

# Compilation of Laboratory Scale Aluminum Wash and Leach Report Results

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**Key Words:** Aluminum; Leach; Caustic; Laboratory Data; Hanford; Tank Waste; Boehmite; Gibbsite; Aluminosilicate; Cancrinite.

**Abstract:** This report compiles and analyzes all known wash and caustic leach laboratory studies. As further data is produced, this report will be updated. Included are aluminum mineralogical analysis results as well as a summation of the wash and leach procedures and results. Of the 177 underground storage tanks at Hanford, information was only available for five individual double-shell tanks, forty-one individual single-shell tanks (e.g. thirty-nine 100 series and two 200 series tanks), and twelve grouped tank wastes. Seven of the individual single-shell tank studies provided data for the percent of aluminum removal as a function of time for various caustic concentrations and leaching temperatures. It was determined that in most cases increased leaching temperature, caustic concentration, and leaching time leads to increased dissolution of leachable aluminum solids.

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## EXECUTIVE SUMMARY

This report summarizes published laboratory results for aluminum removal through inhibited water wash and caustic leaching tests performed on Hanford tank wastes. The data compilation and evaluation provided in this document is part of an analysis described in RPP-PLAN-46002, *Wash and Leach Factor Work Plan*. The objective of this report is to analyze the available data and ease the process of determining what information is available. As more test results are published, this compilation will be updated to incorporate the new information.

The laboratory studies currently available provide data on the effects of temperature, caustic concentration, and leaching time on the dissolution of aluminum from archived radioactive tank waste samples. A total of 39 laboratory reports are compiled. In particular, information is provided for five individual double-shell tanks, forty-one individual single-shell tanks (e.g. thirty-nine 100 series and two 200 series tanks), and twelve grouped tank wastes. Of these, only three of the individual double-shell, twelve of the individual single-shell, and seven of the composite grouped tank analyses were performed within the most recent proposed operation temperature range (80 to 90°C) for the Waste Treatment Plant caustic leaching to be performed at the Pretreatment Facility. Of these, only three individual single-shell tank samples and one composite tank waste sample were analyzed without inhibited water washing; a technique that is not planned for use at the Pretreatment Facility. Seven of the individual single-shell tank studies performed within the appropriate temperature range provided concentration versus time data for aluminum leaching at various temperatures and caustic concentrations. This information, provided in the Appendix A3.0, can be used to evaluate how changes in temperature, caustic concentration, and leaching time could affect the extent of aluminum leaching for these waste samples. Overall, increases in these three parameters result in improvements to the removal of aluminum from the solid phase in most cases. Analyses of the solid phases present after inhibited water washing and caustic leaching show that there are three aluminum phases present in the majority of tank waste analyses. Of these, gibbsite was most easily removed, whereas boehmite and aluminosilicates/cantrinites were the most dominant aluminum mineral phases present after leaching.

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## Abbreviations and Acronyms

~	approximately
#	number
1C	first decontamination cycle from bismuth phosphate process at B and T plants
2C	second decontamination cycle from bismuth phosphate process at B and T plants
Al	aluminum
am	amorphous
BARD	Flowsheet Bases, Assumptions, and Requirements Document
BBI	Best Basis Inventory
BiPO <sub>4</sub>	Bismuth Phosphate
CC	Complex Concentrate
Cr	Chromium
cr	crystalline
CW	Cladding Waste
DN/PD	Dilute Non-complexed/PUREX neutralized cladding removal waste (NCRW) solids or sludge remaining after decanting
DST	Double-shell tank
EB	Evaporator bottoms
ECN	Engineering Change Notice
EDS	Energy dispersive spectroscopy
FeCN	Ferrocyanide
FY	fiscal year
HDPE	high-density polyethylene
HLW	High-level Waste
ID	identification
IX	ion exchange
LAW	Low-activity Waste
Na	sodium
NA	ungrouped waste type
N/A	not applicable
NP	not performed
NaOH	sodium hydroxide (caustic)
OWW	Organic wash waste
PO <sub>4</sub>	phosphate
PTF	Pretreatment Facility
PUREX	plutonium-uranium extraction
REDOX	reduction-oxidation
rpm	rotations per minute
RT	room temperature
SEM	scanning electron microscopy
SORWT	Sort on Radioactive Waste Type
SST	Single-shell tank
T	temperature
TBP	Tributyl phosphate

TEM	transmission electron microscopy
TRU	transuranic
UDS	undissolved solids
w/	with
WRPS	Washington River Protection Solutions
WTP	Waste Treatment and Immobilization Plant
XRD	X-ray diffraction

### Units

°C	degrees Celsius
g	gram
hr	hour
ug	microgram
uL	microliter
um	micrometer; micron
mL	milliliter
min	minute
M	mole/liter
%	percent
wt	weight

## 1.0 INTRODUCTION

One of the primary missions at the Hanford site is to manage the 53 million gallons of radioactive waste stored in the 149 single-shell and 28 double-shell underground storage tanks. This includes reducing the worker, public, and environmental risk of contamination by ensuring tank integrity is maintained, ensuring safe working and handling practices, and preparing to transfer and dispose of the waste at the Waste Treatment and Immobilization Plant (WTP) currently being constructed.

The underground storage tanks, which are grouped into 18 tank farms, store wastes generated from the production of nuclear materials which began in 1944. These tanks range in volume from 55 thousand to one million gallon capacity. The waste categories still held within these tanks include aqueous supernatants and solid saltcakes and sludge. These waste categories are subsequently grouped by waste types representative of the processes which produced them. The solid waste types are provided in Table 1-1 below with information from RPP-8847, *Best-Basis Inventory Template Composition of Common Tank Waste Layers*.

**Table 1-1. Solid Waste Types Found in the BBI Calculation Detail Report, as Described in RPP-8847 (3 sheets).**

Waste Type	Waste Type Process Description
1C (solid)	BiPO <sub>4</sub> first cycle decontamination waste and coating waste (1944 to 1949 and 1950 to 1956).
1CFeCN (solid)	Ferrocyanide sludge from in-plant scavenging of 1C waste (without coating waste) from T plant that was transferred to 241-TY tank farm (October 1954 to 1956).
224-1 (solid)	Lanthanum fluoride process 224 building waste (1944 to 1948).
224-2 (solid)	Lanthanum fluoride process 224 building waste (1949 to 1956).
2C (solid)	BiPO <sub>4</sub> second cycle decontamination waste (1944 to 1949 and 1950 to 1956) with low activity cell 5-6 drainage waste (June 1951 to 1956).
A1-SltCk (solid)	Saltcake from the first 242-A evaporator campaign using 241-A-102 as the feed tank (1977 to 1980).
A2-SltSlr (solid)	Saltcake from the second 242-A evaporator campaign using 242-AW-102 as the feed tank (1981 to 1988).
AR (solid)	Water washed PUREX sludge entrained in decants of recovered sludge or the water washes of this sludge and the solids remaining after acidification (1967 to 1976).
B (solid)	B Plant high-activity waste – Rare earth (RE) fission products, recovered current acid waste (CAW), solvent wash waste, and any solution containing high activity including cask station receipts, cell drainage containing product spills, etc. (1963 to 1972).
BL (solid)	B Plant low-activity waste – 1AW solvent extraction waste stream (which includes complexants added for solvent extraction), the 1CP/organic wash waste during PUREX acidified sludge (PAS) processing, and insoluble solids remaining after treatment of solids centrifuged from CAW feed (i.e. acid leached and water washed PUREX HLW sludge). Cell drainage and Waste Encapsulation Storage Facility (WESF) transfers with low radionuclide content (1967 to 1976).
B-SltCk (solid)	Saltcake from the 242-B evaporator operation (1951 to 1954).

**Table 1-1. Solid Waste Types Found in the BBI Calculation Detail Report, as Described in RPP-8847 (3 sheets).**

Waste Type	Waste Type Process Description
BY-SltCk (solid)	Saltcake from in-tank solidification in 241-BY farm (1965 to 1974).
CWP1 (solid)	PUREX cladding waste from aluminum clad fuel (1956 to 1960).
CWP2 (solid)	PUREX cladding waste from aluminum clad fuel (1961 to 1972).
CWR1 (solid)	REDOX cladding waste from aluminum clad fuel (1952 to 1960).
CWR2 (solid)	REDOX cladding waste from aluminum clad fuel (1961 to 1966).
CWZr1 (solid)	PUREX (and REDOX) zirconium cladding waste (1968 to 1972).
CWZr2 (solid)	PUREX zirconium cladding waste (1983 to 1989).
DE (solid)	Diatomaceous earth added to tanks 241-BX-102, 241-SX-113, 241-TX-116, 241-TX-117, 241-TY-106, and 241-U-104.
HS (solid)	Hot Semiworks strontium and RE purification waste (1961 to 1968).
MW1 (solid)	BiPO <sub>4</sub> Metal Waste (1944 to 1949).
MW2 (solid)	BiPO <sub>4</sub> Metal Waste (1950 to 1956).
NA (SltCk)	Mixture of saltcakes from tank transfers and retrievals.
NA (Sludge)	Mixture of sludge material from tank transfers and retrievals.
P1 (solid)	PUREX high-level waste (1956 to 1962).
P2 (solid)	PUREX high-level waste (1963 to 1967).
P3AZ1 (solid)	PUREX high-level waste to AZ-101 (1983 to March 13, 1986).
P3AZ2 (solid)	PUREX high-level waste to AZ-102 (March 13, 1986 to 1990).
PFeCN (solid)	Ferrocyanide sludge from TBP in-plant scavenged supernatant and co-disposed TBP sludge (1954 to 1955).
PL2 (solid)	PUREX organic wash waste and non-boiling waste (1983 to 1988).
Portland Cement (solid)	Portland cement added to tank 241-BY-105.
R1 (solid)	REDOX high-level waste (1952 to 1958).
R2 (solid)	REDOX high-level waste (1959 to 1966).
R-SltCk (solid)	Saltcake from self-concentration in 241-S and 241-SX tank farms (1952 to 1966).
S1-SltCk (solid)	Saltcake from the first 242-S evaporator campaign using 241-S-102 as a feed tank (1973 to 1976).
S2-SltSlr (solid)	Saltcake from the second 242-S evaporator campaign using 241-SY-102 as a feed tank (1977 to 1980).
SRR (solid)	High-activity waste from B Plant processing of PAS, solids centrifuged from AR vault feed, strontium purification wastes after solvent extraction, RE carrier precipitation or ion exchange rework, and other solutions containing activity including cask station receipts, cell drainage containing product spills, WESF returns unsuitable for rework, and crude RE disposal (1969 to 1985).
T1-SltCk (solid)	Saltcake from the 242-T evaporator (1951 to 1955).
T2-SltCk (solid)	Saltcake from the last 242-T evaporator campaign (1965 to 1974).
TBP (solid)	Tributyl phosphate process waste (1952 to 1957).

**Table 1-1. Solid Waste Types Found in the BBI Calculation Detail Report, as Described in RPP-8847 (3 sheets).**

Waste Type	Waste Type Process Description
TFeCN (solid)	Ferrocyanide sludge from supernatant scavenging in the 244-CR Vault (1955 to 1958) consisting of Tributyl phosphate (TBP) supernatant and the comingled supernatants from other wastes stored in the same tanks.
TH1 (solid)	Thoria process wastes (1966).
Z(solid)	PPF waste (1974 to 1988).

In preparation for operation of the WTP, the solid and liquid wastes stored in the tank farms will be separated into two feed types: High-level waste (HLW) and Low-activity waste (LAW). The LAW will be made up of salt solutions containing minimal undissolved solids, whereas the remainder of the radioactive waste will be processed as HLW. In order to reduce the volume of HLW, as well as costs, a Pretreatment Facility (PTF) will be utilized to separate out some of the more soluble non-radioactive species. This facility will implement processes such as caustic leaching to remove aluminum at elevated temperature, oxidative leaching to remove chromium at ambient temperature, ultra filtration solids/liquid concentration, and slurry washing to remove soluble species [24590-WTP-RPT-PT-02-005, *Flowsheet Bases, Assumptions, and Requirements Document (BARD)*, Rev. 5].

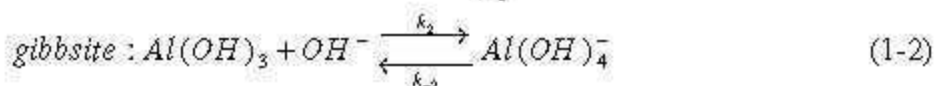
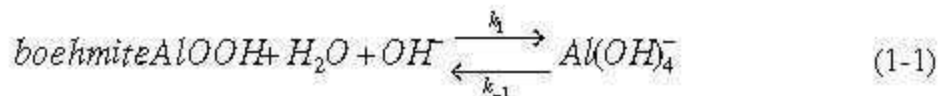
Here, we compile previously documented laboratory data gathered while investigating the removal efficiency of aluminum from tank waste solids through washing and caustic leaching. This is part of the work described in RPP-PLAN-46002, *Wash and Leach Factor Work Plan*. Aluminum concentration has been estimated to be as high as 30 weight percent (wt %) of the sludge material in certain tanks, as detailed in the Best Basis Inventory (BBI) Calculation Detail (WRPS, 2010). When a significant portion of aluminum is removed through caustic leaching, the loading of solid oxides in the HLW glass will be reduced resulting in a reduction of the volume of HLW glass formed.

Removal of aluminum from tank waste through inhibited water washing and caustic leaching has been tested in laboratory settings. The available mineralogical and leaching data has been used in conjunction with other reports (e.g. RPP-RPT-47306, *Waste Type Analysis for Aluminum Leachability Estimates of All Non-Retrieved Hanford Tank Waste* and RPP-RPT-46618, *Hanford Waste Mineralogy Reference Report*) to group the aluminum solid species into three categories:

- (1) Easily leachable aluminum (e.g. gibbsite, dawsonite, nordstrandite, etc.);
- (2) Slow leaching/kinetically controlled aluminum (e.g. boehmite); and
- (3) Non-leachable/refractory aluminum (e.g. aluminosilicates and cancrinites).

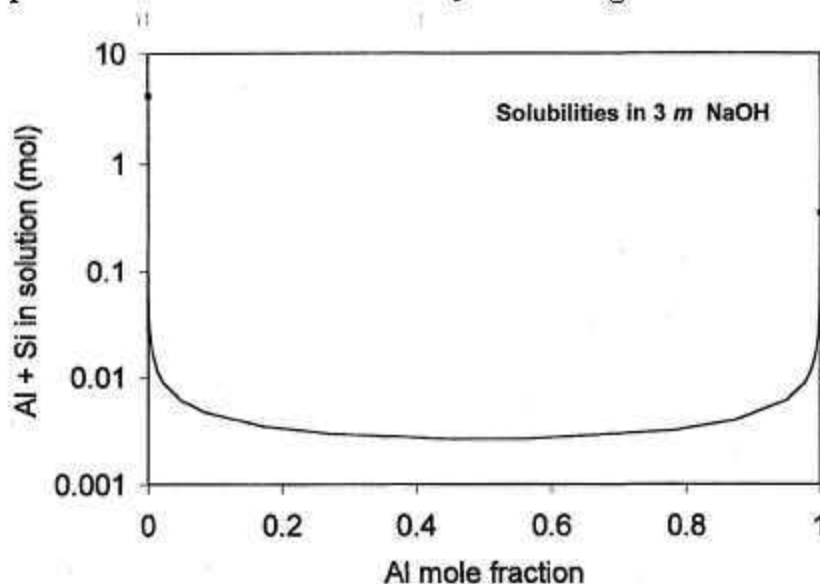
Examples of the chemical structures documented for the non-leachable/refractory aluminum include aluminosilicates ( $H_{12}Al_{12}Si_{36}O_{96}$ ), sodium aluminosilicates hydrate  $[(Na_2O)_{1.31}Al_2O_3(SiO_2)_{2.01}(H_2O)_2]$ , cancrinite  $[(Na_{7.14}Al_6Si_{7.08}O_{26.73}(H_2O)_{4.87})$  or  $(Na_8(AlSiO_4)_6(CO_3)(H_2O)_2)]$ , hydroxycancrinite  $(1.06Na_2O \cdot Al_2O_3 \cdot 1.6SiO_2 \cdot 1.6H_2O)$ , and nitrate cancrinite  $(Na_{8.16}(Al_6Si_6O_{24})(NO_3)_{2.16}(H_2O)_{1.62})$  as provided in RPP-RPT-46618 as well as later in this report.

The three aluminum categories described above have been designed to predict the extent of aluminum solids removal that will be possible during caustic leaching at the PTF. The leachable solids (e.g. gibbsite and, to a small extent, boehmite) dissolve in caustic to form the aluminate ion  $[Al(OH)_4^-]$ , as shown in Equations 1.1 and 1.2, whereas the insoluble solids (e.g. aluminosilicates, cancrinites, etc.) are not easily leached into the aqueous phase with the addition of caustic.



As described in ORNL/TM-1999/263, *Prevention of Solids Formation: Results of the FY 1999 Studies*, the formation of aluminosilicates is a counterintuitive process. Even though both aluminum and silicon have increased solubilities as pH is increased, when both elements are present their solubility drops drastically resulting in the precipitation of aluminosilicates. This trend is shown in Figure 1 of ORNL/TM-1999/263, provided below as Figure 1-1.

**Figure 1-1. Reprint of Aluminosilicate solubility at 25°C figure from ORNL/TM-1999/263.**



The increased pH during caustic leaching would increase the potential for aluminosilicate precipitation, provided that both aluminum and silicon ions are present in solution. This was observed in the caustic leaching of solids from tank 241-BX-112 in PNNL-12026, *Washing and Caustic Leaching of Hanford Tank Sludge: Results of FY 1998 Studies*. Even after performing several wash cycles with the tank 241-BX-112 solids, results of mineralogical analysis indicated the formation of aluminosilicates during leaching, with an increase in formation as temperature was increased.

Many of the 'wash and leach' studies analyzed in this document were performed on archived core and auger samples. For the studies which identified the material used, analyses were performed on samples from one core, composites of many different core samples from the same

tank, or even combinations of tank wastes that fit into specific tank waste groups. Some of these waste samples had been in storage for up to 15+ years and had dried out. Therefore they were reconstituted using water, inhibited water (0.01M NaOH + 0.01M NaNO<sub>2</sub>), or a low concentration (0.01M to 0.1M NaOH) caustic solution in order to obtain a slurry pH near the original measured value. Temperatures used for caustic leaching performance analyses were between room temperature (RT) and 100 degrees Celsius (°C); the majority of which were performed at 100°C (see Table A1-1 and Table A1-2 in the Appendix).

