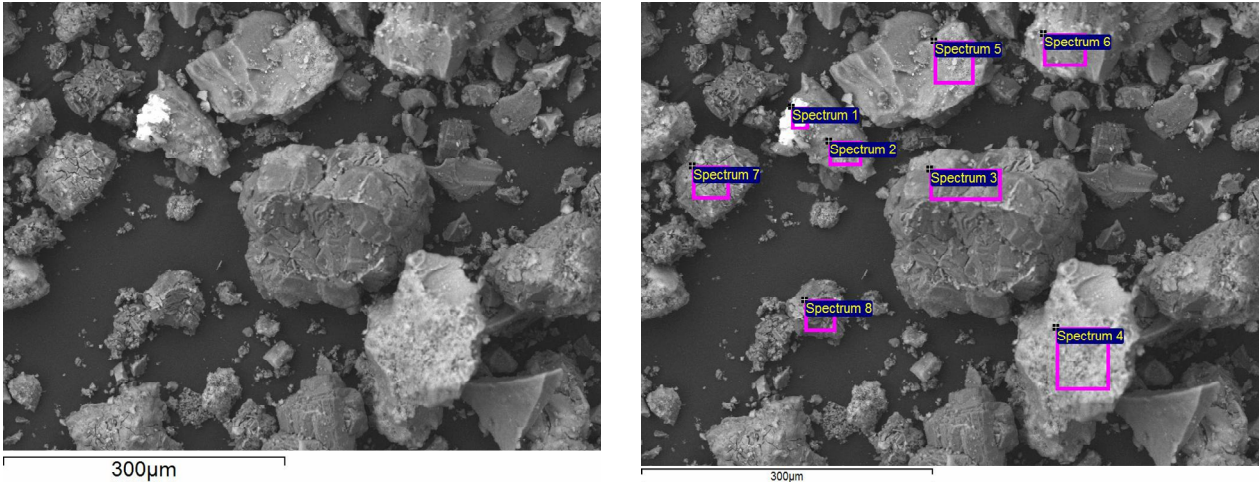


Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.7	7.2		13.3	65.5	9.7	3.7							
2	10.6	1.5		8.4	71.6	5.9	2.0							
3	21.3	0.4		3.0	73.6	1.6								
4	13.9	0.7		13.0	62.1	6.1	3.8				0.3			
5	16.9	0.5		5.5	74.8	2.4								
6	10.8	0.6		7.3	73.8	3.4	4.2							
7	23.7			4.2	70.9	1.2								



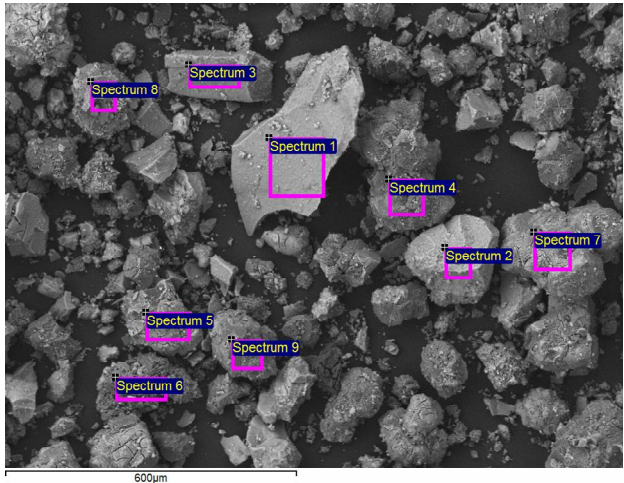
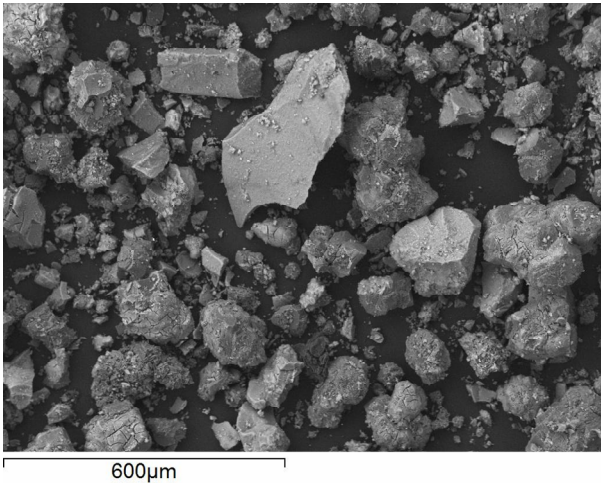
Backscattered Electron (BSE) Micrograph of Particles at Site 6 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.7	5.6	2.8	5.0	74.7	2.8							3.1	Sr 4.1, Pb 0.2
2	0.3	0.7	0.2	16.8	69.6	6.2	6.1							Sr 0.1
3	15.5	1.4		3.9	74.4	3.2	1.5							
4	2.2		0.5	30.5	60.6	6.3								
5	3.8			19.1	73.7	3.4								
6	0.7	0.4	0.2	33.8	54.3	7.7	2.8							
7	7.2	2.6		14.2	63.6	9.5	2.9							
8	19.1	0.8		1.1	78.2	0.9								