An important aspect for this investigation is to determine how much of the information from the available laboratory analyses is still directly pertinent to the current operational parameters for the PTF at the WTP. To do this, we used the latest revision to 24590-WTP-RPT-PT-02-005 which is a compilation of information describing how the WTP will be run and the operational parameters which will be used for the various treatment processes. The laboratory studies which analyzed inhibited water washing and caustic leaching at 100°C were acceptable according to revision 4 of 24590-WTP-RPT-PT-02-005. Revision 4 allowed for caustic leaching temperatures to range from 85 to 100°C. In revision 5 of 24590-WTP-RPT-PT-02-005, the PTF operational conditions were adjusted, the most noticeable being (a) the removal of the preliminary inhibited water washing and (b) caustic leaching temperatures adjusted to a range of 80 to 90°C. These were important changes, as inhibited water washing was performed in nearly all previously performed laboratory studies, with the majority of caustic leaching tests performed at 100°C. Since both of these conditions are not within the scope of the current operational plan for the PTF, uncertainties in the relevancy of the laboratory results currently available for aluminum leaching exist.

A majority of the laboratory data currently available are from tests performed with preliminary inhibited water washing and caustic leaching outside of the current acceptable temperature range (80 to 90°C). Even though this information is not directly relevant to the current WTP PTF operational parameters, important characteristics can be derived from the available data. These characteristics include the effects of leaching time on aluminum dissolution, the quantities of easily leachable aluminum solids, chemical and physical properties of the various solids currently in the as-received tank waste samples, the types of solids formed during leaching, etcetera.

This report compiles all of the currently available laboratory data available to date. Due to the numerous documents available with pertinent information on this subject matter, all of the existing laboratory results are incorporated within this document in order to provide one reference that contains all of the available data. As further analytical results are documented, the new information will be added.

## **2.0 ALUMINUM CAUSTIC LEACHING LABORATORY STUDIES**

Thirty-nine reports have been generated to date to analyze the effects of inhibited water washing and high temperature caustic leaching on the removal of aluminum from tank waste samples. Significant information concerning aluminum removal has been compiled into Table A1-1 and



Table A1-2 in the appendix of this report, with mineralogy data provided in Table 2-1. The information in Table 2-1 is based solely on information contained in the referenced report and not on any other known information. Similarly, the mineralogical information in the table is based on the analytical results from the referenced report which may not have observed a specific aluminum mineral phase even though aluminum was found in the solids.

Some of the caustic leaching laboratory studies identify aluminum mineral phases present in the as-received and treated materials, as well as analyze the effects of temperature, caustic concentration, and leaching time on the extent of aluminum leaching. Included in Table A1-1 and Table A1-2 is a brief description of the processes that generated the waste found in the individual tanks. These processes have been included to indicate where the aluminum in the specific wastes originated (as referenced in Table 1-1). Understanding these processes provides insight into the conditions (e.g. temperature) at which the aluminum in a waste was exposed to in the past. This information has previously been used to develop trends in the types of aluminum solids present in untested tanks (see RPP-RPT-47306).

Additional mineralogy information was obtained from PNNL-13394, *Status Report on Phase Identification in Hanford Tank Sludges*, and PNWD-3300 (WTP-RPT-076), *Identification of Washed Solids from Hanford Tanks 241-AN-102 and 241-AZ-101 with X-Ray Diffraction, Scanning Electron Microscopy, and Light-Scattering Particle Analysis*, with additional insight into the waste sources stored in the various tanks obtained from PNL-9814, *The Sort On Radioactive Waste Type Model: A Method to Sort Single-Shell Tanks Into Characteristic Groups*, and WHC-EP-0625, *Hanford Site Waste Storage Tank Information Notebook*. Information from these reports is also included in Table 2-1. Tanks that have not had leaching studies performed up to this point are detailed in Table A2-1.

Aluminum mineral phases were observed using techniques such as transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). These analytical techniques were used to determine which aluminum species were dominant prior to and following caustic leaching. As shown in Table 2-1, three aluminum minerals were found to be in a majority of either the removed species due to caustic leaching or still mostly present after caustic leaching. The species dissolved in a majority of the studies was gibbsite (17 studies), since it was observed in the as-received solids but not in the remaining leached solids. The other two, various aluminosilicates (including cancrinites; 16 studies) and boehmite (8 studies), were in a majority of the studies as still present following caustic leaching. The observed reactions to the leaching events for these three mineral phases, which were in a majority of the tank waste samples analyzed, justifies the three solid aluminum mineral categories that have previously been identified in RPP-RPT-47306: (1) easily leachable aluminum (e.g. gibbsite, dawsonite, nordstrandite, etc.); (2) slow leaching/kinetically controlled aluminum (e.g. boehmite); and (3) non-leachable/refractory aluminum (e.g. aluminosilicates).

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-AN-102	None	PNWD-3300 (WTP-RPT-076)	A2-SltSlr (solid) and NA (liquid).	Complexant Concentrate (CC) Waste. <sup>1</sup>	Washed solids: boehmite, zeolite, sodium aluminate, and diaspore.
241-AN-104	100°C	PNNL-11636	A2-SltSlr (liquid and solid).	Primarily "salt cake", characterized as Double-shell slurry feed. Double-shell Slurry Feed. <sup>1,2</sup>	Fine particles (likely sodium aluminate) and Al and Si species with clay-like (or film-like) morphologies. Amorphous (am) aluminosilicate (AlSi). AlSi (am). <sup>2</sup> AlSi (am) in as-received; none identified in leached sludge using TEM. <sup>3</sup>
241-AW-105	None	PNNL-13394	CWZr2 (solid), PL2 (solid), and NA (liquid).	Primarily Neutralized Cladding Removal Waste Dilute Non-complexed/PUREX neutralized cladding removal waste (NCRW) solids or sludge remaining after decanting (DN/PD). <sup>1</sup>	As-received sludge: gibbsite and crystalline (cr) AlSi. Leached sludge: AlSi (cr) identified using TEM/SEM/XRD.
241-AZ-101	80 to 85°C	PNWD-3206 (WTP-RPT-043)	NA (sludge) and P3AZ1 (liquid and solid).	—	In washed solids: Gibbsite, boehmite, and Al-Zr-Fe phase. <sup>4</sup>
241-AZ-101 and 241-AZ-102	80 to 85°C	PNNL-11580	—	Aging. <sup>1</sup>	None identified in as-received; analysis not performed for leached sludge using SEM. <sup>3</sup>
241-AZ-102	80 to 85°C	PNWD-3045 (BNFL-RPT-038)	NA (sludge), P3AZ2 (solid), PL2 (solid), SRR (solid), NA Lower (liquid), and NA Upper (liquid).	—	AlSi (am) in as-received; none identified as present in leached sludge using SEM. <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-SY-101	None	PNNL-13394	S2-SltSlr (solid) and NA (liquid).	CC waste. <sup>1</sup>	As-received sludge: NaAlO <sub>2</sub> and amorphous gibbsite. None identified in leached sludge using TEM/SEM/XRD.
241-SY-102	80 to 85°C	PNWD-3512 (WTP-RPT-117)	R1 (solid), NA (liquid and sludge), and Z (liquid and solid).	DN/PD. <sup>1</sup>	Gibbsite (from washed solids).
241-SY-103	100°C	PNL-10712	S2-SltSlr (liquid and solid).	CC Waste. <sup>1,2</sup>	Al(OH) <sub>3</sub> (am), Al(OH) <sub>3</sub> (cr), Al <sub>2</sub> O <sub>3</sub> ·xH <sub>2</sub> O (cr). <sup>2</sup> Gibbsite (am) and Al <sub>2</sub> O <sub>3</sub> (H <sub>2</sub> O) <sub>x</sub> (cr) in as-received; gibbsite (am) and Al <sub>2</sub> O <sub>3</sub> (H <sub>2</sub> O) <sub>x</sub> (cr) in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>
241-SY-103	100°C	PNNL-11089	S2-SltSlr (liquid and solid).	Primarily CC.	Major phases present in untreated solids: Al(OH) <sub>3</sub> (am and weakly cr), with minor phases of crystalline aluminum oxide hydrate. Neither present after treatment.
241-B-104	100°C	LAUR 96-2839	1C (solid), 2C (solid), and B-SltCk (solid and liquid).	Second decontamination cycle of the BiPO <sub>4</sub> process at B and T Plants (2C); slurry product from evaporator bottoms (EB); TBP waste from U-extraction process at U Plant; 1 <sup>st</sup> decontamination cycle from BiPO <sub>4</sub> process at B and T Plants (1C). Non-Complexed Waste. <sup>1</sup> <u>Primary waste: 2C, Secondary: EB.</u> <sup>2</sup> <u>Primary waste type: 2C. Secondary: EB. Tertiary: TBP. Other: 1C.</u> <sup>3</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-B-106	100°C	LAUR 97-2889	1C (solid), TBP (solid, and B-SltCk (liquid).	1C, TBP from U-extraction process at U Plant, lab waste from the 300 area. Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 1C. <u>Secondary:</u> TBP. <sup>2</sup> <u>Primary waste type:</u> 1C. <u>Secondary:</u> TBP. <u>Tertiary:</u> Hanford Laboratory Operations. <u>Other:</u> mixture of several miscellaneous wastes. <sup>5</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>
241-B-110	100°C	PNL-9387	2C (solid), B (solid), and CSR (liquid).	2C in '40-'50 to recover & purify Pu from irradiated fuel, adjusted to high pH; fission product waste produced in campaigns ('60s) to isolate fission products; ion exchange waste from B Plant Cs recovery process. Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 2C. <u>Secondary:</u> High-level B Plant waste from bottom of Section 5. <sup>2</sup> <u>Primary waste type:</u> 2C. <u>Secondary:</u> high-level B Plant waste. <u>Tertiary:</u> Fission Products Waste. <u>Other:</u> IX waste. <sup>5</sup>	Sodium AlSi hydrate in as-received; none in leached sludge (determined by SEM/XRD). <sup>3</sup>
241-B-111	100°C	PNL-10712	2C (solid), B (solid), and CSR (liquid).	Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 2C. <u>Secondary:</u> High-level B Plant waste from bottom of Section 5. <sup>2</sup> <u>Primary waste type:</u> 2C. <u>Secondary:</u> high-level B Plant waste. <u>Tertiary:</u> Fission Products Waste. <u>Other:</u> IX waste. <sup>5</sup>	AlSi (cr)  AlSi (cr) in as-received; AlSi (cr) in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-B-111	100°C	PNNL-11089	2C (solid), B (solid), and CSR (liquid).	Primarily 2C, with secondary waste from HLW from Tank 5-6 at B Plant.	Minor phase in untreated and treated solids: AlSi (cr).
241-B-202	100°C	LAUR 95-2070	224-2 (solid).	Final decontamination and concentration stage of BiPO <sub>4</sub> process.  <u>Primary waste:</u> Lanthanum fluoride decontamination waste. <sup>2</sup>  <u>Primary waste type:</u> Lanthanum fluoride decontamination waste. <sup>3</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>
241-BX-103	100°C	LAUR 97-2889	CWP2 (solid), TBP (solid), and CWP2 (liquid).	TBP from U-extraction process at U Plant, CW produced at PUREX plant from dissolution of Zr or Al fuel cladding, organic solvent wash waste (OWW) from PUREX.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste type:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> OWW. <u>Other:</u> mixture of several miscellaneous wastes. <sup>5</sup>	Gibbsite in as-received sample.  Gibbsite in as-received; none in leached sludge (determined by XRD). <sup>3</sup>
241-BX-105	100°C	LAUR 95-2070	BY-SrCk (solid), CWP2 (solid), MW1 (solid), TBP (solid), and CWP2 (liquid).	First waste received was TBP from TBP uranium extraction process at U plant; PUREX plant dissolution of zirconium or aluminum fuel cladding; ion exchange (IX) waste from cesium recovery process at B plant; Last was EB consisting of a slurry product from evaporators.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> TBP, <u>Secondary:</u> CW. <sup>2</sup>  <u>Primary waste type:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> IX waste. <u>Other:</u> EB. <sup>5</sup>	<u>As-received XRD:</u> Al hydroxide compound (Gibbsite Al(OH) <sub>3</sub> ). <u>Treated XRD:</u> Al hydroxide compound (Gibbsite Al(OH) <sub>3</sub> ).  Gibbsite in as-received; gibbsite in leached sludge (determined by XRD). <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-BX-107	100°C	PNL-10712	1C (solid).	Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 1C, <u>Secondary:</u> TBP. <sup>2</sup> <u>Primary waste type:</u> 1C. <u>Secondary:</u> TBP. <u>Tertiary:</u> CW. <u>Other:</u> IX waste. <sup>5</sup>	Al(OH) <sub>3</sub> (am), Al(OH) <sub>3</sub> (cr), AlPO <sub>4</sub> , AlSi (am), AlSi (cr)  AlPO <sub>4</sub> , Al(OH) <sub>3</sub> (am), and AlSi (cr and am) in as-received; AlSi (cr, am) in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>
241-BX-107	100°C	PNNL-11089	1C (solid).	Primarily 1C, with secondary waste from the TBP uranium extraction process at U Plant.	Major phases present in untreated tank solids: AlPO <sub>4</sub> , AlSi (cr and am), and aluminum hydroxide (am). AlSi (cr and am) present after treatment.
241-BX-109	100°C	LAUR 96-2839	TBP (solid).	TBP from U-extraction process at U Plant; waste produced at PUREX Plant from dissolution of Zr or Al fuel cladding; 1C; IX waste from Cs recovery process at B Plant.  Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> TBP. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary waste type:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> 1C. <u>Other:</u> IX waste. <sup>5</sup>	Nordstrandite [Al(OH)3] in non-treated waste sample.  Nordstrandite in as-received; none in leached sludge (determined by XRD). <sup>3</sup>
241-BX-112	Multiple Leaching Temperatures	PNNL-12026	1C (solid) and 1C2 (liquid).	<u>Primary waste:</u> 1C. <u>Secondary waste:</u> EB.  Non-Complexed Waste. <sup>1</sup> <u>Primary waste type:</u> 1C. <u>Secondary:</u> EB. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	AlSi formed during leach step - increase w/ increasing temperature (T). Washed solids contained significant concentrations of aluminum phosphates.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-BY-104	100°C	PNNL-11278	BY-SltCk (liquid and solid) and PFeCN (solid).	<p><u>Primary:</u> TBP extraction process waste and FeCN-scavenged waste, <u>Secondary:</u> EB and in-tank solidification.</p> <p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary Waste:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification.<sup>2</sup></p> <p><u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> CW. <u>Other:</u> IX waste.<sup>3</sup></p>	<p><u>Untreated:</u> aluminum oxide and Si/Al clay material (am). <u>Treated:</u> aluminum oxide.</p> <p>Al<sub>2</sub>O<sub>3</sub>•xH<sub>2</sub>O (cr), AlSi (am).</p> <p>(Al<sub>2</sub>O<sub>3</sub>)<sub>x</sub>-(H<sub>2</sub>O)<sub>y</sub> and AlSi (am) in as-received; (Al<sub>2</sub>O<sub>3</sub>)<sub>x</sub>-(H<sub>2</sub>O)<sub>y</sub> in leached sludge (determined by TEM).<sup>3</sup></p>
241-BY-108	100°C	PNNL-11636	BY-SltCk (liquid and solid) and PFeCN (solid).	<p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary Waste:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification.<sup>2</sup></p> <p><u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> 1C. <u>Other:</u> CW.<sup>3</sup></p>	No specific Al mineral phase determined by TEM. <sup>3</sup>
241-BY-110	100°C	PNNL-11278	BY-SltCk (liquid and solid) and PFeCN (solid).	<p><u>Primary:</u> TBP extraction process waste and FeCN-scavenged waste, <u>Secondary:</u> EB and In-tank solidification.</p> <p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary Waste:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification.<sup>2</sup></p> <p><u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> 1C. <u>Other:</u> CW.<sup>3</sup></p>	No specific Al mineral phase determined by TEM. <sup>3</sup>



**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-C-104	Multiple Leaching Temperatures	ORNL TM-13500	CWPI (solid) and NA (sludge).	High aluminum, low phosphorous and bismuth.  High phosphorous sludge with moderate concentrations of aluminum and chromium. Waste types include PUREX aluminum waste and zirconium CW; CC Waste. <sup>1</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>
241-C-105	100°C	LAUR 97-2889	CWPI (solid) and TBP (solid).	TBP waste from U-extraction process at U plant, Sr wash of PUREX waste in AR vault, waste from dissolution of Zr or Al fuel cladding at PUREX, high-activity neutralized acid waste generated by PUREX.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> TBP. <u>Secondary:</u> Particulates from Sr wash of PUREX wastes in the AR vault. <sup>2</sup>  <u>Primary waste type:</u> TBP. <u>Secondary:</u> particulates from Sr wash of PUREX wastes in AR-vault. <u>Tertiary:</u> CW. <u>Other:</u> neutralized acid waste. <sup>5</sup>	Gibbsite in as-received samples.  Al(OH) <sub>3</sub> (cr).  Gibbsite in as-received; none in leached sludge (determined by XRD). <sup>3</sup>
241-C-106	100°C	PNNL-11381	NA (liquid and sludge).	Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> Sr leached sludge. <u>Secondary:</u> Particulates from Sr wash of PUREX wastes in the AR vault. <sup>2</sup>  <u>Primary waste type:</u> Sr sludge. <u>Secondary:</u> particulates from Sr wash of PUREX wastes in the AR-vault. <u>Tertiary:</u> neutralized acid waste. <u>Other:</u> TBP. <sup>5</sup>	<u>Untreated sludge solids:</u> aluminum hydroxide (am) and AlSi (am). <u>After leaching (solids):</u> predominantly AlSi (am).  Al(OH) <sub>3</sub> (am), AlSi (am).  Gibbsite (am) and AlSi (am) in as-received; AlSi (am) in leached sludge (determined by TEM). <sup>3</sup>



**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-C-107	100°C	PNNL-11278	1C (solid), CWP2 (solid), and SRR (solid).	<u>Primary:</u> 1C. <u>Secondary:</u> CW. Dilute Complexed Waste. <sup>1</sup>  <u>Primary waste type:</u> 1C. <u>Secondary:</u> Sr sludge. <u>Tertiary:</u> CW. <u>Other:</u> IX waste. <sup>5</sup>	<u>Untreated and Treated solids:</u> Si/Al clay material (am) and aluminum oxide.  $Al_2O_3 \cdot xH_2O$ (cr), AlSi (am).  ( $Al_2O_3$ ) <sub>x</sub> -(H <sub>2</sub> O) <sub>y</sub> and AlSi (am) in as-received; ( $Al_2O_3$ ) <sub>x</sub> -(H <sub>2</sub> O) <sub>y</sub> and AlSi (am) in leached sludge (determined by TEM). <sup>3</sup>
241-C-108	100°C	LAUR 95-2070	1C (solid).	First waste received was TBP from TBP uranium extraction process at U plant; 1C; CW produced at PUREX plant from dissolution of Zr or Al fuel cladding; final waste was OWW from PUREX plant, containing carbonate, permanganate, and nitrate.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> TBP and FeCN-scavenged waste, <u>Secondary:</u> 1C. <sup>2</sup>  <u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> OWW. <sup>5</sup>	As-received XRD: Al hydroxide compound (Gibbsite Al(OH) <sub>3</sub> ).  Gibbsite in as-received; none identified in leached sludge (determined by XRD). <sup>3</sup>
241-C-109	100°C	PNL-9387	1C (solid).	TBP U extraction process operated at U Plant (wastes treated w/ FeCN to scavenge 137-Cs from supernatant solution); 1C; Al &/or Zr CW; IX waste from B Plant Cs recovery process.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> TBP and FeCN-scavenged waste, <u>Secondary:</u> 1C. <sup>2</sup>  <u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX waste. <sup>5</sup>	Gibbsite in as-received; gibbsite, boehmite, and nickel aluminum oxide in leached sludge (determined by SEM/XRD). <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-C-112	100°C	PNL-9387	1C (solid), CWP1 (solid), CWP2 (solid), HS (solid), and TFeCN (solid).	TBP U extraction process operated at U Plant (wastes treated w/ FeCN to scavenge 137-Cs from supernatant solution); 1C; Al &/or Zr CW; IX waste from B Plant Cs recovery process.  Non-Complexed Waste. <sup>1</sup>  <u>Primary waste:</u> TBP and FeCN-scavenged waste. <u>Secondary:</u> 1C. <sup>2</sup>  <u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX waste. <sup>5</sup>	Al(OH) <sub>3</sub> (cr)  Gibbsite in as-received; none identified in leached sludge (determined by SEM/XRD). <sup>3</sup>
241-S-101	100°C	PNNL-11636	S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), and NA (sludge).	Non-Complexed Waste. <sup>1</sup>  <u>Primary Waste:</u> High-Level REDOX, <u>Secondary:</u> EB. <sup>2</sup>  <u>Primary waste type:</u> High-level REDOX waste. <u>Secondary:</u> EB. <u>Tertiary:</u> IX waste. <u>Other:</u> mixture of several miscellaneous wastes. <sup>5</sup>	Boehmite was the predominant solid phase; leached solids contained AlSi (am) and rod-like aluminum oxyhydroxide (diaspore).  Al(O)OH (boehmite), Al(O)OH (diaspore), Al(OH) <sub>3</sub> (cr), AlSi (am).  Boehmite in as-received; boehmite, AlSi (am), and diaspore in leached sludge (determined by TEM). <sup>3</sup>
241-S-104	100°C	LAUR 95-2070	R-SltCk (liquid and solid), CWR1 (solid), and R1 (solid).	<u>Primary waste:</u> High level waste from the REDOX process; Non-Complexed Waste. <sup>1</sup>	<u>As-received XRD:</u> two Al oxide-hydroxide compounds [AlO(OH) and Al <sub>45</sub> O <sub>45</sub> (OH) <sub>45</sub> Cl]. <u>Treated XRD:</u> Al oxide-hydroxide compound [AlO(OH)].
241-S-104	100°C	PNNL-11089	R-SltCk (liquid and solid), CWR1 (solid), and R1 (solid).	Primarily reduction oxidation (REDOX) process HLW.	Boehmite present in treated and untreated tank solids.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-S-104	100°C	PNNL-11636	R-SltCk (liquid and solid), CWR1 (solid), and R1 (solid).	Non-Complexed Waste. <sup>1</sup> <u>Primary Waste:</u> High-Level REDOX. <sup>2</sup>	Boehmite (AlOOH) indicated as the predominant phase present. Some clay, some iron-containing particles, and some other minor phases were also observed.  Al(O)OH (boehmite), (PNL-10712)  Boehmite in as-received; boehmite and AlSi (am) in leached sludge (determined by TEM). <sup>3</sup>
241-S-107	100°C	PNNL-11278	S1-SltCk (solid), S2-SltSlr (solid), T2-SltCk (solid), CWR1 (solid), CWR2 (solid), CWZr1 (solid), and R1 (solid).	<u>Primary:</u> High-level REDOX process waste. <u>Secondary:</u> EB. Non-Complexed Waste. <sup>1</sup> <u>Primary Waste:</u> High-Level REDOX, <u>Secondary:</u> EB. <sup>2</sup>	Untreated and Treated solids: boehmite and Si/Al (am) clay material: Al(O)OH (boehmite), AlSi (am)  Boehmite and AlSi (am) in as-received; boehmite and AlSi (am) in leached sludge (determined by TEM). <sup>3</sup>
241-S-110	Multiple Leaching Temperatures	PNNL-13702	S1-Sltck (liquid and solid), CWR1 (solid), and R1 (solid).	Non-Complexed Waste. <sup>1</sup> <u>Primary waste type:</u> High-level REDOX waste. <u>Secondary:</u> EB. <u>Tertiary:</u> mixture of several miscellaneous wastes. <sup>5</sup>	XRD analysis of washed solids indicated crystalline forms of Al were 10-20% gibbsite and 80-90% boehmite.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-S-110	Caustic and Oxidative Leaching Performed	PNNL-14018	S1-Sltck (liquid and solid), CWR1 (solid), and R1 (solid).	<p>Primarily REDOX waste, with secondary stream from EB and tertiary stream composed as a mixture of several miscellaneous wastes.</p> <p>From cluster analysis, contains saltcake from 242A campaign (1976-1980 and 1981-1988), saltcake from the 242-B operation (1951-1953), saltcake from the 242-S campaign (1973-1976), saltcake from self condensation, REDOX waste, and saltcake from the 242-S campaign (1977-1980).</p> <p>Non-Complexed Waste.<sup>1</sup></p>	XRD analysis of washed sludge shows both (80-90%) boehmite and (10-20%) gibbsite present.
241-S-111	100°C	PNNL-11636	S1-Sltck (liquid and solid), CWR1 (solid), and R1 (solid).	<p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary Waste:</u> High-Level REDOX, <u>Secondary:</u> EB.<sup>2</sup></p> <p><u>Primary waste type:</u> High-level REDOX waste. <u>Secondary:</u> EB.<sup>3</sup></p>	<p>Leached solids major phases were bayerite [Al(OH)3] and boehmite [AlOOH].</p> <p>Al(O)OH (boehmite), Al(OH)3 (cr).<sup>2</sup></p> <p>Bayerite and boehmite in leached sludge (determined by TEM).<sup>3</sup></p>
241-SX-101	80 to 85°C	PNWD-3512 (WTP-RPT-117)	R-SltCk (liquid and solid), S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), and R1 (solid).	Dilute Complexed Waste. <sup>1</sup>	"Some sort of Al(OH) <sub>3</sub> is postulated from washed solids analysis."

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-SX-108	100°C	PNNL-11278	R1 (solid) and R2 (solid).	<u>Primary:</u> High-level REDOX process waste. Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> High level REDOX. <sup>2</sup>	<u>Untreated:</u> boehmite, aluminum oxide, Al/Si clay (am). <u>Treated:</u> boehmite, aluminum oxide, AlSi (am), and Ca <sub>3</sub> Al <sub>2</sub> O <sub>6</sub> . Al(O)OH (boehmite), Al <sub>2</sub> O <sub>3</sub> •xH <sub>2</sub> O (cr), AlSi (am), Ca <sub>3</sub> Al <sub>2</sub> O <sub>6</sub> . Boehmite, AlSi (am), and (Al <sub>2</sub> O <sub>3</sub> ) <sub>x</sub> -(H <sub>2</sub> O) <sub>y</sub> in as-received; boehmite, AlSi (am), (Al <sub>2</sub> O <sub>3</sub> ) <sub>x</sub> -(H <sub>2</sub> O) <sub>y</sub> , and Ca <sub>3</sub> Al <sub>2</sub> O <sub>6</sub> in leached sludge (determined by TEM). <sup>3</sup>
241-SX-113	100°C	LAUR 97-2889	DE (solid) and R1 (solid).	Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> High level REDOX, <u>Secondary:</u> Diatomaceous earth. <sup>2</sup> <u>Primary waste type:</u> high-level REDOX waste. <u>Secondary:</u> diatomaceous earth. <sup>3</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>
241-T-104	100°C	PNNL-11089	1C (solid).	Primarily 1C.	Major phases present in untreated solids: AlPO <sub>4</sub> , AlSi (am and cr), and aluminum hydroxide (am). AlSi (am and cr) present after treatment. Al(OH) <sub>3</sub> (am), AlPO <sub>4</sub> , AlSi (am), AlSi (cr), (PNL-10712) AlPO <sub>4</sub> , gibbsite (am), AlSi (am and cr), and Na <sub>2</sub> Fe <sub>2</sub> Al(PO <sub>4</sub> ) <sub>3</sub> in as-received. AlSi (am and cr) and Bi <sub>24</sub> Al <sub>2</sub> O <sub>39</sub> in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>
241-T-104	100°C	LAUR 95-2070	1C (solid).	1C. Non-Complexed Waste. <sup>1</sup>	<u>In as received:</u> Na <sub>2</sub> Fe <sub>2</sub> Al(PO <sub>4</sub> ) <sub>3</sub> - Ferrowyllieite and Bi <sub>24</sub> Al <sub>2</sub> O <sub>39</sub> - Aluminum Bismuth Oxide. <u>In treated:</u> 2 types of sodium AlSi.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-T-107	100°C	LAUR 95-2070	1C (solid), CWP2 (solid), and TBP (solid).	Three significant types of waste: 1C, PUREX CW, and TBP from the TBP uranium-extraction process at U Plant. Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 1C. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX waste. <sup>5</sup>	<u>In as received:</u> gibbsite. <u>In treated:</u> sodium aluminum carbonate, albite, sodium aluminum nitrate silicon, berlinite.  Gibbsite in as-received; AlSi (cr) and Al(PO <sub>4</sub> ) in leached sludge (determined by XRD). <sup>3</sup>
241-T-111	100°C	PNNL-11089	224-2 (solid) and 2C (solid).	<u>Primary:</u> 2C. <u>Secondary:</u> Lanthanum Fluoride Decontamination waste from BiPO <sub>4</sub> Process. Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> 2C, <u>Secondary:</u> Lanthanum fluoride decontamination. <sup>2</sup> <u>Primary waste type:</u> 2C. <u>Secondary:</u> lanthanum fluoride decontamination waste. <sup>5</sup>	No specific phase identified in as-received or in leached sludge using TEM/SEM/XRD. <sup>3</sup>

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
241-TY-104	100°C	LAUR 96-2839	1C/FeCN (solid), TBP (solid), and DW (liquid).	TBP waste from U-extraction process at U Plant; 1C which had been FeCN scavenged; decontamination waste wash solution from equipment decontamination efforts at T Plant (mostly dilute NaNO <sub>2</sub> solution). Non-Complexed Waste. <sup>1</sup> <u>Primary waste:</u> TBP. <u>Secondary:</u> 1C and FeCN-scavenged waste. <sup>2</sup> <u>Primary waste type:</u> TBP <u>Secondary:</u> 1C/FeCN-scavenged waste. <u>Tertiary:</u> decontamination waste. <u>Other:</u> mixture of several miscellaneous wastes/high-level REDOX waste. <sup>3</sup>	No specific phase identified in as-received or in leached sludge using XRD. <sup>3</sup>
241-U-108	Caustic and Oxidative Leaching Performed	PNNL-14019	S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), and CWR2 (solid).	Tank waste group 17. Non-Complexed Waste. <sup>1</sup>	Two Al species found in washed tank sludge: gibbsite, Al(OH) <sub>3</sub> , and an AlSi <sub>2</sub> Hydroxycancrinite, (Na <sub>2</sub> O) <sub>1.06</sub> (Al <sub>2</sub> O <sub>3</sub> )(SiO <sub>2</sub> ) <sub>1.6</sub> (H <sub>2</sub> O) <sub>1.6</sub> .
241-U-110	100°C	PNL-9387	1C (solid), CWR1 (solid), and R1 (solid).	Non-Complexed Waste <sup>1</sup> . <u>Primary waste:</u> 1C. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary waste type:</u> 1C. <u>Secondary:</u> CW. <u>Tertiary:</u> high-level REDOX waste. <u>Other:</u> Laboratory waste from 222-S building. <sup>3</sup>	Gibbsite and boehmite in as-received (determined by SEM/XRD). <sup>3</sup>
Group 1 Bismuth Phosphate Sludge (1C and 2C) - Tanks 241-B-104, 241-BX-112 and 241-T-104	Multiple Leaching Temperatures	PNNL-17992 (WTP-RPT-166)	—	Composite of 31 samples. 93% from B-104, 4% from BX-112, and 3% from T-104. Solids passed through 3.2 mm sieve. Additional 742.97 g DI added during compositing.	<u>Washed solids:</u> Probable phase is ammonium aluminum hydrogen phosphate (NH <sub>4</sub> AlHPO <sub>3</sub> O <sub>10</sub> ). Possible phases present include boehmite and vauxite [FeAl <sub>2</sub> (PO <sub>4</sub> ) <sub>2</sub> (OH) <sub>2</sub> •6H <sub>2</sub> O]. <u>Leached solids:</u> Boehmite and possible berlinite (AlPO <sub>4</sub> ).

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
Group 2 Bismuth Phosphate Saltcake (BY and T saltcake) - Tanks 241-BX-110, 241-BX-111, 241-BY-104, 241-BY-105, 241-BY-107, 241-BY-108, 241-BY-109, 241-BY-110, 241-BY-112, 241-T-108, 241-T-109, 241-TX-104, and 241-TX-113.	Multiple Leaching Temperatures	PNNL-17992 (WTP-RPT-166)	—	Composite of 41 samples from 13 tanks: 7.1% from BX-110, 11.6% from BX-111, 5.9% from BY-104, 4% from BY-105, 10.4% from BY-107, 24.9% from BY-108, 11% from BY-109, 8.2% from BY-110, 2.4% from BY-112, 1% from T-108, 1.8% from T-109, 4% from TX-104, and 7.6% from TX-113. Total of 1966 g solids passed through 3.2 mm sieve. Additional 2973.6 g of water added. Homogenized sample allowed settling over the weekend to allow separation of settled solids for sampling.	<u>Washed solids:</u> gibbsite (cr) and cancrinite [cr; $\text{Na}_{7.92}(\text{AlSiO}_4)_6(\text{NO}_3)_{1.7}(\text{H}_2\text{O})_{2.34}$ ], also AlSi (possibly cancrinite) determined using TEM. <u>Leached solids:</u> cancrinite (cr) [ $\text{Na}_{7.14}\text{Al}_6\text{Si}_{7.08}\text{O}_{26.73}(\text{H}_2\text{O})_{4.87}$ ].
Group 3 PUREX Cladding Waste Sludge - Tanks 241-C-103, 241-B-109, 241-C-105, 241-B-108, 241-C-104, and 241-BY-109.	Multiple Leaching Temperatures	PNNL-18054 (WTP-RPT-167)	—	Composite of 15 samples from 6 tanks: 51% from BY-109, 11% from B-108, 14% from C-105, 11% from B-109, 10% from C-104, and 3% from C-103. Total of 509.23 g from original samples transferred to composite. Total composite slurry weight transferred (including transfer water) was 947,245 g. This was passed through a stainless steel strainer to ensure no large pieces of foreign material in composite. Total sample weight was 1016.81 g after 11 days.	<u>Washed solids:</u> 87.6 wt% gibbsite and ~5.5 wt% Nitrate Cancrinite [ $\text{Na}_{8.16}(\text{Al}_6\text{Si}_6\text{O}_{24})(\text{NO}_3)_{2.16}(\text{H}_2\text{O})_{1.62}$ ] and/or Hydroxycancrinite [ $1.06\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 1.6\text{SiO}_2\cdot 1.6\text{H}_2\text{O}$ ]. <u>Leached solids:</u> no specific aluminum species found.