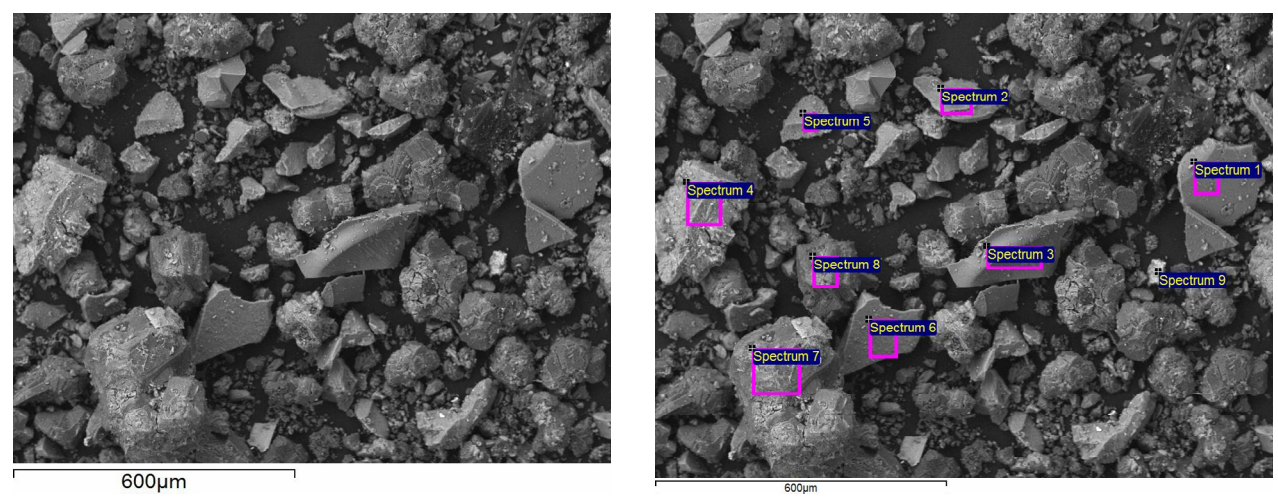
Backscattered Electron (BSE) Micrograph of Particles at Site 7 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	2.4	0.5		24.6	68.9	3.5								
2	0.9	0.7		21.4	64.0	8.2	4.6	0.1			0.1			
3	1.7	0.4	0.2	28.1	60.1	7.6	1.8							
4	16.2	1.1		5.2	74.2	3.4								
5	5.8	2.1		11.6	70.0	7.5	3.1							
6	18.6	0.3		2.8	77.0	1.2								
7	12.0	1.5		9.3	69.9	5.1	2.3							
8	2.3	2.7		13.7	68.8	9.3	3.4							
9	14.1	0.9		2.8	80.3	1.9								