**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<b>Tank</b>	<b>Leaching Temp. (°C)</b>	<b>Lab Study Reference</b>	<b>Waste type (WRPS, 2010).</b>	<b>Waste Sources.</b>	<b>Aluminum minerals determined in as-received, washed, or caustic leached solids.</b>
Group 4 REDOX Cladding Waste Sludge - Tanks 241-U-105, 241-U-201, 241-U-202, 241-U-203, and 241-U-204.	Multiple Leaching Temperatures	PNNL-18054 (WTP-RPT-167)	—	Composite of 14 samples from 5 tanks: 32% from U-202, 34% from U-105, 17% from U-204, 13% from U-201, and 4% from U-203. Total of 572.02 g from original samples transferred to composite. Total composite slurry weight transferred (including transfer water) was 666.02 g. This was passed through a stainless steel strainer to ensure no large pieces of foreign material in composite. Total sample weight was 1055.14 g after 4 days.	<u>Washed solids:</u> 91.2 wt% gibbsite (cr), 1.5 wt% sodium aluminum silicate hydrate $[(\text{Na}_2\text{O})_{1.31}\text{Al}_2\text{O}_3(\text{SiO}_2)_{2.01}(\text{H}_2\text{O})_{1.65}]$ , and 0.73 wt% boehmite (possible) determined using XRD. TEM showed presence of boehmite, gibbsite, and cancrinite (Al:Si ratio of 1:1 = zeolitic aluminosilicate). <u>Leached solids:</u> tests not performed, but nearly completely dissolved Al (<20% remained).
Group 5 REDOX - Tanks 241-S-101, 241-S-110, 241-S-107, and 241-SX-103.	Multiple Leaching Temperatures	PNNL-17368 (WTP-RPT-157)	—	1283 g from 241-S-101, 397 g from 241-S-110, 213 g from 241-S-107, and 78 g from 241-SX-103. Composite of 39 different archived samples. Solids passed through 1/8 inch stainless steel sieve, with ~ 4 L DI added to solids to homogenize.	<u>Washed solids XRD:</u> dominantly boehmite (73.8% of mass fraction) with minor gibbsite (5.7% of mass fraction) with the average gibbsite to boehmite phase ratio being 0.077. <u>Washed and leached solids XRD:</u> Boehmite (15 wt%) and Sodium AlSi (9 wt%; $\text{NaSiAlO}_4$ ).
Group 6 S-Saltcake - Tanks 241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106.	100°C	PNNL-17368 (WTP-RPT-157)	—	965.3 g from SX-106, 227.3 g from SY-103, 151.7 g from SX-105, 131.5 g from SX-102, 76.2 g from S-111, 55.1 g from U-108, 53.4 g from U-103, 30.3 g from S-106. Composite of 28 different archived samples. Solids passed through 1/8 inch stainless steel sieve to remove large particles (-5.7 g). Added DI to make total volume ~3 L.	<u>Washed solids XRD:</u> dominantly gibbsite (39.4 wt%), with minor boehmite (8.9 wt%), cancrinite (6.7 wt%), and aluminum oxide. The material was dominated by aluminum. <u>Washed and leached solids XRD:</u> dominantly boehmite, followed by hydroxycancrinite $(1.06\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 1.6\text{SiO}_2\cdot 1.6\text{H}_2\text{O})$ and gibbsite; also calcium aluminum oxide ( $\text{CaAl}_2\text{O}_4$ ) seen in one peak.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
Group 7 TBP Waste Sludge - Tanks 241-BX-109 and 241-B-106.	Multiple Leaching Temperatures	PNNL-18119 (WTP-RPT-169)	—	Composite of 33 samples from 2 tanks. 92% from BX-109 and 8% from B-106. Total of 1479.16 g from original samples transferred to composite. A total of 779.7 g DI added during compositing. This was passed through a stainless steel strainer to ensure no large pieces of foreign material in composite.	<u>Washed solids:</u> Zeolite [NaAlSiO <sub>4</sub> (H <sub>2</sub> O) <sub>1.1</sub> ], Thredgoldite [Al(UO <sub>2</sub> ) <sub>2</sub> (PO <sub>4</sub> ) <sub>2</sub> (OH)(H <sub>2</sub> O) <sub>8</sub> ], and Gibbsite[Al(OH) <sub>3</sub> ]. <u>Leached solids:</u> a sodium aluminophosphate phase and Al-Zr-U region determined by TEM analysis.
Group 8 FeCN Waste Sludge - Tanks 241-BY-110, 241-BY-108, 241-BY-105, 241-BY-104, and 241-BY-106.	60°C	PNNL-18120 (WTP-RPT-170)	—	Composite of 30 samples from 5 tanks. 45% from BY-110, 22% from BY-108, 21% from BY-105, 10% from BY-104, and 2% from BY-106. Total of 1358.8 g from original samples transferred to composite. A total of ~960 g DI added during compositing. This was passed through a sieve (collect objects with greater than 3mm diameter) to ensure no large pieces of foreign material in composite. No significant sample was trapped in sieve.	<u>Washed solids:</u> Gibbsite, Sodium Aluminum Iron Oxide (possible) [Na <sub>2</sub> Al <sub>0.5</sub> Fe <sub>0.5</sub> O <sub>15</sub> ], Hydroxycancrinite [1.06Na <sub>2</sub> O•Al <sub>2</sub> O <sub>3</sub> •1.6SiO <sub>2</sub> •1.6H <sub>2</sub> O], and Ammonium Aluminum Hydrogen Phosphate Hydrate (possible) [NH <sub>4</sub> AlH <sub>2</sub> (PO <sub>4</sub> ) <sub>2</sub> •0.5H <sub>2</sub> O]. <u>Leached solids:</u> 63 wt% Al removed. All Gibbsite dissolved. Hydroxycancrinite determined by SEM-EDS.

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<u>Tank</u>	<u>Leaching Temp. (°C)</u>	<u>Lab Study Reference</u>	<u>Waste type (WRPS, 2010).</u>	<u>Waste Sources.</u>	<u>Aluminum minerals determined in as-received, washed, or caustic leached solids.</u>
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant - Tanks 241-S-101, 241-S-110, 241-S-107, and 241-SX-103 from Group 5 and 241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106 from Group 6.	100°C	PNNL-18007 (WTP-RPT-171)	—	Group 5 solids contained 33 wt% Al, with ~90 wt% Al in the form of boehmite. The individual tank waste material sources, compositions, and physical properties are described in PNNL-17368 above.	<u>Initial solids:</u> boehmite ( $\text{AlOOH}$ ), gibbsite ( $\text{Al}(\text{OH})_3$ ), hydroxycancrinite ( $1.06\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 1.6\text{SiO}_2\cdot 1.6\text{H}_2\text{O}$ ), and cancrinite ( $\text{Na}_{7.14}\text{Al}_6\text{Si}_{7.08}\text{O}_{26.73}(\text{H}_2\text{O})_{4.87}$ ). <u>Caustic wash and leached solids:</u> boehmite ( $\text{AlOOH}$ ), cancrinite ( $\text{Na}_{7.14}\text{Al}_6\text{Si}_{7.08}\text{O}_{26.73}(\text{H}_2\text{O})_{4.87}$ ), and hydroxycancrinite ( $1.06\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 1.6\text{SiO}_2\cdot 1.6\text{H}_2\text{O}$ ), with possible additional phases of calcium aluminum oxide hydrate ( $\text{Ca}_2\text{Al}_2\text{O}_5\cdot 8\text{H}_2\text{O}$ ) and hydrogen aluminum silicate ( $\text{H}_{12}\text{Al}_{12}\text{Si}_{36}\text{O}_{96}$ ).

**Table 2-1. Aluminum mineralogical data as described in individual laboratory studies. Included is information prior to leaching, after washing, and after caustic leaching (19 sheets).**

<b>Tank</b>	<b>Leaching Temp. (°C)</b>	<b>Lab Study Reference</b>	<b>Waste type (WRPS, 2010).</b>	<b>Waste Sources.</b>	<b>Aluminum minerals determined in as-received, washed, or caustic leached solids.</b>
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant. Tanks 241-S-101, 241-S-110, 241-S-107, and 241-SX-103 from Group 5 and 241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106 from Group 6.	100°C	PNNL-17965 (WTP-RPT-172)	—	<p><u>Group 5 Caustic Leach Test:</u> 1.8 L Group 5 composite [18 wt% undissolved solids (UDS)] diluted with 2.1 L simulant supernate solution based upon permeate composition reported in PNNL-17368 (WTP-RPT-157).</p> <p><u>Group 5 + Group 6 Caustic Leach Test:</u> 1157 g Group 5 slurry + 613 g Group 6 slurry.</p>	<p>Pre-leached slurry had ~60 mg/g Al; Wash/Leached slurry had ~10 mg/g Al.</p> <p><u>Group 5 Leached Slurry:</u> boehmite [AlO(OH)], with minor contributions of cancrinite [Na<sub>7.6</sub>(Al<sub>6</sub>Si<sub>6</sub>O<sub>24</sub>)(HCO<sub>3</sub>)<sub>12</sub>(CO<sub>3</sub>)<sub>0.2</sub>(H<sub>2</sub>O)<sub>7.26</sub>].</p> <p><u>Group 5 + 6 Leached Slurry:</u> Boehmite [AlO(OH)] was the predominant phase found by XRD analysis along with cancrinite [Na<sub>7.14</sub>Al<sub>5</sub>Si<sub>7.08</sub>O<sub>26.73</sub>(H<sub>2</sub>O)<sub>4.87</sub>] and anorthite [(Ca,Na)(Si,Al)<sub>4</sub>O<sub>8</sub>] which was possibly present, but not confirmed.</p>
Mixture of Group 3 and Group 4 composites - Tanks 241-C-103, 241-B-109, 241-C-105, 241-B-108, 241-C-104, and 241-BY-109 from Group 3 and Tanks 241-U-105, 241-U-201, 241-U-202, 241-U-203, and 241-U-204 from Group 4.	100°C	PNNL-18048 (WTP-RPT-181)	—	490 mL Group 3 composite (598 g at 28.8 wt% UDS) mixed with 540 mL Group 4 composite (710 g at 29 wt% UDS) with 2.88 L simulant supernate added to dilute blended slurry to 9 wt% UDS. Filtered solids were subsequently caustic leached. Solids estimated at 94 g Al in 390 g UDS.	<p><u>Initial washed solids:</u> Insoluble Al in these tanks were mainly gibbsite, with minor phases of sodium AlSi [(Na<sub>2</sub>O)<sub>1.31</sub>Al<sub>2</sub>O<sub>3</sub>(SiO<sub>2</sub>)<sub>2.01</sub>(H<sub>2</sub>O)<sub>2</sub>] and cancrinite [Na<sub>8</sub>(AlSiO<sub>4</sub>)<sub>6</sub>(CO<sub>3</sub>)(H<sub>2</sub>O)<sub>2</sub>].</p> <p><u>Leached solids:</u> Aluminum fluoride hydroxide [AlF<sub>1.96</sub>(OH)<sub>1.04</sub>] possible, calcium iron aluminum oxide [Ca<sub>2</sub>Fe<sub>1.28</sub>Al<sub>0.72</sub>O<sub>5</sub>] possible, cancrinite [Na<sub>8</sub>(AlSiO<sub>4</sub>)<sub>6</sub>(CO<sub>3</sub>)(H<sub>2</sub>O)<sub>2</sub>] observed, aluminophosphate zeolite [(NH<sub>3</sub>(C<sub>4</sub>H<sub>9</sub>))<sub>2</sub>]H(Si<sub>12</sub>P<sub>3</sub>O<sub>12</sub>) possible,</p>
References: <sup>1</sup> WHC-EP-0625; <sup>2</sup> PNNL-11636, <i>Washing and Caustic Leaching of Hanford Tank Sludge: Results of FY 1997 Studies</i> ; <sup>3</sup> PNNL-13394; <sup>4</sup> PNWD-3300 (WTP-RPT-076); and <sup>5</sup> PNL-9814.					

## 2.1 SUMMARY OF TANK LEACHING DATA

As shown in Table A1-1 and Table A1-2 of Appendix A1.0, a total of 5 double-shell tanks (DSTs), 41 single-shell tanks (SSTs), and 12 composite mixtures of tank wastes have had water washing and caustic leaching analyses performed to determine the extent of aluminum dissolution from their respective solid phases. A brief summary of the tanks and the conditions of their analyses are provided in Table 2-2 below. The remaining 108 SSTs and 23 DSTs have not had aluminum leaching tests performed to our knowledge.

**Table 2-2. DSTs and SSTs with Available Laboratory Scale Aluminum Leaching Data (4 sheets).**

SST/DST	Tank number <sup>1</sup>	Temperatures studied; °C	Caustic or Caustic and Oxidative leaching	Reference(s)
DST	241-AN-104	100 °C	caustic	PNNL-11636
DST	241-AZ-101	85 °C	caustic	PNWD-3206 (WTP-RPT-043)
DST	241-AZ-101 + 241-AZ-102	80 °C	caustic	PNNL-11580
DST	241-AZ-102	85 °C	caustic	PNWD-3045 (BNFL-RPT-038)
DST	241-SY-102	85 °C	caustic	PNWD-3512 (WTP-RPT-117)
DST	241-SY-103	100 °C	caustic	PNL-10712 and PNNL-11089
SST	241-B-101	60 °C and 100 °C	caustic	PNNL-12026
SST	241-B-104	100 °C	caustic	LAUR 96-2839
SST	241-B-106	100 °C	caustic	LAUR 97-2889
SST	241-B-110	100 °C	caustic	PNL-9387 and Internal Letter 9404238
SST	241-B-111	100 °C	caustic	PNL-10712 and PNNL-11089
SST	241-B-201	100 °C	caustic	PNL-10078
SST	241-B-202	100 °C	caustic	LAUR 95-2070
SST	241-BX-103	100 °C	caustic	LAUR 97-2889
SST	241-BY-105	100 °C	caustic	LAUR 95-2070
SST	241-BY-107	100 °C	caustic	PNL-10712 and PNNL-11089
SST	241-BX-109	100 °C	caustic	LAUR 96-2839
SST	241-BX-110	60 °C, 80 °C, and 95 °C	caustic	PNNL-12026
SST	241-BX-112	60 °C, 80 °C, and 100 °C	caustic	PNNL-12026
SST	241-BY-104	100 °C	caustic	PNNL-11278

<sup>1</sup> Multiple tank names indicate a composite was made with samples from all of the tanks indicated.

**Table 2-2. DSTs and SSTs with Available Laboratory Scale Aluminum Leaching Data (4 sheets).**

SST/DST	Tank number <sup>1</sup>	Temperatures studied; °C	Caustic or Caustic and Oxidative leaching	Reference(s)
SST	241-BY-108	100 °C	caustic	PNNL-11636
SST	241-BY-110	100 °C	caustic	PNNL-11278
SST	241-C-102	60 °C and 100 °C	caustic	PNNL-12026
SST	241-C-103	100 °C	caustic	PNL-10712 and PNNL-11089
SST	241-C-104	50 °C, 80 °C, 85 °C, 93 °C, and 100 °C	caustic	LAUR 97-2889, ORNL/TM-13500, PNWD-3024 (BNFL-RPT-030), and PNWD-3027 (BNFL-RPT-021)
SST	241-C-105	70°C and 100°C	caustic	LAUR 97-2889 and ORNL/TM-13500
SST	241-C-106	85°C and 100°C	caustic	PNWD-3013 (BNFL-RPT-017), PNNL-11381, and PNNL-11432
SST	241-C-107	70°C and 100°C	caustic	ORNL/TM-13500, LAUR 96-2839, and PNNL-11278
SST	241-C-108	100°C	caustic	LAUR 95-2070
SST	241-C-109	100°C	caustic	PNL-9387
SST	241-C-112	100°C	caustic	PNL-9387
SST	241-S-101	70°C, 95°C, 93°C, and 100°C	caustic	PNNL-12026, PNNL-11636, ORNL/TM-13500, and ORNL/TM-13655
SST	241-S-104	67°C, 70°C, 80°C, and 100°C	caustic	ORNL/TM-13500, PNL-10712, LAUR 95-2070, PNNL-11089, and PNNL-11636
SST	241-S-107	100°C	caustic	PNNL-11278 and PNNL-12010
SST	241-S-110	60°C, 80°C, and 100°C	caustic	PNNL-13702
SST	241-S-110	30°C, 80°C, and 85°C	Caustic and oxidative	PNNL-14018
SST	241-S-111	100°C	caustic	PNNL-11636
SST	241-SX-101	85°C	caustic	PNWD-3512 (WTP-RPT-117)
SST	241-SX-108	100°C	caustic	PNNL-11278
SST	241-SX-108	80°C	Caustic and oxidative	PNNL-11908
SST	241-SX-113	70°C, 80°C, and 100°C	caustic	LAUR 97-2889 and ORNL/TM-13500

**Table 2-2. DSTs and SSTs with Available Laboratory Scale Aluminum Leaching Data (4 sheets).**

SST/DST	Tank number <sup>1</sup>	Temperatures studied; °C	Caustic or Caustic and Oxidative leaching	Reference(s)
SST	241-T-104	RT, 60°C, 95°C, and 100°C	caustic	ORNL/TM-13660, PNL-10712, PNNL-11089, and LAUR 95-2070
SST	241-T-107	100°C	caustic	LAUR 95-2070
SST	241-T-110	60°C, 80°C, and 100°C	caustic	PNNL-13956
SST	241-T-111	100°C	caustic	PNL-10712 and PNNL-11089
SST	241-TY-104	100°C	caustic	LAUR 96-2839
SST	241-U-108	RT, 80°C, 30°C, and 85°C	Caustic and oxidative	PNNL-11908 and PNNL-14019
SST	241-U-109	RT and 80°C	Caustic and oxidative	PNNL-11908
SST	241-U-110	100°C	caustic	PNL-9387, PNL-10078, and PNNL-11779
SSTs	241-B-104, 241-BX-112, and 241-T-104. (BiPO <sub>4</sub> sludge waste)	40°C, 60°C, and 80°C	caustic	PNNL-17992 (WTP-RPT-166)
SSTs	241-BX-110, 241-BX-111, 241-BY-104, 241-BY-105, 241-BY-107, 241-BY-108, 241-BY-109, 241-BY-110, 241-BY-112, 241-T-108, 241-T-109, 241-TX-104, and 241-TX-113. (BiPO <sub>4</sub> Saltcake waste)	60°C, 80°C, and 100°C	caustic	PNNL-17992 (WTP-RPT-166)
SSTs	241-C-103, 241-B-109, 241-C-105, 241-B-108, 241-C-104, and 241-BY-109. (PUREX Cladding waste sludge)	60°C, 80°C, and 100°C	caustic	PNNL-18054 (WTP-RPT-167)
SSTs	241-U-105, 241-U-201, 241-U-202, 241-U-203, and 241-U-204. (REDOX Cladding waste sludge)	60°C, 80°C, and 100°C	caustic	PNNL-18054 (WTP-RPT-167)
SSTs	241-S-101, 241-S-110, 241-S-107, and 241-SX-103. (REDOX Sludge waste)	80°C, 90°C, and 100°C	caustic	PNNL-17368 (WTP-RPT-157)
SSTs	241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106. (S-Saltcake waste)	100°C	caustic	PNNL-17368 (WTP-RPT-157)
SSTs	241-BX-109 and 241-B-106. (TBP waste)	40°C, 60°C, and 80°C	caustic	PNNL-18119 (WTP-RPT-169)
SSTs	241-BY-110, 241-BY-108, 241-BY-105, 241-BY-104, and 241-BY-106. (FeCN Tank Sludge)	60°C	caustic	PNNL-18120 (WTP-RPT-170)



**Table 2-2. DSTs and SSTs with Available Laboratory Scale Aluminum Leaching Data (4 sheets).**

SST/DST	Tank number <sup>1</sup>	Temperatures studied; °C	Caustic or Caustic and Oxidative leaching	Reference(s)
SSTs	241-S-101, 241-S-110, 241-S-107, 241-SX-103, 241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106. (REDOX Sludge and S-Saltcake waste mixture)	100°C	caustic	PNNL-18007 (WTP-RPT-171)
SSTs	241-S-101, 241-S-110, 241-S-107, 241-SX-103, 241-SX-106, 241-SY-103, 241-SX-105, 241-SX-102, 241-S-111, 241-U-108, 241-U-103, and 241-S-106. (REDOX Sludge and S-Saltcake waste mixture)	100°C	caustic	PNNL-17965 (WTP-RPT-172)
SSTs	241-C-103, 241-B-109, 241-C-105, 241-B-108, 241-C-104, 241-BY-109, 241-U-105, 241-U-201, 241-U-202, 241-U-203, and 241-U-204. (PUREX and REDOX Cladding waste sludge mixture)	100°C	caustic	PNNL-18048 (WTP-RPT-181)

As can be seen in Table 2-2, twenty-four of the SSTs, two of the DSTs, and four composite groups have had caustic leach tests performed only at 100°C, which is outside the currently acceptable temperature range for the PTF at the WTP. Also, of all of the caustic leach tests performed, only eight SSTs and three of the composite groups did not perform a wash step prior to caustic leaching (see Table A1-1). Of the remaining aluminum leaching analyses, three of the DSTs, twelve of the SSTs, and seven of the composite tank waste tests have been performed in a temperature range relevant to the current PTF plan, albeit with water washing performed (24590-WTP-RPT-PT-02-005, Rev. 5). Only the DST composite and three SSTs (241-C-104, 241-S-104, and 241-SX-113) were within the currently appropriate temperature range and had not had water washing performed prior to caustic leaching.