**Backscattered Electron (BSE) Micrograph of Particles at Site 8 in Ca(OH)<sub>2</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	1.3	1.1	0.5	18.6	65.3	8.0	5.2							
2	0.3	0.9	0.3	12.4	72.1	6.2	7.7							
3	0.9	2.4		23.2	60.2	10.3	3.0							
4	1.3	0.6	0.2	21.1	66.5	7.2	2.9				0.3			
5	0.8	0.4	0.3	19.8	67.7	7.1	3.9							
6	0.9	0.4		18.5	66.9	8.3	5.0							
7	14.1	0.9		5.1	76.4	3.4								
8	15.3	1.0		6.6	72.6	3.9	0.0				0.6			
9	3.8	0.8	4.8	4.5	67.8	2.2		0.1			10.8	0.7		Pb 0.3, Mg 4.3

## **Appendix I**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for CaCO<sub>3</sub> Leached Residual Waste from Tank C-108**

## **Appendix I**

### **Scanning Electron Microscopy Micrographs and Energy Dispersive Spectrometry Results for CaCO<sub>3</sub> Leached Residual Waste from Tank C-108**

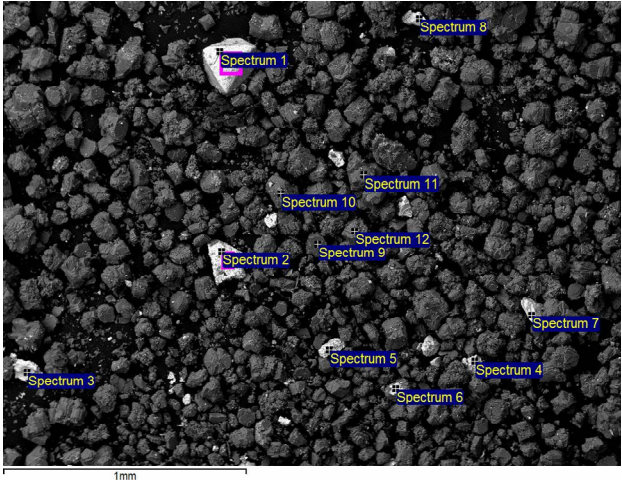
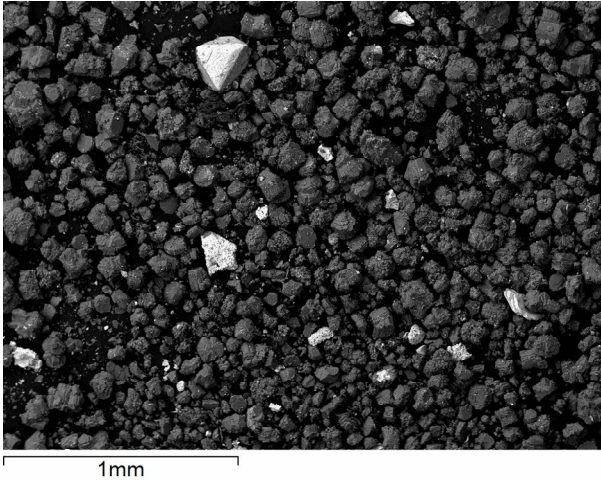
This appendix includes scanning electron microscope (SEM) micrographs for areas of interest in the sample of tank C-108 CaCO<sub>3</sub> leached residual waste, and element compositions derived from energy dispersive spectroscopy (EDS) analyses of particles in each of those areas of interest. The operating conditions for the SEM and procedures used for mounting the SEM samples are described in Section 2.4 of the main report. All SEM micrographs were collected by backscattered electron (BSE) emission.

Each of the following pages in this appendix contains 1) two electron micrographs (top of each page) of particles in the same area of interest in the CaCO<sub>3</sub> leached sample, and 2) the tabulated element compositions (in at.%) (bottom of each page) derived from EDS analyses of particles identified in the top-right electron micrograph. The electron micrograph at the top left of each page shows the same area of interest, but unobstructed by the identification numbers and locations of the EDS analyses.

In the composition tables that follow, concentrations are listed for elements expected to be common and/or important for the different samples analyzed by X-ray diffraction and SEM/EDS, contaminants of potential concern, and elements typically present as primary or trace constituents in Fe oxide/hydroxide phases. Cells in a table that contain no values indicate that those elements were not detected by EDS. Occasionally, elements other than those listed in the column headers were detected often at concentrations less than 1 at.%. In these cases, these elements and their concentrations in at.% are listed in the last column.



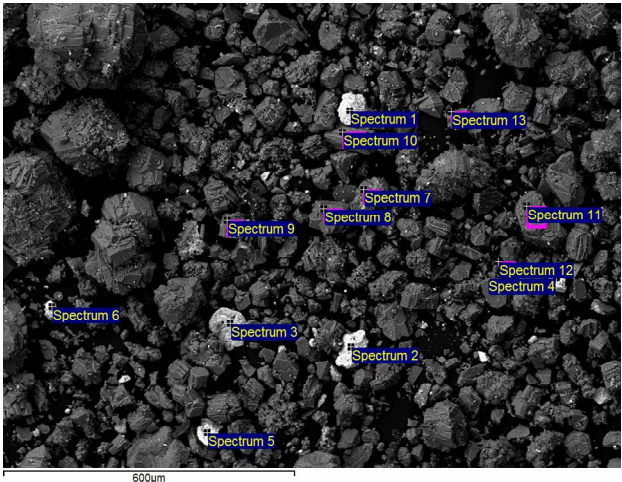
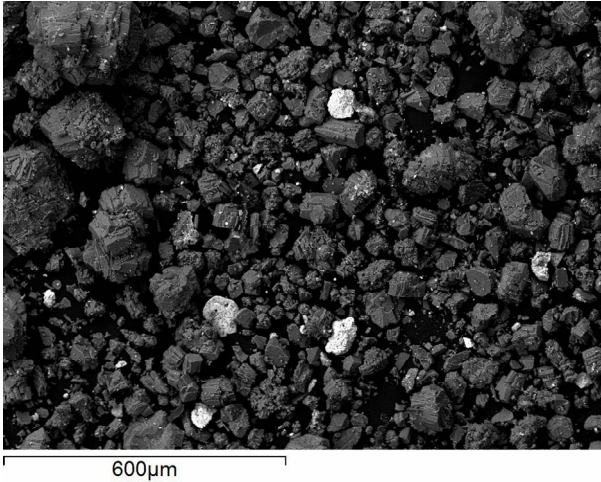
**Backscattered Electron (BSE) Micrograph of Particles at Site 1 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.9	10.9		0.2	65.1	11.5								Sr 11.5
2	4.0	3.3	1.9	0.9	64.0	2.4					20.3		0.2	Pb 1.4, Zn 0.7, Mg 1.0
3	14.0				49.4						36.6			
4	9.8				30.2						57.5	2.6		
5	1.6	7.8	1.0	0.2	60.2	3.8		0.1			24.0			Pb 1.2, Zn 0.3
6	19.4	3.0	1.2		74.9	0.4		1.1			0.2			
7	0.7	0.8			66.6	0.3					30.9		0.5	S 0.2
8	5.0	2.0	0.5	0.9	33.9	1.6					54.4			Pb 1.1, Zn 0.6
9	38.7				61.3									
10	41.0	0.4			58.7									
11	20.8	0.3			78.6	0.2					0.0			
12	24.6	0.3			74.9	0.2					0.1			

**Backscattered Electron (BSE) Micrograph of Particles at Site 2 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	3.0	2.4	0.7	0.4	70.0	1.7					19.8	0.3		Pb 1.6, Zn 0.2
2	2.4	3.4	1.2	0.4	62.8	2.4					24.5			Pb 2.4, Zn 0.5
3	1.2	9.2		1.1	63.0	12.9								Sr 12.7
4	1.4	2.0	6.3	3.2	74.3	3.5		0.1			2.6			Pb 0.2, Mg 6.4
5	6.8	8.2	3.4	0.3	24.9	5.0		0.3			45.5		0.3	Pb 3.7, Zn 0.6, Mg 1.0
6	11.4	0.9			35.9						50.8			Zn 1.0
7	17.8	2.6	0.7	0.8	73.8	1.3					2.6	0.3		Pb 0.1
8	22.0	0.7			77.2	0.2								
9	21.8	0.6		0.1	76.8	0.3					0.4	0.1		
10	23.3	0.4		0.1	75.8	0.2					0.1	0.0		
11	21.5	0.7			77.4	0.3					0.1			
12	22.2	0.6			77.0	0.2								
13	20.0	0.8	0.2		78.6	0.2					0.1	0.1		