Washing of the solids prior to leaching has been performed in many ‘wash and leach’ tests up to several times prior to caustic leaching. This resulted in the rehydration of potentially dehydrated solids which had been in archives for sometimes over a decade, and was necessary in order to begin leaching at a state (e.g. hydroxide concentration) similar to the original tank waste sample in some cases. Secondary consequences of the wash step included (1) rehydration of solid species, (2) reduction in the overall ionic strength, (3) reduction in solid and/or aqueous concentrations of soluble constituents such as aluminum, chromium, silica, phosphates, and cesium, and (4) reduced potential of certain precipitate formation, including aluminosilicates as was described in ORNL/TM-1999/263. The washing step seemed to be a benefit in some instances, while not in others. This can be observed in Table A1-1 which indicates that some



washing steps resulted in large solids dissolution while others only had a small ( $\leq 1\%$ ) or no reduction in solids concentration.

Of the 12 SSTs with laboratory studies performed within the relevant temperature range, ten tanks were analyzed to determine the affects of different temperatures and caustic concentration on the extent of leaching, as detailed in Table 2-3. Seven of these provided information for the dissolution of aluminum over time, with data provided at individual sample times to show the progression of leaching. This information is provided in Table A3-1, with a plot of all of the percent aluminum leached versus time data depicted in Figure A3-1. More specific plots for individual tanks or temperatures are provided in Figure A3-2 through Figure A3-14.

**Table 2-3. SSTs Analyzing the Effects of T and [OH<sup>-</sup>] on Aluminum Leaching (2 sheets).**

SST	Temperature	Caustic concentration (NaOH)	Total time for Caustic leaching	% Al leached	Reference
241-B-101	60°C	1 M	168 hr	56	PNNL-12026
	60°C	3 M	168 hr	63	
	100°C	1 M	168 hr	59	
	100°C	3 M	168 hr	62	
241-BX-110	60°C	1 M	168 hr	95	PNNL-12026
	60°C	3 M	168 hr	99	
	80°C	1 M	168 hr	98	
	80°C	3 M	168 hr	97	
	95°C	1 M	168 hr	99	
	95°C	1 M	168 hr	99	
241-BX-112	60°C	1 M	168 hr	64	PNNL-12026
	60°C	3 M	168 hr	69	
	80°C	1 M	168 hr	56	
	80°C	3 M	168 hr	65	
	100°C	1 M	168 hr	53	
	100°C	3 M	168 hr	61	
241-C-102	60 °C	1 M	72 hr	27	PNNL-12026
	60°C	3 M	72 hr	95	
	100°C	1 M	72 hr	20	
	100°C	3 M	72 hr	95	
241-C-104	50°C	4 M	65 hr	39.7	ORNL TM-13500
	80°C	4 M	65 hr	28.7	
	93°C	4 M	65 hr	90.2	
241-S-101	70°C	1 M	168 hr	70	PNNL-12026
	70°C	3 M	168 hr	63	
	95°C	1 M	168 hr	88	
	95°C	3 M	168 hr	90	
241-S-101	70°C	1 M	168 hr	78.4	ORNL TM-13655
	70°C	3 M	168 hr	64.9	
	95°C	1 M	168 hr	89	
	95°C	3 M	168 hr	91.5	
241-S-104	67°C	3.8 M	24 hr	98.5	ORNL TM-

**Table 2-3. SSTs Analyzing the Effects of T and [OH<sup>-</sup>] on Aluminum Leaching (2 sheets).**

SST	Temperature	Caustic concentration (NaOH)	Total time for Caustic leaching	% Al leached	Reference
	70°C	3.99 M	21 hr	20.8	13500
	70°C	6.33 M	21 hr	26.6	
	80°C	6.33 M	126 hr	96.3	
241-S-110	60°C	1 M	168 hr	39	PNNL-13702
	60°C	3 M	168 hr	47	
	60°C	5 M	168 hr	50	
	80°C	1 M	168 hr	69	
	80°C	3 M	168 hr	91	
	80°C	5 M	168 hr	96	
	100°C	1 M	168 hr	91	
	100°C	3 M	168 hr	100	
241-SX-113	70°C	6.33 M	21 hr	51	ORNL TM-13500
	80°C	6.33 M	126 hr	78.7	
241-T-110	60°C	0.6 M	168 hr	27	PNNL-13956
	60°C	2.5 M	168 hr	42	
	60°C	4 M	168 hr	59	
	80°C	0.6 M	168 hr	53	
	80°C	2.5 M	168 hr	67	
	80°C	4 M	168 hr	83	
	100°C	0.6 M	168 hr	50	
	100°C	2.5 M	168 hr	75	
	100°C	4 M	168 hr	77	

The extent of aluminum removal due to inhibited water washing and/or caustic leaching varied considerably depending upon the temperature, caustic concentration, and leaching time. Other important parameters which affect the extent of aluminum dissolution include the solid to solution ratio, the aluminum mineralogy, initial aqueous and solid chemistry, and the degree of mixing. A kinetic model, which takes these factors into consideration, is currently being developed.

### 3.0 DISCUSSION

Due to the chemical variability within the 177 radioactive storage tanks, laboratory studies have been performed on samples to determine the extent of aluminum leaching possible through inhibited water washing and caustic leaching. In this report we have compiled the available information on aluminum removal from tank waste solids from a total of thirty-nine laboratory studies (see Section A1.0 of the appendix). This collection summarizes the results for aluminum removal and the conditions used for their analyses.

Within these thirty-nine laboratory reports, a total of 5 DSTs, 41 SSTs, and 12 composite tank waste samples were investigated for aluminum leachability. Of these, only 3 of the DSTs, 12 of the SSTs, and 7 of the composite tank studies were performed within the most recent acceptable operation temperature range of the WTP PTF. Seven of the SSTs were studied within the current acceptable temperature range, providing concentration versus time data for aluminum leaching. This information (shown in Figure A3-1 through Figure A3-14) can be used as an indication for how changes in temperature and caustic concentration could affect aluminum leaching on prewashed solids during caustic leaching of these waste samples.

An initial trend observed in Figure A3-2 through Figure A3-8 was that an increase in the caustic concentration in nearly all analyses increased the percent of aluminum removal. This is evident due to the need for free hydroxide for both gibbsite and boehmite dissolution, as shown in Equations 1.1 and 1.2. Both of these reactions will only proceed to a point where the saturation limit of aluminate is reached in solution. Therefore, the solid to solution ratio and aluminate concentration initially in solution are also important pieces of information, yet are not always provided during these analyses. Caustic is also necessary for the removal of other nonradioactive components that limit HLW loading, including chromium (Cr) and phosphate ( $\text{PO}_4$ ) (24590-WTP-RPT-PT-02-005, Rev. 5). Therefore, additional caustic will be necessary to ensure adequate concentrations for dissolution of not only aluminum, but also the other bulk soluble nonradioactive species during this step in the PTF.

Gibbsite has been found to be easily leachable in caustic due to its favorable kinetics, whereas boehmite dissolution is more rate limited, leading to slower dissolution [LBL-21482, *Thermochemical properties of gibbsite, bayerite, boehmite, diaspore, and the aluminate ion between 0 and 350°C*, "Dissolution/precipitation kinetics of boehmite and gibbsite: Application of a pH-relaxation technique to study near-equilibrium rates" (Benezeth et al. 2008), "Comprehensive Model of Synthetic Bayer Liquors. Part 3. Sodium Aluminate Solutions and the Solubility of Gibbsite and Boehmite" (Königsberger et al. 2006), "Studies on the Gibbsite to Boehmite Transition" (Ruff et al., 2008), and "Boehmite Actual Waste Dissolution Studies" (Snow et al., 2008)]. Aluminosilicate solids, as previously described, are more 'refractory,' meaning that they have less of a tendency to dissolve with the addition of caustic and will remain as solids in most cases. These three mineral phases account for the majority of the aluminum species observed within these tanks, leading to a reinforcement of the theory that the majority of the aluminum can be grouped into three categories: (1) easily leachable (e.g. gibbsite, dawsonite, etc.), (2) slow leaching/kinetically controlled (e.g. boehmite), and (3) non-leachable/ refractory (e.g. aluminosilicates and cancrinites).

It is evident from this compilation, there is a lack of information concerning the effects of unwashed, caustic leached actual waste samples within the acceptable temperature range of 80°C to 90°C (as detailed in 24590-WTP-RPT-PT-02-005, Rev. 5). From the 46 individual SSTs and DSTs with laboratory data available, only three analyses were performed with no inhibited water wash prior to caustic leaching between 80°C and 90°C. These were for SSTs 241-C-104, 241-S-104, and 241-SX-113 (all documented in ORNL TM-13500). Of the remaining composite studies, only the 241-AZ-101 + 241-AZ-102 analysis described in PNNL-11580 was performed without water washing and leached in the temperature range of 80-90°C. All of the remaining

laboratory studies were either performed by washing the solids with inhibited water prior to caustic leaching, or performing caustic leaching at a temperature outside the current acceptable range.

Preliminary washing was found to remove soluble aluminum species from the solids prior to caustic leaching, and in some cases (e.g. that for tanks 241-AN-104 and 241-BY-110) dissolved over 90 percent of the solid aluminum present in the sample. Then again, preliminary washing sometimes had little to no effect on the solids concentration or even increased the amount of aluminum in solid form. Even though this process will not be used at the PTF, it does provide an indication of the fraction of solid aluminum which is in an easily leachable form for these tanks. It does not indicate, however, the extent of inhibition that will occur due to aluminum saturation limits.

The removal of easily leachable aluminum during inhibited water washing reduces the solid aluminum concentration by transferring it to the aqueous phase. After washing in the referenced laboratory analyses, this aqueous fraction is typically removed by centrifugation and decanting. The aluminate ion in solution is therefore removed, allowing further dissolution of aluminum from the remaining solids. If the aluminate ion concentration in solution were not removed, the initial aqueous aluminate would have to be considered during caustic leaching. This is due to its influence on the reactions shown in Equations 1.1 and 1.2, and its inhibitory effect when the aluminate ion concentration reaches its saturation point. The initial aluminate ion concentration in solution will reduce the total amount of solid aluminum which could be dissolved. Measurements of both the aqueous and solid phase soluble aluminum are therefore key aspects in determining how much aluminum can be removed through high temperature caustic leaching at the WTP PTF prior to vitrification.

Results of aluminum leaching varied between the thirty-nine laboratory studies. In a majority, increases in the caustic concentration in solution, leaching time, and leaching temperature resulted in higher removal of aluminum from the solid phase. Also, the major aluminum solid species found within the wastes initially or following inhibited water washing and/or caustic leaching were gibbsite, boehmite, and aluminosilicates/cancrinites. Gibbsite was found to be easily soluble, being removed from the solid phase in many cases, whereas boehmite and aluminosilicates/cancrinites were found to remain in the solid phase after caustic leaching. These three solid aluminum phases will be used in further analyses to determine the potential extent of aluminum removal due to caustic leaching.

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APPENDIX A.  
**INFORMATION FROM LABORATORY STUDIES**

## **A1.0 SINGLE- AND DOUBLE-SHELL TANKS WITH LABORATORY WASH AND LEACH INFORMATION**

Pertinent information gathered from laboratory reports have been compiled here to provide all of the known data concerning the effects of washing and caustic leaching on the removal of aluminum from actual waste solids. This report analyzes 39 of these reports. This information is detailed in Table A1-1 and Table A1-2 below. These two tables are meant to be placed side-by-side to provide pertinent information gathered from the individual reports.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
<b>DSTs</b>													
241-AN-104	PNNL-11636	FY97	Caustic leaching.	—	Primarily "salt cake", characterized as Double-shell slurry feed. Double-shell Slurry Feed. <sup>1,2</sup>	222-S Lab ID S96T005974; Jar # 12039; Core # 164	Wash 3 times by adding 10 ml 0.01 M NaOH to 4.702 g slurry; mix 1 hr at 100°C; cool; centrifuge; decant; repeat w/ solids portion.	791 ug/mL	30.4 ug/mL	951 ug/mL	99%	Component amounts in washed and untreated solids normalized to amount of Fe present	Add 4.5 ml of 10 M NaOH to 21.485 g slurry; mix 5 hr at 100°C; cool; centrifuge; decant.
241-AZ-101	PNWD-3206 (WTP-RPT-043)	2000	Crossflow filtration, water washing, and caustic leaching	Material was very cohesive/adhesive in nature (pg. 2.8)	—	See Table 2.1 of WTP-RPT-048, <i>Chemical Analysis and Physical Property Testing of 241-AZ-101 Tank Waste – Supernatant and Centrifuged Solids.</i>	Following initial filtration of 4312.5 g composite sample (7.6 wt% undissolved solids), performed two washes using batch additions of 1L 0.01M NaOH at 25±5°C and removing permeate by filtration.	95000 ug Al/g slurry or 5325 ug Al/mL supernate.	186000 ug Al/g slurry	9% removed in wash. Wash 1 permeate: 2350 ug Al/mL. Wash 2 permeate: 1210 ug Al/mL.	—	Physical properties (i.e. density and particle size) and rheological data available in report.	Combine NaOH with washed slurry to ~3M OH <sup>-</sup> by adding NaOH (1120 g of 3M NaOH and 645 g 9M NaOH added), heat to 85°C, and mix for 8 hr. Dewater by filtration at 25°C.
241-AZ-101 + 241-AZ-102	PNNL-11580	FY96	Lab scale caustic leaching: Six leaches performed at 80°C in series, each 5 hours long.	—	Aging <sup>1</sup>	—	—	6 wt% Al	—	—	—	Combined 24 g (16.1 mL) 'reconstituted' AZ-102 with 9.55 g (5.9 mL) AZ-101. Add 67.8 g water, mixed and remove 2.924 g slurry, then performed 6 caustic leaches and 3 wash steps.  'Reconstituted' AZ-102 sludge: add equal weight water to dried sludge.	Caustic Leach #1: Add 0.627 ml 12M NaOH (0.23M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, remove 15.15 ml supernate sample. Caustic Leach #2: Add 10.5 ml 12M NaOH to solids (1.19M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, remove 14.4 ml supernate sample. Caustic Leach #3: Add 115.65 ml water & 8.63 ml 12M NaOH to solids (1.04M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, decant, remove 14.27 ml supernate sample. Caustic Leach #4: Add 17.6 ml 12M NaOH to solids (2.14M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, decant, remove 12.98 ml supernate sample. Caustic Leach #5: Add 16 ml 12M NaOH to solids (2.92M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, decant, remove 157.4 ml supernate sample. Caustic Leach #6: Add 159.8 ml 3M NaOH (3.27M free OH <sup>-</sup> ), mix 5 hr at 80°C, cool, settle, decant, remove 148.1 ml supernate sample.

<sup>2</sup> Percent recovery, in most cases, is a comparison of measured concentrations using two methods: (1) summation of the individual solid and liquid samples following the laboratory study and (2) a direct analysis of the total concentration in the initial sample.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-AZ-102	75764-PCS95-086	1994	Sludge water washing/ caustic washing	—	—	—	<p>Sludge sample (labeled 102-AZ-4) was agitated until homogeneous and allowed to settle 24 hrs. The sludge was agitated again, with 25 mL aliquots transferred to 4 centrifuge cones, with weights and volumes recorded. After centrifuging 3 hrs, the supernatant was decanted. Water or caustic solution (1, 2, or 3 M) was added to each cone to perform a wash of the solids to remove soluble species. Cones were tumbled for 24 hrs at ambient T and subsequently centrifuged for 1 hr. The water wash and 1 M Caustic wash cones broke during centrifuging, losing all supernate and some solids.</p>	<p>65100 ug Al/g sludge.</p> <p>Cone 1 (water wash): 35.33 g sludge; 24.61 g solids + 10.72 g supernate + 32.9 g water.</p> <p>Cone 2 (1M NaOH wash): 31.92 g sludge; 22.41 g solids + 9.51 g supernate + 35.5 g NaOH.</p> <p>Cone 3 (2M NaOH wash): 37.19 g sludge; 25.97 g solids + 11.22 g supernate + 35.39 g NaOH.</p> <p>Cone 4 (3M NaOH wash): 34.66 g sludge; 24.37 g solids + 10.29 g supernate + 35.85 g NaOH.</p>	<p>Cone 1: 6.468 g Al/100 g solids.</p> <p>Cone 2: 5.597 g Al/100 g solids.</p> <p>Cone 3: 2.007 (4.057<sup>3</sup>) g Al/100 g solids.</p> <p>Cone 4: 3.803 g Al/100 g solids.</p>	<p>Al in washed supernate: Cone 1: 0.018 g/100 g sludge. Cone 2: 0.095 g/100 g sludge. Cone 3: 0.131 g/100 g sludge. Cone 4: 0.155 g/100 g sludge.</p> <p>Al in interstitial liquid: Cone 1: 0.005 g/100 g sludge. Cone 2: 0.025 g/100 g sludge. Cone 3: 0.042 g/100 g sludge. Cone 4: 0.049 g/100 g sludge.</p>	—	—	—

<sup>3</sup> Aluminum values were believed to be erroneous for this data set, even though a specific analytical error was not determined. The values reported in parentheses are based on a factor-of-two correction to reported values, which was more believable to the authors.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-AZ-102	PNWD-3045 (BNFL-RPT-038)	1999-2000	Crossflow filtration, water-washing, and caustic-leaching	—	—	Four jars - sample #17165 = 153.53g; #17166 = 134.15g; #17167 = 128.85g; #17168 = 145.32g. Total of 538.9 g composite sample mixed for 29.42 hr and allowed to settle 90 hr. Material was very cohesive/adhesive, being hard to transfer and leaving a thick clinging layer on tools and vessels. Also formed strong agglomerations: easy to break apart when allowed to sit for a few days, difficult when allowed to sit for weeks. Material also entrained air after filtration in quantities that increased the sample volume by a third.	Following initial filtration of sample performed three washes using 0.01M NaOH at 25°C. Washing solution added, followed by removal of an equal amount of liquid as permeate using the crossflow filter.	1.25E+5 ug Al/g dry solids	—	Wash 1: 146 ug Al/mL. Wash 2: 108 ug Al/mL. Wash 3: 93 ug Al/mL.	Following Wash: 1.85E+5 ug Al/g dry solids. Wash efficiency = 2.5%	Physical properties (i.e. density and particle size) and rheological data available in report.	Combine NaOH with washed slurry to ~3M OH <sup>-</sup> by adding 609.16 g of 7.37M NaOH, heat to 85°C, and mix for 8 hr. Dewater by filtration.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-SY-102	PNWD-3512 (WTP-RPT-117)	Sample shipped to PNWD in July 2003	Lab scale caustic and oxidative leaching	—	DN/PD. <sup>1</sup>	Mixture of solids and liquids from two cores (284 & 286), 11 jars, and 11 sample IDs (see end of row for details)	Combine all samples in single 2L jar; mix, transfer portion into separate 50 mL centrifugation cone. Wash 2 times w/ 0.01 M NaOH so total volume = 50 mL. Centrifuge and separate solid/liquid. Transfer solid to 50 mL centrifuge bottles and contact approximately 5 times (until bulk of color removed) with fresh 0.01 M NaOH w/ solution/solid ratio of approximately 5:1. Decant supernate after each contact and discard. Prepare final slurry by adding a portion of 0.01 M NaOH to washed, centrifuged solids. Remove two weighed aliquots of stirred suspension and dry to constant wt at 105°C. Repeat above procedure to obtain enough solid for analysis.	—	171000 ug Al/g dried solids	—	—	—	Use samples SY-102-3 and SY-102-4 with slurry weights of 15.813 and 15.767 g, respectively. Add 4.8 ml 10M NaOH and 8.2 ml DI to each. Heat at 85 ± 5°C for 8 hr. Cool to RT overnight. Centrifuge and decant supernate to 30 mL plastic vials. Add 0.1M NaOH at 3:1 vol:vol ratio to remaining solids. Mix, separate, and decant to same container as initial leachate solution. Continue washings until supernate was colorless. Filter samples (0.2 um syringe filter), and place 1 ml aliquot in container w/ 10 mL of 1M HNO <sub>3</sub> for ICP-AES.
241-SY-103	PNL-10712	FY95	Lab scale caustic leaching	—	CC. <sup>1,2</sup>	Composite of segments 10 through 14 from Core 62 (222-S Jar #6590)	Add 40.2 g water to 10.2 g SY103-1, mix, and sample. Use 1.11 g slurry: Wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to slurry; mix 0.5 hr at RT; centrifuge; decant; repeat w/ solids portion. Dry remaining solids at 80°C (0.016 g solid remains)	—	—	—	—	Retrieval Wash prior to caustic leach: add 0.43 g 1M NaOH/1M NaNO <sub>2</sub> to 6.05 g dry solids in slurry. Mix 1h at 100°C, cool, measure settling, centrifuge, decant.	Add 1.3 g water and 1.6mL of 10 M NaOH to 4mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant
241-SY-103	PNNL--11089	FY95	Sludge washing and caustic leaching	—	Primarily CC.	Core # 62	Add water and inhibited water to sludge sample to get ~2.3 wt% solids and 0.01 M NaOH/NaNO <sub>2</sub> . Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	9% recovery of Al	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 2.2M. Mix at 100°C for 5 hr. Cool, centrifuge, decant.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
SSTs													
241-B-101	PNNL-12026	FY98	Lab scale caustic leaching: compare 60 and 100°C caustic leaching over 168 hr.	—	Primary: EB; Secondary: CW.  Non-Complexed Waste. <sup>1</sup>  Primary: CW. Secondary: EB. Tertiary: B Plant low-level waste. <sup>5</sup>	Composite mixture of two segments from Core #90 and two segments from Core #91	Mix 50.15 g B-101 composite with 100 mL 0.1 M NaOH in 225 mL bottle; stir 30 min at RT; centrifuge at 1200 G for 15 min; decant. Repeat wash with NaOH for 7 total wash cycles.	3.04 wt% Al; 30378 ug Al/g solids	82615 ug Al/g washed solids; 1148348 ug Al	473 ug Al/mL or 375089 ug Al	25% Al removed	Shows time versus concentration graph for leaching at different temperature and NaOH concentrations. Diluted washed solids w/ 50 mL DI & stir 30 min. Distribute ~15 g aliquots between 5 bottles	Add appropriate amount of 10 M NaOH and water to each vessel to provide desired NaOH concentration and 5 mL leachate per gram as-received sludge. (1 mol OH <sup>-</sup> per mol Al or Cr & 2 mol OH <sup>-</sup> per mol P). Bottle 1: 1.1 M NaOH at 60°C. Bottle 2: 1.0 M NaOH at 100°C. Bottle 3: 3.2 M NaOH at 60°C. Bottle 4: 3.1 M NaOH at 100°C. Mix slurry 5 min; settle 5 min; check free OH <sup>-</sup> w/ 100 uL sample & adjust if not within 0.2 M target; heat at appropriate temperature & stir (add DI to make up for liquid loss if necessary); sample leachate at 5, 24, 72, & 168 hr (at sample times, stop stir and settle 30 min) by filtering ~2.5 mL; acidify 2 mL aliquot w/ 1.5 mL concentrated HNO <sub>3</sub> & 16 mL DI; replace unused portion back to reaction vessel and continue leaching.
241-B-104	LAUR 96-2839	FY96	Lab scale caustic leaching	reddish-brown thin paste	2C; slurry product from evaporators (EB); TBP waste from U-extraction process at U Plant; 1C.  Non-Complexed Waste. <sup>1</sup>  Primary: 2C, Secondary: EB. <sup>2</sup>  Primary: 2C. Secondary: EB. Tertiary: TBP. Other: 1C. <sup>5</sup>	—	Mix 13.54 g wet sludge with 27 mL water. With 4.8 g slurry, wash 3 times by adding 10mL of 0.01 M NaOH; mix for 1 hr at 100°C; cool; centrifuge; decant; repeat wash with solids. Dry solids at 105°C. Produced 0.148 g or 9.45 wt % insoluble solids	0.3 wt% Al; 2118.03 ug Al; 14349.8 ug Al/g solids	94% Al	140.67 ug Al; 4.91 ug Al/mL	6% Al removed by simple wash	—	Add 10M NaOH and water to 10.97 g mixed wet sludge (5.76 g dry sludge) to get ~5 wt% solids and final NaOH of ~2M (4 mL of 10 M NaOH); mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-B-106	LAUR 97-2889	FY97	Lab scale caustic leaching	reddish-brown liquid paste; dried material: tannish-brown in color; dry treated material: dark brown in color	1C, TBP from U-extraction process at U Plant, lab waste from the 300 area. Non-Complexed Waste <sup>1</sup> . <u>Primary:</u> 1C, <u>Secondary:</u> TBP. <sup>2</sup> <u>Primary:</u> 1C. <u>Secondary:</u> TBP. <u>Tertiary:</u> Hanford Laboratory Operations. <u>Other:</u> mixture of several miscellaneous wastes. <sup>5</sup>	Composite of sludge from segments 1 & 2 from cores 93 and 94 (222-S lab ID S96T001862, jar #9794)	Mix 11.27 g wet sludge with 22.5 mL water. With 4.47 g slurry, wash 3 times by adding 10mL of 0.01 M NaOH; mix for 1 hr at 100°C; cool; centrifuge; decant; repeat with solids. Dry solids at 105°C. (8.55 wt % insoluble solids, 33.1 mL supernate)	0.7 wt% Al	23858 ug Al/g, 3061 ug Al (72% Al remaining)	35.7 ug Al/ml, 1181.67 ug Al (28% Al removed)	28% removed (4242.59 ug Al)	—	Mix 11.27 g wet sludge w/ 22.5 mL water. Subsample to get 8.8 g wet sludge (3.53 g dry sludge). Add 7.23 mL of 10 M NaOH and water (2.1 wt% solids and ~2M NaOH). Mix 5 hr at 100°C; cool; measure settling; centrifuge; decant. Measured 26.08 mL supernate w/ 2.33 M free OH <sup>-</sup> , 9.5 mL settled solids, and 4.1 mL centrifuged solids.
241-B-110	PNL-9387	FY93	Lab scale caustic and acid leaching	—	2C in 1940s to recover & purify Pu from irradiated fuel, adjusted to high pH; fission product waste produced in campaigns ('60s) to isolate fission products; IX from B Plant Cs recovery process. Non-Complexed Waste <sup>1</sup> . <u>Primary waste:</u> 2C. <u>Secondary:</u> High-level B Plant waste from bottom of Section 5. <sup>2</sup> <u>Primary waste type:</u> 2C. <u>Secondary:</u> high-level B Plant waste. <u>Tertiary:</u> Fission Products Waste. <u>Other:</u> IX. <sup>5</sup>	A composite of the 5 segments obtained in the 1989 Core 1. C1 Composite (90-4316)	Mix 5.15 g C1 composite with 14.5 mL of 0.1M NaOH. With 2.2 g slurry, stir 1 hr at 100°C; centrifuge; decant. Add 2 mL of 0.1M NaOH to solids; mix 1 hr at 100°C; centrifuge; decant; add 5 mL water to solids; stir 0.5 hr at RT; centrifuge; decant; add 1 mL water to solids; stir 0.5 hr at RT; save 0.13 g slurry for analysis	1.5E-3 to 7E-4 g Al/g sludge	Less than or equal to 27% Al remaining	—	—	Sludge washing and acid dissolution experiment done as well - not included here.	Add 0.1 mL water and 0.3 mL of 10 M NaOH to slurry (Effectively 2.3M NaOH); mix 5 hr at 100°C; centrifuge; decant. Add 1 mL 3M NaOH to solids; mix 5 hr at 100°C; centrifuge; decant. Add 5 mL water to solids; mix 0.5 hr at RT; centrifuge; decant. Add 1 mL water to solids; mix 0.5 hr at RT; take 0.05 g slurry subsample; centrifuge; decant. (8.1 mL sludge leach solution)
241-B-110	Internal Letter 9404238	1989	Lab scale wash, caustic leach, and acid dissolution	—	<u>Primary waste:</u> 2C. <u>Other waste:</u> fission product waste produced in campaigns (in the 1960s) to isolate various fission products, and IX from B Plant Cs recovery process.	A 9.34 gram wet composite of cores 1, 2, 3, and 4 from the 1989 sampling event.	Mix 9.34 g wet sludge composite with three times its weight in water for 1 hr at RT using an orbital shaker. Allow the sludge to settle ~20 hr after mixing. Decant wash liquor, replace with fresh water, and repeat the process twice.	Between 2.1E-3 and 2.2E-3g Al/g sludge	≥94% remaining	≤6% removed	—	Acid dissolution of the solids was performed after caustic leaching and final wash of solids. This is not included in this table.	Add 8 mL water and 9 mL of 10M NaOH to solids. Mix, using a magnetic stirrer, for 5 hr at 100°C. Cool, then settle and decant liquid.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-B-111	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> 2C. <u>Secondary:</u> High-level B Plant waste from bottom of Section 5. <sup>2</sup> <u>Primary:</u> 2C. <u>Secondary:</u> high-level B Plant waste. <u>Tertiary:</u> Fission Products Waste. <u>Other:</u> IX. <sup>5</sup>	Composite sample comprised of 2.65 g Core 29 composite 1 [Analytical Lab Operations #93-04314], 1.96 g Core 29 composite 2 [ALO #93-04315], 2.64 g Core 30 composite 1 [ALO # 93-04322], and 2.57 g Core 30 composite 2 [ALO #93-04323]	Add 39.1 g water to 9.67 g B111-1, mix, and then sample. Wash 3 times by adding 5 ml of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 1.05 g slurry; mix 0.5 hr at RT; centrifuge; decant; repeat w/ solids portion. Dry remaining solids at 80°C (0.021 g solid remains)	—	—	—	—	<u>Retrieval Wash prior to caustic leach:</u> Add 2.14 g water and 0.39 g 1M NaOH/1M NaNO <sub>2</sub> to 4.03 g dry solids in slurry. Mix 1h at 100°C, cool, measure settling, centrifuge, decant.	Add 0.4 g water and 2.8 g 10 M NaOH to 4 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant
241-B-111	PNNL--11089	FY95	Sludge washing and caustic leaching	—	<u>Primary:</u> 2C. <u>Secondary:</u> HLW from Tank 5-6 at B Plant.	Core # 29 and 30	Add water and inhibited water to sludge sample to get ~2.3 wt% solids and 0.01M NaOH/NaNO <sub>2</sub> in solution. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	—	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 2.3M. Mix at 100°C for 5 hr. Cool, centrifuge, decant.
241-B-201	PNL-10078	FY94	Caustic leaching	—	<u>Primary waste:</u> Lanthanum fluoride decontamination waste. <sup>2,5</sup>	Two composite samples which represented 2 core samples (26 and 27). Two dry composite analyses from cores 26 and 27, and one wet core 26 composite (untreated)	<u>Dry cores:</u> Add 5 mL of 0.1 M NaOH & mix at 100°C for 1 hr, centrifuge, decant. Add 2 mL 0.1 M NaOH & mix at 100°C for 1 hr, centrifuge, decant. Add 5 mL water & mix at RT for 0.5 hr, centrifuge, decant; dry at 80°C. <u>Wet core 26:</u> same as dry cores, but after 3 <sup>rd</sup> wash do not dry.	—	—	<u>Core 26:</u> 0-1% Al dissolved <u>Core 27:</u> 0-2% Al dissolved	—	—	<u>Dry cores:</u> Add 6 mL of 3M NaOH to solids from wash, mix at 100°C for 5 hr, centrifuge, decant. Add 5 mL 3M NaOH to solids, mix at 100°C for 5 hr, centrifuge, decant. Add 5 mL water to solids, mix at RT for 0.5 hr, centrifuge, decant. <u>Wet core 26:</u> Add 1 mL 3 M NaOH to solids from wash, mix at 100°C for 5 hr, centrifuge, decant; repeat. Add 5 mL water to solids, mix at RT for 0.5 hr, centrifuge, decant.
241-B-202	LAUR 95-2070	1995	Lab scale caustic leaching	thick black paste	Final decontamination and concentration stage of BiPO <sub>4</sub> process. <u>Primary waste:</u> Lanthanum fluoride decontamination waste. <sup>2,5</sup>	—	Add 4.2 mL water to get ~2.3 wt% solids. Add NaOH/NaNO <sub>2</sub> to 0.01M (0.37 mL), mix at 100°C for 1 hr, cool, measure settling, centrifuge, decant (~22.3 mL settled solids/ ~10 mL centrifuged solids)	—	—	Less than 4.25 ug Al/mL (Less than 2.89% Al removed)	—	—	Add 2 mL of 10 M NaOH to get ~8 wt% solids and final NaOH of ~3 M, mix at 100°C for 5 hr, cool, measure settling, centrifuge, decant (Vol. settled solids ~9mL; Vol. centrifuged solids ~ 4.1 mL)