## I.4

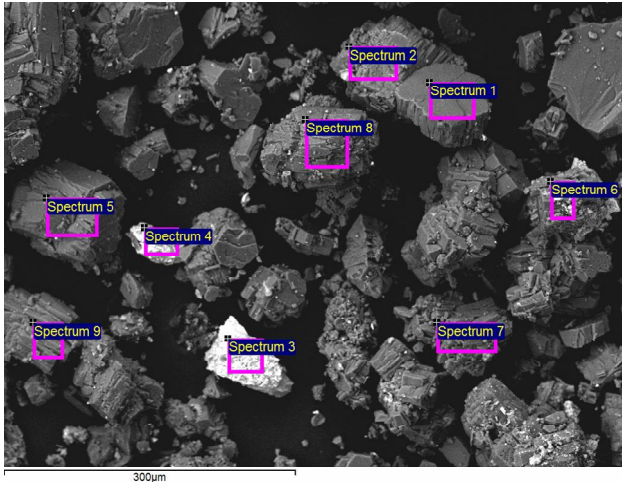
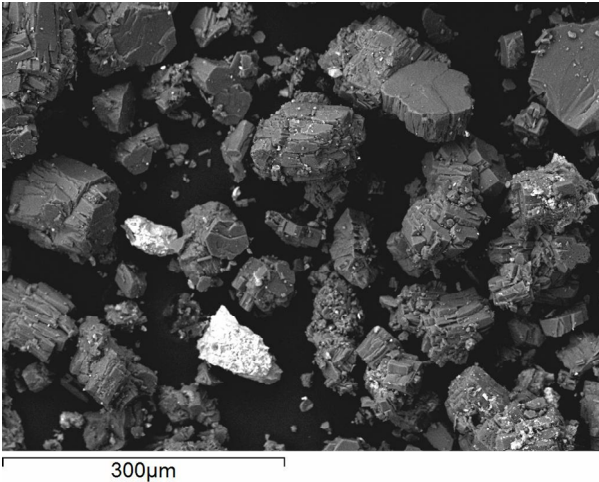


## I.4

## I.4



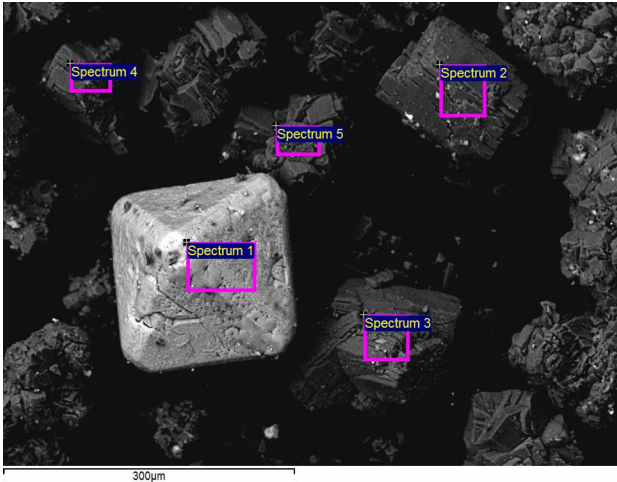
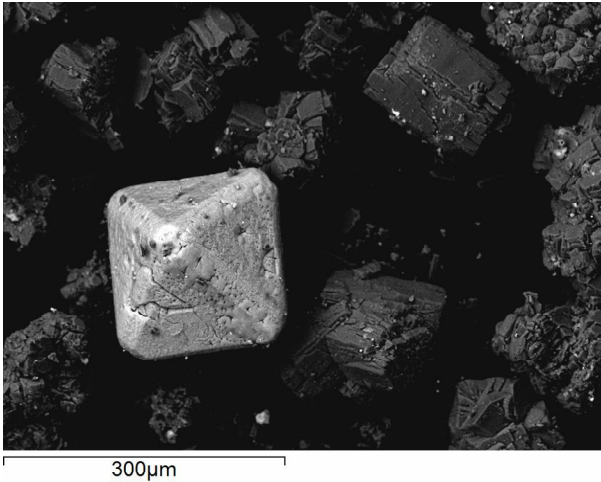
**Backscattered Electron (BSE) Micrograph of Particles at Site 4 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	18.0	0.8			81.0	0.2								
2	20.6	0.7		0.3	77.8	0.5					0.1	0.1		
3	2.6	1.9	1.3	1.6	67.5	1.8					21.2	0.4	0.2	Pb 0.4, Mg 1.2
4	8.6	1.4			29.6	0.4					59.9			
5	22.6	0.6			76.6	0.2								
6	16.8	3.5		1.4	75.6	2.1					0.3	0.4		
7	21.4	0.5		0.1	77.8	0.2					0.1			
8	22.2	0.8		0.1	76.3	0.4					0.1	0.1		
9	21.1	0.4		0.1	78.1	0.2					0.1	0.1		