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-BX-103	LAUR 97-2889	FY97	Lab scale caustic leaching	Wet dark brown thick paste	TBP from uranium extraction process at U Plant, CW produced at PUREX plant from dissolution of Zr or Al fuel cladding, and OWW from PUREX. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> OWW. <u>Other:</u> mixture of several miscellaneous wastes. <sup>5</sup>	Composite of sludge from portions of segment 2, core 86 (222-S Lab ID S96T005277, jar # 11629)	Mix 10.05 g wet sludge with 20.1 mL water then subsample 4.82 g. Wash 3 times by adding 10 mL 0.01 M NaOH; mix 1 hr at 100°C; cool; centrifuge; decant. Obtain 32.94 mL supernate. Dry solids at 105°C to obtain 55.7 wt% insoluble solids.	21 wt% Al	244437.2 ug Al/g, 218991 ug Al (99% Al remaining)	62.055 ug Al/mL, 2044.09 ug Al (1% Al removed)	—	—	Mix 10.05 g wet sludge with 20.1 mL water and subsample (7.41 g wet sludge, 5.24 g dry sludge). Add 18.2 mL water & 14.8 mL 10 M NaOH to get ~5 wt% solids & ~2M NaOH; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant. Supernate: 66.52 mL w/ 2.21M free OH <sup>-</sup> (4.61 mL settled solids, 2.28 mL centrifuged solids)
241-BX-105	LAUR 95-2070	1995	Lab scale caustic leaching	Composite sludge - wet yellow-white material	First waste received was TBP from TBP uranium extraction process at U plant; PUREX plant dissolution of Zr or Al fuel cladding; IX waste from cesium recovery process at B plant; Last was EB consisting of a slurry product from evaporators. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> IX. <u>Other:</u> EB. <sup>5</sup>	—	Add 49.5 mL water to 12.381 g wet sludge. With 1.0971 g slurry, add NaOH/NaNO <sub>2</sub> to 0.01 M (5 mL), mix at RT for > 0.5 hr, centrifuge, decant, and repeat 2 more times. Dry solids at 80°C (0.1329 g solids; 60.5 wt% insoluble solids)	—	—	—	—	Detection limit = 4.25 ug Al/mL; 541.13 ug Al/g	<u>Retrieval Wash:</u> Add 158 mL water and 2 mL NaOH/NaNO <sub>2</sub> to 0.01M, mix 1 hr at 100°C, cool, measure settling, centrifuge, decant (Vol. settled solids ~ 12.5 mL, Vol. Centrifuged solids ~ 11.5 mL, 205.34 mL liquid from decant). <u>Caustic Leach 1:</u> Add 16.5 mL 10 M NaOH to ~3M solution and 25 mL water, mix 5 hr at 100°C, cool, measure settling, centrifuge, decant.  (Vol. settled solids ~ 4 mL, Vol. Centrifuged solids ~ 3.8 mL, 41.83 mL liquid from decant).
241-BX-107	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> 1C. <u>Secondary:</u> TBP. <sup>2</sup> <u>Primary:</u> 1C <u>Secondary:</u> TBP. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	Composite from core 41 (222-S Jar #J941)	Add 37g water to 10.01g B107-2, mix, and then sample. Wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 1.08 g slurry; mix 0.5 hr at RT; centrifuge; decant; repeat w/ solids portion. Dry remaining solids at 80°C (0.041 g solid remains)	—	—	—	—	<u>Retrieval Wash prior to caustic leach:</u> add 36 g water and 0.67 g 1M NaOH/1M NaNO <sub>2</sub> to 4.89 g dry solids in slurry. Mix 1hr at 100°C, cool, measure settling, centrifuge, decant.	Add 4.4 g water and 6.6 g of 10 M NaOH to 6.1 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-BX-107	PNNL-11089	FY95	Sludge washing and caustic leaching	—	Primarily 1C, with secondary waste from the TBP uranium extraction process at U Plant.	Core # 41	Add water to sludge sample to get ~2.3 wt% solids, as well as inhibited water (NaOH/NaNO <sub>2</sub> ) to 0.01 M. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	1% Al removed	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 2.0M. Mix at 100°C for 5 hr. Cool, centrifuge, decant.
241-BX-109	LAUR 96-2839	FY96	Lab scale caustic leaching	reddish-brown clay material	TBP from U-extraction process at U Plant; waste produced at PUREX Plant from dissolution of Zr or Al fuel cladding; 1C; IX waste from Cs recovery process at B Plant. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary:</u> TBP. <u>Secondary:</u> CW. <u>Tertiary:</u> 1C. <u>Other:</u> IX. <sup>5</sup>	—	Mix 7.23 g wet sludge with 14 mL water; with 4.62 g slurry, wash 3 times by adding 10 mL of 0.01 M NaOH; mix for 1 hr at 100°C; cool; centrifuge; decant; repeat with solids portion. Dry solids at 105°C to get 0.132 g dry / 8.58 wt % insoluble solids	0.1 wt % Al; 397.71 ug Al; 3017.6 ug Al/g	39% Al remaining	625.58 ug Al; 19.231 ug Al/mL	61% removed by simple wash	—	Add 10 M NaOH and water to 4.73 g mixed wet sludge (2.37 g dry sludge) to get ~5 wt% solids and final NaOH of ~2M; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.
241-BX-110	PNNL-12026	FY98	Lab scale caustic leaching: compare 60, 80, and 95°C caustic leaching for total of 168 hr.	—	<u>Primary:</u> 1C. <u>Secondary:</u> EB - In-tank solidification Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> 1C. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	A composite mixture of three segments from Core 198	Mix 75.86 g BX-110 composite with enough 0.1 M NaOH to reach 200 mL in 225 mL bottle; stir overnight at RT; centrifuge at 1200 G for 15 min; decant. Repeat wash with NaOH, stirring 30 min, for 4 total wash cycles.	3.39 wt% Al; 33863 ug Al/g	247500 ug Al/g; 2472773 ug Al	134 ug Al/mL; 96078 ug Al	4% Al removed	Shows time versus concentration graph for leaching at different T and NaOH concentrations. Dilute washed solids w/ 75 mL DI & stir. Distribute ~5 g aliquots between six 60 mL bottles, one 10 g aliquot in a 20 mL vial for analysis, and one 0.53 g aliquot taken from 10 g aliquot for microscopic analysis.	Add appropriate amount of 10M NaOH and water to each vessel to provide desired NaOH concentration and 10 mL leachate per gram as-received sludge. (1 mol OH <sup>-</sup> per mol Al or Cr & 3 mol OH <sup>-</sup> per mol P). Bottle 1: 1.2 M NaOH at 60°C. Bottle 2: 3.2 M NaOH at 60°C. Bottle 3: 1.2 M NaOH at 80°C. Bottle 4: 3.1 M NaOH at 80°C. Bottle 5: 1.2 M NaOH at 95°C. Bottle 6: 3.2 M NaOH at 95°C. Heat at appropriate temp & stir (add DI to make up for liq. loss if necessary); sample leachate at 5, 24, 72, & 168 hr (at appropriate sample times, stop stirring and settle for 30 min) by filtering ~2 mL; acidify 1 mL aliquot w/ 15 mL 0.3M HNO <sub>3</sub> ; replace unused portion back to reaction vessel and continue leaching. After final sampling, 2 aliquots were taken and titrated with standard HCl to determine free OH <sup>-</sup> concentration.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-BX-112	PNNL-12026	FY98	Lab scale caustic leaching; compare 60, 80, and 100°C caustic leaching for total of 168 hr.	—	<p><u>Primary:</u> 1C. <u>Secondary:</u> EB.</p> <p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary:</u> 1C. <u>Secondary:</u> EB. <u>Tertiary:</u> CW. <u>Other:</u> IX.<sup>5</sup></p>	A mixture of segment 3 from core 118 and segment 1 from core 119, with segment 3 providing most of the sample (actual proportions undocumented)	Mix ~100 mL centrifuged solids and ~100 mL liquid supernate in 200 mL bottle stirred 30 min; centrifuge 30 min at 1200 G; decant. Replace decanted liquid w/ 0.1 M NaOH to 200 mL mark; repeat for 7 total wash cycles.	3.8 wt% Al; 38000 ug Al/g; 3157800 ug Al (1.25 g Al/g Fe).	63000 ug Al/g; 2431800 ug Al (1.17 g Al/g Fe)	191 ug Al/mL; 140194 ug Al	5% Al removed; 81% Al mass recovery (6% Al removed)	Shows time versus concentration graph for leaching at different temp and NaOH concentrations. Dilute washed sludge to 200 mL w/ DI & stir. Distribute ~8 mL aliquots between seven 60 mL bottles & one 25 mL glass vial (dried at 105°C to obtain solids measurement)	Add appropriate amount of 10 M NaOH and water to each vessel to provide desired NaOH concentration and 15 mL leachate per gram untreated solids. (1 mol OH per mol Al or Cr & 3 mol OH per mol P). Bottles 1: 1.1 M NaOH at 60°C. Bottles 2: 2.9 M NaOH at 60°C. Bottles 3: 1.3 M NaOH at 80°C. Bottles 4: 3.4 M NaOH at 80°C. Bottles 5: 1.1 M NaOH at 100°C. Bottles 6: 3.4 M NaOH at 100°C. Bottles 7: 3 M NaOH and 5g Tiron <sup>4</sup> at 100°C. Heat at appropriate temp & stir (add DI to make up for liquid loss if necessary); sample leachate at 5, 24, 72, & 168 hr (at appropriate sample times, stop stirring and allow samples to settle) by filtering ~2 mL; acidify 1 mL aliquot w/ 15 mL 0.3M HNO <sub>3</sub> ; replace unused portion back to reaction vessel and continue leaching. After final sampling, 2 aliquots were taken & titrated with standard HCl to determine free OH concentration.
241-BY-104	PNNL-11278	FY96	Lab scale caustic leaching	—	<p><u>Primary:</u> TBP extraction process waste and FeCN-scavenged waste, <u>Secondary:</u> EB and In-tank solidification.</p> <p>Non-Complexed Waste.<sup>1</sup></p> <p><u>Primary:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification.<sup>2</sup></p> <p><u>Primary waste type:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> CW. <u>Other:</u> IX.<sup>5</sup></p>	Core 116: Core composite (222-S LABCORE sample #S96T000371)	Mix 7.588 g sample with 15.36 g water. With 4.83 g slurry add 10 mL 0.01 M NaOH. Mix at 100°C for 1 hr, cool, centrifuge, and decant. Repeat for a total of 3 washes. Dry solids after washes at 105°C. 1.622 g sludge initially in sample, 0.042 g washed solids, 2.7 wt% insoluble solids in sludge.	1.9 wt% Al	—	—	65% Al removed	An initial wash was performed on two sludge samples, with the solids and slurry mixed for use with caustic leach test. <u>Sample 1:</u> 25.27 g sludge - wash 3 times. <u>Sample 2:</u> 26.47 g sludge - wash 3 times. For wash, add 24 mL 0.01M NaOH, mix at RT for 0.5 hr, centrifuge, decant, and repeat.	After mixing washed solids and sludge from both wash samples, centrifuge and decant. With solids add 7.2 mL 10 M NaOH and water to a total volume of 22 mL (3 wt% solids), mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant.  19.9 mL decanted liquid, 1.4 mL interstitial liquid, 2.6 M OH.

<sup>4</sup> Tiron is also known as 4,5-dihydroxy-1,3-benzenedisulfonate.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-BY-108	PNNL-11636	FY97	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification. <sup>2</sup> <u>Primary:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB/in-tank solidification. <u>Tertiary:</u> 1C. <u>Other:</u> CW. <sup>3</sup>	222-S Lab ID S96T002035; Jar # 10527; composite of segment 4 from Core 99	Wash 3 times by adding 10 ml 0.01 M NaOH to 4.891 g slurry; mix 1 hr at 100°C; cool; centrifuge; decant; repeat w/ solids portion.	12600 ug Al/g	5993 ug Al (37% Al)	10055 ug Al (63% Al)	111% Al	—	Add 7.586 ml 10 M NaOH to 36.672 g slurry; mix 5 hr at 100°C; cool; gravity settle; decant
241-BY-110	PNNL-11278	FY96	Lab scale caustic leaching	—	<u>Primary:</u> TBP extraction process waste and FeCN-scavenged waste, <u>Secondary:</u> EB and In-tank solidification. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP and FeCN-scavenged, <u>Secondary:</u> EB and in-tank solidification. <sup>2</sup> <u>Primary:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> EB /in-tank solidification. <u>Tertiary:</u> 1C. <u>Other:</u> CW. <sup>3</sup>	Core 113: Core composite (222-S LABCORE sample #S96T000497)	Mix 8.802 g sample with 17.27 g water. With 4.85 g slurry add 10 mL 0.01 M NaOH. Mix at 100°C for 1 hr, cool, centrifuge, and decant. Repeat for a total of 3 washes. Dry solids after washes at 105°C. 1.639 g sludge initially in sample, 0.134 g in washed solids, and 8.2 wt% insoluble solids in sludge.	3.4 wt% Al; <u>Summation:</u> 4.28E+4 ug Al/g; <u>Direct:</u> 3.37E+4 ug Al/g; 127% Al recovery	21750 ug Al/mL; 2915 ug Al (6 wt% Al)	1407 ug Al/mL; 42351 ug Al (94 wt% Al)	94% Al removed; Total mass = 45265 ug Al	—	Wt. Sludge Solids in sample = 4.01 g. Add 4 mL 10 M NaOH and water to a total volume of 20 mL (2.3 wt% solids), mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant. 16.5 mL decanted liquid, 1.7 mL interstitial liquid, and 2.6 M OH.
241-C-102	PNNL-12026	FY98	Lab scale caustic leaching: compare 60 and 100°C caustic leaching at 1 or 3M NaOH	dry greenish-brown powder	<u>Primary:</u> CW. <u>Secondary:</u> waste from TBP extraction process. Dilute Complexed Waste. <sup>1</sup> <u>Primary:</u> CW. <u>Secondary:</u> TBP. <u>Tertiary:</u> OWW. <sup>3</sup>	Auger sample taken in August of 1995 (represents top portion of sludge).	Add 25.55 g composite sample to 50 mL 0.1M NaOH, stir 30 min at RT, remove 2 mL slurry, centrifuge 15 min at 1200 G, decant. Add 100 mL 0.1 M NaOH to solids, stir 30 min at RT, centrifuge 15 min at 1200 G, decant. Repeat for total of 3 washes. Wash solution: 224 mL (231.5 g solution. 1.0329 g/mL).	10.95 wt% Al; 109542 ug Al/g; 2711153 ug Al	189297 ug Al/g; 3180186 ug Al (#1=611429 ug Al; #2=670111 ug Al; #3=620894 ug Al; #4=569783 ug Al)	53.35 ug Al/mL; 11950 ug Al	0.4% Al removed; 118% Al mass recovery; 16.36 g Al/g Fe in washed solids = -10% Al removal	Estimated concentrations of Al, Cr, and P in as-received sludge from Agnew (1997): 93000 ug Al/g (4.8 g/g Fe), 92 ug Cr/g (0.01 g/g Fe), and 255 ug P/g sludge (0.0005 g/g Fe). Actual untreated concentrations: 14.9 g Al/g Fe, 0.6 g P/g Fe, and 1.5 g Si/g Fe. Percent weight reduction after 72 hr leach: #1=17%; #2=17%; #3=58%; #4=58%.	Four bottles were leached with varying conditions: #1 - 1.1M NaOH, 60°C, 17.7 wt% solids, 18 mL solution volume; #2 - 1M NaOH, 100°C, 24.8 wt% solids, 13 mL solution volume; #3 - 2.9M NaOH, 60°C, 9.5 wt% solids, 40 mL solution volume; and #4 - 2.9M NaOH, 100°C, 15.4 wt% solids, 19 mL solution volume. Each slurry was mixed 5 min, settled 5 min, adjusted for OH, heat & mix, sample at 5 hr & 72 hr (stop stir and settle 30 min prior to sampling), filter ~2.5 mL leachate, acidify 2 mL filtered solution w/ 1.5 mL 16M HNO <sub>3</sub> and dilute w/ 16.5 mL DI, add remaining filtered solution back to reaction vessel and continue leaching.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-103	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> Sr leached sludge. <u>Secondary:</u> Particulates from Sr wash of PUREX wastes in the AR vault. <sup>2</sup> <u>Primary:</u> Sr sludge. <u>Secondary:</u> particulates from Sr wash of PUREX wastes in the AR-vault. <u>Tertiary:</u> neutralized acid waste. <u>Other:</u> TBP/CW. <sup>5</sup>	Composite sample of segments 2-4 from core 63 (222-S Jar #6770).	Wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 0.21 g sludge/0.82 g water; mix 0.5 hr at RT; centrifuge; decant; repeat w/ solids portion. Dry remaining solids at 80°C (0.027 g solid remains).	—	—	—	—	Retrieval Wash prior to caustic leach: add 23.3 g water and 0.86 g 1M NaOH/1M NaNO <sub>2</sub> to 6.3 g dry solids in slurry. Mix 1hr at 100°C, cool, measure settling, centrifuge, decant.	Add 2.6 g water and 4.4 g 10 M NaOH to 6.7mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.
241-C-103	PNNL-11089	FY95	Sludge washing and caustic leaching	—	Primarily sludge feed for the Sr extraction process at B Plant, with secondary waste from particulates from the Sr wash of PUREX wastes in AR Vault.	Core # 63	Add water to sludge sample to get ~2.3 wt% solids, as well as inhibited water (NaOH/NaNO <sub>2</sub> ) to 0.01 M. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	—	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 0.6M. Mix at 100°C for 5 hr. Cool, centrifuge, decant.
241-C-103	ORNL/TM-13655	1998	Lab scale caustic leaching	—	Group XX waste based on the Sort on Radioactive Waste Type (SORWT) model, with the only other tank in this group being C-106. Primary waste is Sr-leached sludge; next largest fraction of HLW is suspended particulates from strontium washes of the PUREX wastes. Also has a separate layer of organic liquid. Non-Complexed Waste. <sup>1</sup>	WHC 222-S lab # S97M000283, jar # 13633	Wash 4 times by adding 120.95 g inhibited water (0.01M NaOH/NaNO <sub>2</sub> ) to 83.19 g sludge, wash at RT for 143 hr, centrifuge 20 min at 4500 rotations per minute (rpm), decant. Add 116.78 g inhibited water to solids, mix 22 hr at RT, centrifuge, decant. Add 126.7 g inhibited water to solids, mix 29 hr at RT, centrifuge, decant. Add 113.11 g inhibited water, mix 2 hr at 97°C, centrifuge, decant.	From Table 2.1: 4.56E+3 kg Al/3.14E+5 kg total sludge mass	—	—	—	No measurements of Al in wash solutions. Only measured the following: Cs-137 activity and conductance of wash solutions and wet weights of centrifuged solids.	No caustic leach performed.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-104	LAUR 97-2889	FY97	Lab scale caustic leaching	reddish-brown; dried material was dark brown	CW produced at PUREX Plant from dissolution of Zr or Al fuel cladding. OWW from PUREX, Sr wash of PUREX waste in AR vault, sludge feed for Sr extraction process at B Plant.  CC. <sup>1</sup>  <u>Primary:</u> CW. <u>Secondary:</u> OWW from PUREX. <sup>2</sup>	Composite of sludge made from portions of segments 1, 2, & 3 from core 165 (222-S lab ID S96T005188, jar # 11620).	Mix 11.76 g wet sludge with 23.5 mL water; with 4.7 g of this slurry. Wash 3 times by adding 10mL 0.01 M NaOH; mix for 1hr at 100°C; cool; centrifuge; decant; repeat with solids. Dry solids at 105°C to obtain 28.13 wt % insoluble solids & 32.68 mL supernate.	6.3 wt% Al	92793 ug Al/g, 41247 ug Al (91% Al)	129.15 ug Al/mL, 4220.62 ug Al (9% Al)	—	—	Mix 11.76 g wet sludge w/ 23.5 mL water; subsample to obtain 9.17 g wet sludge (4.22 g dry sludge). Add 9.8 mL water & 9.72 mL 10M NaOH to get ~5 wt% solids & ~2M NaOH; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant. Supernate: 36.44 mL w/ 2.72 M free OH, 12.25 mL settled solids, and 5.58 mL centrifuged solids.
241-C-104	PNWD-3027 (BNFL-RPT-021)	1999	Lab scale caustic leaching	—	CC. <sup>1</sup>	Sample received in 14 glass jars and composited as per test plan BNFL-29953-031. Composite was mixed for 1hr 20 min at 34°C prior to sub-sampling. Composite sample made of the following (sample #/weight in grams): 16273/150.046; 16274/157.638; 16275/176.435; 16276/157.212; 16277/162.65; 16278/164.872; 16279/149.645; 16280/141.802; 16281/142.608; 16282/160.345; 16283/159.172; 16284/160.251; 16285/147.301; and 16286/151.652.	Filter sample through 0.45 um nylon filter membrane. Transfer filtered solids to 125 mL HDPE bottle, with residual solids transferred using 0.01 M NaOH. Fill bottle to capacity with 0.01 M NaOH. Heat and stir at 85°C for 16.5 hr, filter with 0.45 um nylon filtration unit. Transfer solids back to bottle using 0.01 M NaOH, filling to capacity (123 g slurry). Heat and stir for 22.5 hr, then filter. Repeat transfer and heat for 24 hr, and then filter. Analyze composite sample of three wash solutions.	49461 ug Al (??? Read from Table 8???)	Mean value from KOH fusion = 146000 ug Al/g dry solid; Mean value from Na <sub>2</sub> O <sub>2</sub> Fusion = 157500 ug Al/g dry solid. Total weight = 2178963 ug Al.	126 ug Al/g, 36901 ug Al	2% Al removed	Begin wash with 50.8765 g aliquot (44.8 g as-received C-104 sample). After 1 <sup>st</sup> wash, had 100.13 g filtrate and 41.64 g solids. After 2 <sup>nd</sup> wash, had 82.79 g washing solution and 40.49 g wet solids. Third wash produced 93.11 g washing solution and 48.55 g wet solids. After evaporation of water at 80°C, dried overnight at 105°C (after 3 <sup>rd</sup> wash), had 14.3589 g dry washed solids.	Filter 45.8422 g (40.4 g as-received) sample through a 0.45 um nylon filter membrane. Transfer filtered solids to HDPE bottle using 3M NaOH, filling bottle to capacity yielding ~140 g slurry. Heat & stir for 21.5 hr at 85°C. Filter hot using a 0.45 um nylon filtration unit. (98.84 g filtrate; 41.47 g wet solids).