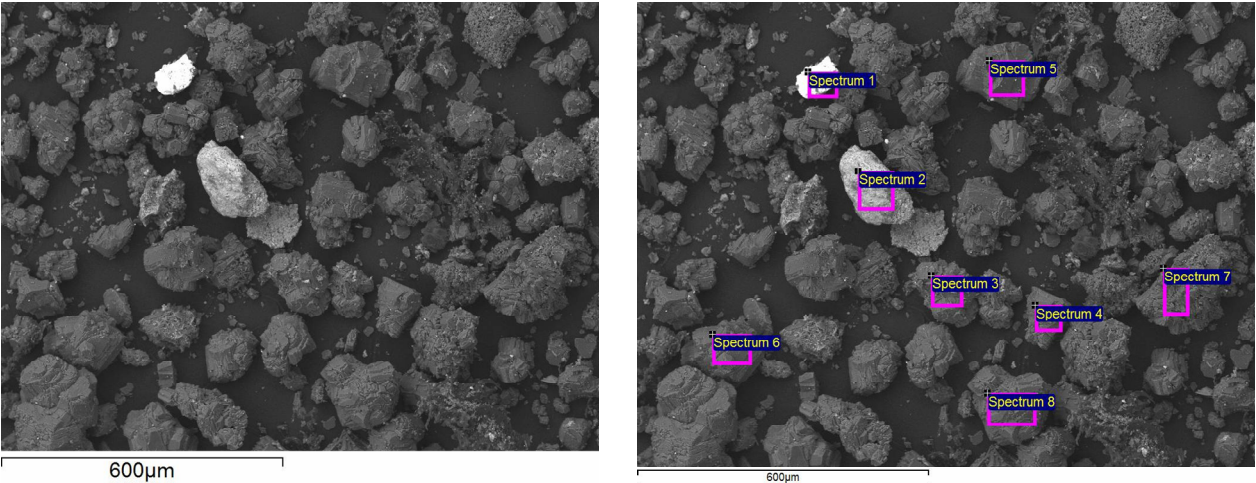
Backscattered Electron (BSE) Micrograph of Particles at Site 5 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste



Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	0.6	11.2		0.2	69.2	9.8								Sr 9.2
2	20.8	0.8		0.1	77.8	0.3						0.1		
3	20.9	0.9		0.2	77.4	0.5						0.1		
4	19.4	0.7		0.0	79.7	0.2								
5	22.3	0.9		0.1	76.1	0.4					0.1	0.1		

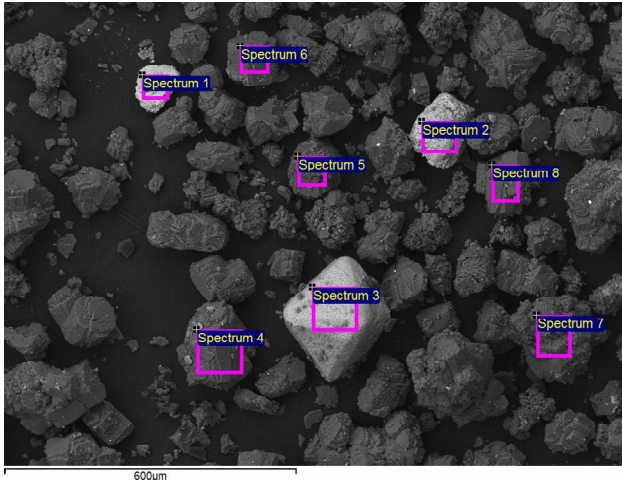
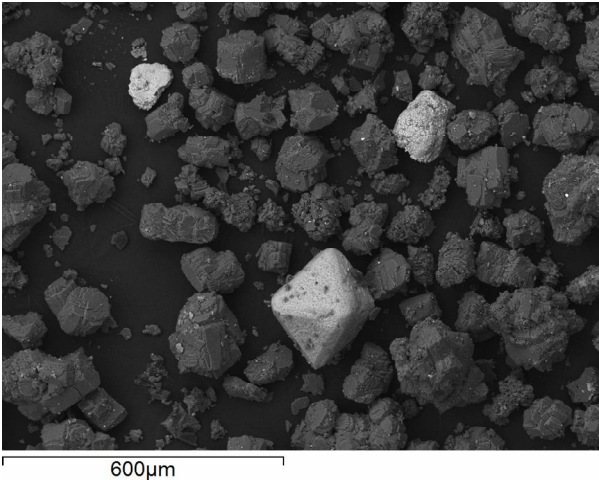
**Backscattered Electron (BSE) Micrograph of Particles at Site 6 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	10.1	5.2	1.4	0.7	75.9	0.9		5.3			0.7			
2	6.8	2.5	1.4	0.5	63.5	1.6					22.0			Pb 0.7, Mg 1.0
3	20.9	1.2		0.2	76.6	0.4					0.6	0.2		
4	21.0	0.7		0.1	77.9	0.2					0.2			
5	19.6	0.7		0.0	79.5	0.2								
6	21.0	0.6		0.1	78.0	0.2						0.1		
7	20.5	0.8		0.1	78.2	0.2						0.2		
8	21.8	0.6		0.1	76.9	0.2					0.1	0.2		

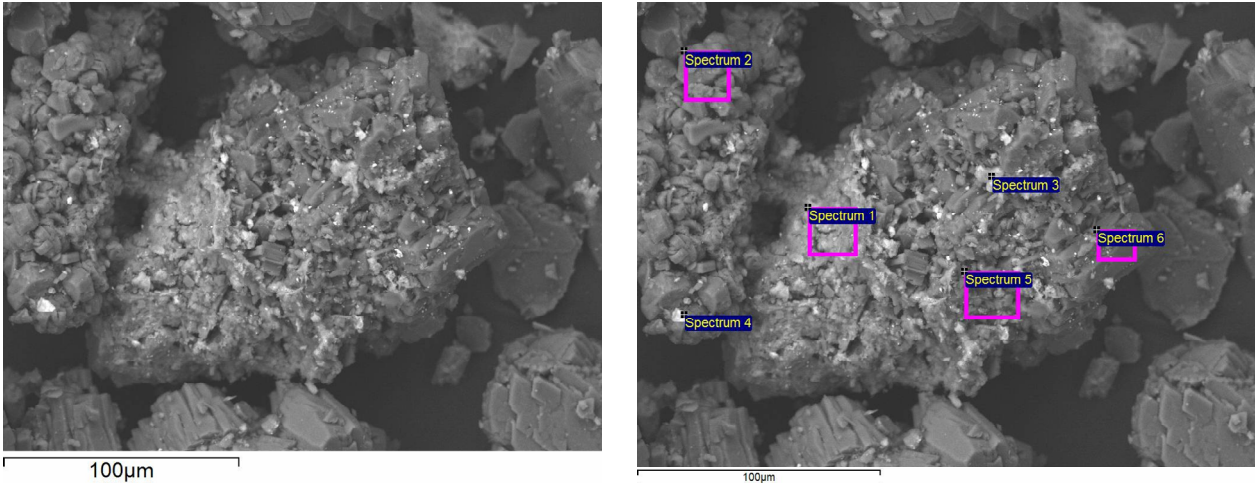
**Backscattered Electron (BSE) Micrograph of Particles at Site 7 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others	
	at.% normalized to 100%														
1	2.3	3.5	0.6	0.3	64.1	1.8					25.1	0.6	0.2	Pb 1.3, Zn 0.3	
2	4.4	3.4	0.9	0.7	63.5	2.1					24.3	0.2		Pb 0.7	
3	1.7	9.9		0.2	66.9	10.5								Sr 10.7	
4	20.2	0.6		0.1	78.6	0.3					0.1	0.2			
5	28.1	0.6			70.8	0.3					0.3				
6	22.1	0.9		0.2	76.4	0.4									
7	21.0	1.0		0.4	76.7	0.6					0.2	0.2			
8	24.7	0.5		0.1	74.2	0.3					0.3				

**Backscattered Electron (BSE) Micrograph of Particles at Site 8 in CaCO<sub>3</sub> Leached Sample of C-108 Residual Waste**



**Element Compositions Derived from EDS Analyses of Particles Identified in Electron Micrograph Shown Above Right**

Spectrum	Al	Na	Si	Ca	O	P	F	U	Tc	Cr	Fe	Ni	Mn	Others
	at.% normalized to 100%													
1	9.7	8.9		3.8	68.2	5.1					1.4	2.7		S 0.2
2	24.0	1.3		0.6	72.8	0.7					0.6			
3	9.0	1.9		1.3	30.9	0.4					54.5	0.7		Pb 0.1, Zn 1.2
4	29.6	0.4		0.1	69.6	0.2								
5	19.1	4.5		3.2	67.1	2.7					1.7	1.8		
6	17.9	1.1		0.2	80.1	0.5					0.1	0.1		Cd 0.1



## Distribution

### **No. of Copies**

#### **OFFSITE**

D. A. Dunning (CD)  
Oregon Department of Energy  
625 Marion Street, N.E.  
Salem, OR 97301-3737

D. I. Kaplan (CD)  
Westinghouse Savannah River Company  
Building 774-43A, Room 215  
Aiken, SC 29808

D. Kosson (CD)  
Vanderbilt University  
VU Station B #351831  
2301 Vanderbilt Place  
Nashville, TN 37235-1831

C. Langston (CD)  
Westinghouse Savannah River Company  
Building 774-43A  
Aiken, SC 29808

S. Lilligren (CD)  
Nez Perce Tribe  
P.O. Box 365  
Lapwai, ID 83540

P. R. Reed (CD)  
U.S. Nuclear Regulatory Commission  
MS 9 F39  
Washington, D.C. 20555-0001

W. Rigsbee (CD)  
Yakama Nation  
P.O. Box 151  
Toppenish, WA 98948

### **No. of Copies**

#### **ONSITE**

**2 DOE Office of River Protection**

R. W. Lober (CD) H6-60  
R. A. Quinterro (CD) H6-60

**2 DOE Richland Operations Office**

DOE Public Reading Room (2P) H2-53

**10 Washington River Protection Solutions, LLC**

J. N. Appel (CD) B1-55  
M. P. Connelly (2P) E6-31  
S. J. Eberlein (CD) E6-31  
M. P. Bergeron (CD) E6-31  
D. M. Nguyen (CD) B1-55  
D. Parker (CD) E6-31  
L. Fort (CD) E6-31  
M. B. Skorska (CD) E6-31  
K. D. Quigley (CD) E6-31

**Washington State Department of Ecology**

M. Barnes (CD) H0-57

**16 Pacific Northwest National Laboratory**

B. W. Arey (CD) K8-93  
P. R. Bredt (CD) K6-24  
T. M. Brouns (CD) K9-69  
C. F. Brown (CD) P7-22  
R. W. Bryce (CD) E6-35  
K. J. Cantrell (2P) K6-81  
K. M. Geiszler (CD) P7-22  
A. R. Felmy (CD) K8-96  
M. D. Freshley (CD) K9-33

**No. of  
Copies**

N. Qafoku (CD)  
M. J. Lindberg (CD)  
H. T. Schaef (CD)

K7-58  
P7-22  
K6-81

**No. of  
Copies**

W. Um (CD)  
J. M. Zachara (CD)  
D. E. Kurath (CD)

P7-22  
K8-96  
K3-52



*Proudly Operated by Battelle Since 1965*

902 Battelle Boulevard  
P.O. Box 999  
Richland, WA 99352  
1-888-375-PNNL (7665)

[www.pnl.gov](http://www.pnl.gov)



U.S. DEPARTMENT OF  
**ENERGY**