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-104	PNWD-3024 (BNFL-RPT-030)	1999	Filtration, water-washing, and caustic-leaching tests	—	Primary: CW. Secondary: OWW. Tertiary: particulates from Sr wash of PUREX wastes in the AR-vault. Other: Sr/mixed waste. <sup>5</sup>	14 glass jars rinsed with methanol and baked at 102°C for 12 hr. Mix for 1 hr 20 min and then subsample. Hot-cell temp during mixing was 34°C. Sample #/wt (g): 16273/150.46g, 16274/157.638g, 16275/176.435g, 16276/157.212g, 16277/162.65g, 16278/164.872g, 16279/149.645g, 16280/141.802g, 16281/142.608g, 16282/160.345g, 16283/159.172g, 16284/160.251g, 16285/147.301g, 16286/151.652g. Total=2181.629 g 'as-received' sample.	After initial filtration, wash at 25°C (add 715g 0.01M NaOH 7 times) and filter, collecting three samples for analysis. Add 641.11 g additional inhibited water to reconstitute slurry for leaching.	125000 ug Al/g (7.4E+7 ug Al) in initial slurry sample; 207 ug Al/mL (40674 ug Al) in initial permeate	Slurry prior to leaching: 155667 ug Al/g.	Water wash permeate #1: 324 ug Al/mL (556109 ug Al). Permeate #2: 214 ug Al/mL (370302 ug Al). Permeate #3: 142 ug Al/mL (233659 ug Al).	2.4% Al removed	Slurry samples measured on a dry basis (water removed by drying at 105°C). Permeate samples measured on a wet basis. Physical properties (i.e. density and particle size) and rheology data available in report.	Combine slurry with 2899.26 g 3M NaOH (2M NaOH concentration). Heat to 85°C and mix 8 hr. Dewater via filtration.
241-C-104	ORNL/TM-13500	1998	Lab scale caustic leaching; compare 50, 80, and 93°C caustic leaching at 3.99M NaOH	—	High aluminum, low phosphorous and bismuth. High phosphorous sludge with moderate concentrations of aluminum and chromium. Waste types include PUREX aluminum waste and Zr CW.  CC. <sup>1</sup>	Two bottles used to perform three leach tests. For leach tests 1 & 3: slurry-like material w/ water content of 63.5 wt% water, contained small piece of hard material resembling concrete. For leach test 2: usual consistency w/ water content of 43.7 wt%.	—	Original Sludge: 1 & 3) 31700 ug Al/g; 2) 36000 ug Al/g. Table 4.40 (dry, untreated sludge): 1&3) 86800 ug Al/g; 2) 63900 ug Al/g	—	—	—	Three leach tests performed: 1, 2.20 g sludge; 3.99M (25 mL) NaOH; 80°C; 65hr; 63.5 wt% moisture; 31 mL liquid/g solid. 2, 1.44 g sludge; 3.99M (15 mL) NaOH; 50°C; 63hr; 43.7 wt% moisture; 19 mL liquid/g solid. 3, 2.14 g sludge; 3.99M (21 mL) NaOH; 93°C; 65hr; 63.5 wt% moisture; 27 mL liquid/g solid.	



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-105	LAUR 97-2889	FY97	Lab scale caustic leaching	reddish-brown; dried material was light brown	TBP waste from U-extraction process at U plant, Sr wash of PUREX waste in AR vault, waste from dissolution of Zr or Al fuel cladding at PUREX, high-activity neutralized acid waste generated by PUREX. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP, <u>Secondary:</u> Particulates from Sr wash of PUREX wastes in the AR vault. <sup>2</sup> <u>Primary:</u> TBP, <u>Secondary:</u> particulates from Sr wash of PUREX wastes in AR-vault, <u>Tertiary:</u> CW, <u>Other:</u> neutralized acid waste. <sup>5</sup>	Portions of segment 3 of core 72 (222-S lab ID S96T001826, jar #9765)	Mix 6.71 g wet sludge with 11.4 mL water. With 4.99 g slurry, wash 3 times by adding 10mL 0.01 M NaOH; mix 1 hr at 100°C; cool; centrifuge; decant; repeat with solids. Dry solids at 105°C. 75.5 wt % insoluble solids & 32.97 mL supernate.	27.2 wt% Al	314925 ug Al/g, 398853 ug Al	52.5 ug Al/mL, 1683.68 ug Al	Sum: 3.16E-1% Al; Direct: 2.72E-1% Al; Mass recovery: 115.9%	—	Mix 6.71 g wet sludge w/ 11.4 mL water; subsample to get 2.97 g wet sludge (2.68 g dry sludge). Add 22.4 mL water and 8.7 mL 10M NaOH to get ~5wt% solids & ~2M NaOH; mix 5hr at 100°C; cool; measure settling; centrifuge; decant. Supernate: 35.1 mL w/ 2.55 M free OH, 1.84 mL settled solids, and 1.43 mL centrifuged solids.
241-C-105	ORNL/TM-13500	1998	Lab scale caustic leaching	—	Contains high concentrations of aluminum. Waste types include TBP uranium recovery and sludge wash waste from Sr extraction process. Non-Complexed Waste <sup>1</sup>	—	—	<b>Original Sludge:</b> 1) 247000 ug Al/g (276000 ug Al/g from Table 4.27)	—	—	—	—	1.51 g sludge; 6.33M (15 mL) NaOH; 70°C; 22hr; 4.7wt% moisture; 10 mL liquid/g solid.
241-C-106	PNNL-11381	FY96	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> Sr leached sludge, <u>Secondary:</u> Particulates from Sr wash of PUREX wastes in the AR vault. <sup>2</sup> <u>Primary:</u> Sr sludge, <u>Secondary:</u> particulates from Sr wash of PUREX wastes in the AR-vault, <u>Tertiary:</u> neutralized acid waste, <u>Other:</u> TBP. <sup>5</sup>	17 "grab" samples homogenized prior to testing and analysis	Mix 28.8 g water into 15.14 g sludge. Using 4.45 g slurry, wash 3 times by adding 10 mL 0.01 M NaOH to slurry, mix 1 hr at 100°C, cool, centrifuge, decant. Repeat w/ solids portion. For 3rd wash, add 10.1 mL NaOH. After 3rd wash, dry solids at 105°C.	1.529 g sludge; 0.587 g solids in initial unwashed sample. 4.8 wt% Al in untreated sludge solids.	0.243 g washed solids remaining, 86400 ug Al/g; 20995 ug Al (76% Al)	31.8 mL wash solution produced at pH = 11.1, 205 ug Al/mL; 6519 ug Al (24% Al)	Concentration in initial solids: Sum = 4.69E+04 ug Al/g; Direct = 4.85E+04 ug Al/g. 97% Al recovery.	(16 wt% insoluble solids in sludge)	After mixing 28.8 g water to 15.14 g sludge, begin leach of 4.76 g sludge solids. Add 10 M NaOH (10.6 mL) and water to a total volume of 45 mL (49.4 g slurry; 3.3 wt% solids). Mix 5 hr at 100°C. Cool, settle, and decant off liquid. <u>Liquid:</u> 31.6 mL with 2.6 M OH. <u>Solids:</u> 12.7 g slurry with 9.6 mL interstitial liquid.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-106	PNNL-11432	1997	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>	20 "grab" sample bottles combined to make composite sample. Mixed 3074 g sludge with 1965 g DI (minus 35 mL spilled). Mix 2 days (~9.8 wt% solids). 5 samples (~100 g) sent for analysis (4) or archive (1)	—	—	—	—	—	—	Add ~2340 g 10M NaOH to ~4494 g slurry in sludge receipt tank (lost ~584 g to evaporation). ~3.4M NaOH in solution; measured 2.7 M free OH <sup>-</sup> by titration. Mix & heat to 100°C. Mix for 5 hr at 100°C. Transfer to sludge settler and gravity settle at 85°C (lost ~306 g to evaporation). 5.9 wt% caustic-insoluble solids. Sample supernate. Decant 2578 mL supernate. Add 3286 g 3M NaOH to re-suspend solids (spill ~450 mL when recirculating to rinse settler. Made up by adding 503 g caustic.) Rinse, transfer to sludge receipt tank.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-106	PNWD-3013 (BNFL-RPT-017)	2000	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>	Portion of homogenized C-106 initial composite material prepared from 20 grab samples delivered to PNNL in June 1996. Used 60 g sub-sample of this homogenized dry material for tests. <u>Data from sample analysis is not easily correlated to individual samples from three procedures used in this study.</u>	<b>Determination of Aqueous-insoluble fraction:</b> 23.4777 g transferred to 125 mL HDPE bottle & filled to capacity w/ 0.01 M NaOH (104.41 g). Stir & heat at 85°C for 18 hr. Filtered hot through 0.45 um filtration unit (filtrate wt = 102.76 g). 0.01M NaOH used to transfer filtered solids back to bottle. Slurry volume increased to ~100 mL using 0.01 M NaOH (slurry wt. 108.83 g). Heat & stir at 85°C for 23 hr. Filter hot (94 g washing solution). Repeat a 3rd time, heating at 85°C for 27 hr and collecting 125.92 g washing liquid. Filtered solids transferred to pre-weighed glass jar with DI water. Excess water evaporated at 80°C, then dried overnight at 105°C, yielding 2.0286 g dried washed solids. Significant fraction stuck to magnetic stir bar. Stir bar transferred to glass jar, dissolved using 10 mL 12N HCl for 3.5 hr, with heat added for 1 additional hr. (~0.1014 g stuck to stir bar).	—	—	14% removed by wash	—	<b>Solubility versus temperature test:</b> 10.1157 g aliquot transferred to 60 mL HDPE bottle. ~50 mL (49.43 g) 0.1 M NaOH added to bottle and capped. Sample heated & stirred at 30°C for 19 hr + 3 hr. Two 4 mL aliquots taken & immediately filtered using preheated 0.45 um nylon syringe filter for analysis. T increased to 40°C and stirred for 18.5 hr. Sample again (same as done after first step). Increased T again to 50°C and stir for 24 hr. Sample and filter again.	24.7022 g aliquot transferred to 125 mL HDPE bottle. 108.08 g 3M NaOH added. Heat & stir at 85°C for 20 hr. Filter while still hot using 0.45 um nylon filtration unit. Filtrate wt = 104.07 g. Leachate solution sample analyzed.
241-C-107	LAUR 96-2839	FY95	Lab scale caustic leaching	wet dark red material	1C; waste produced at PUREX Plant from dissolution of Zr or Al fuel cladding; sludge feed from Sr extraction process at B Plant. Dilute Complexed Waste. <sup>1</sup> <u>Primary: 1C. Secondary: CW.<sup>2</sup></u>	—	Mix 11.0187 g wet sludge with 44 mL water. With 1mL of this sample, wash 3 times by adding 5mL 0.01 M NaOH/NaNO <sub>2</sub> ; mix for 0.5 hr at RT; centrifuge; decant; repeat with solids. Dry solids at 80°C = 0.0409 g or 19.4 wt % insoluble solids	10.5 wt % Al	—	—	2% removed by simple wash	Retrieval wash: mix 10.2464 g wet sludge (4.6122 g dry) with 42.3 mL water and 0.85 mL 0.01 M NaOH/NaNO <sub>2</sub> ; mix 1 hr at 100°C, cool, measure settling, centrifuge, decant and send solids to caustic leach 1.	Add 4.6 mL 10 M NaOH and 3.3 mL water to solids (14.5 mL settled solids/6.4 mL centrifuged solids); mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-107	PNNL-11278	FY96	Lab scale caustic leaching	—	<u>Primary:</u> 1C. <u>Secondary:</u> CW.  Dilute Complexed Waste. <sup>1</sup>  <u>Primary:</u> 1C. <u>Secondary:</u> Sr sludge. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	Core 71: Core composite was prepared at PNNL.	Mix 10.07 g sample with 39.73 g water. With 1.06 g slurry add 5 mL 0.01 M NaOH/NaNO <sub>2</sub> . Mix at RT for 0.5 hr, centrifuge, decant. Repeat for a total of 3 washes. Dry at 80°C to get 0.069 g solid. 32.2 wt% insoluble solids, 126 mL retrieval wash solution needed for 2.3 wt% solid yield, 21 mL caustic leach solution needed to yield 8 wt% solids.	9.5 wt%	—	—	1% removed	—	Wt. Sludge Solids in sample = 4.16 g. <u>Retrieval Wash:</u> Add 80.1 g water and 1.7 g 1M NaOH/NaNO <sub>2</sub> solution, mix at 100°C for 1 hr, cool, measure settling, centrifuge, decant (115 mL decanted liquid, 3.7 mL interstitial liquid, pH = 11.1). <u>Caustic Leach 1:</u> Add 9.09 g 10 M NaOH and 9.4 g water to solids, mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant to get 16.7 mL decanted liquid, 3 mL interstitial liquid, and 2.77 M OH <sup>-</sup> .
241-C-107	ORNL/TM-13500	1998	Lab scale caustic leaching	—	High phosphorus concentration waste. Waste types include 1C and CW.  Dilute Complexed Waste. <sup>1</sup>	—	—	<u>Original Sludge:</u> 1) 56600 ug/g (in dry, untreated sludge, 60500 ug/g Al)	—	—	—	—	4.37 g sludge; 6.33M (15 mL) NaOH; 70°C; 22hr; 46.3 wt% moisture; 6 mL liquid/g solid.
241-C-108	LAUR 95-2070	1995	Lab scale caustic leaching	composite sludge - brown paste	First waste received was TBP from TBP uranium extraction process at U plant; 1C; CW produced at PUREX plant from dissolution of Zr or Al fuel cladding; final waste was OWW from PUREX plant, containing carbonate, permanganate, and nitrate.  Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> TBP and FeCN-scavenged waste. <u>Secondary:</u> 1C. <sup>2</sup>  <u>Primary:</u> TBP/ FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> OWW. <sup>3</sup>	—	Add 49.4 mL water to 12.43 g wet sludge. With 1.0881 g slurry add NaOH/NaNO <sub>2</sub> to 0.01M (5 mL), mix at RT for >0.5 hr, centrifuge, decant, repeat 2 more times, dry solids at 80°C to get 0.0576 g solids and 26.4 wt% insoluble solids.	—	—	—	—	Detection limit = 4.25 ug/mL AND 119.96 ug/g	With 11.6871 g wet mixed sludge - <u>Retrieval Wash:</u> Add 84.2 mL water and 1.3 mL NaOH/NaNO <sub>2</sub> (to 0.01M with ~2.3 wt% solids), mix 1 hr at 100°C, cool, measure settling, centrifuge, decant (Vol. settled solids ~ 17.3 mL, Vol. Centrifuged solids ~ 6.1 mL, 128.63 mL liquid from decant). <u>Caustic Leach 1:</u> Add 7 mL 10 M NaOH (to ~3M solution and ~ 8 wt% solids) and 9 mL water, mix 5 hr at 100°C, cool, measure settling, centrifuge, decant (Vol. settled solids ~ 21.4mL, Vol. Centrifuged solids ~ 6.1 mL, 15.09 mL liquid from decant).



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-C-109	PNL-9387	FY93	Lab scale caustic leaching	A portion of the composite sample of Core 47 taken in 1992.	TBP U extraction process operated at U Plant (wastes treated w/ FeCN to scavenge 137-Cs from supernatant solution); 1C; Al &/or Zr CW; IX from B Plant Cs recovery process. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP and FeCN-scavenged waste. <u>Secondary:</u> 1C. <sup>2</sup> <u>Primary:</u> TBP/ FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	Core 47 taken from C-109 in 1992.	Mix 2 g sludge w/ 6 mL 0.1M NaOH. With 2.16 g slurry, mix 1hr at 100°C; centrifuge; decant; add 2 mL 0.1M NaOH to solids; mix 1 hr at 100°C, centrifuge; decant; add 5 mL water to solids; mix 0.5 hr at RT; centrifuge; decant; dry at 50°C (0.16 g dry sludge).	5.4E-2 g/g sludge	—	8% Al	—	Sludge washing and acid dissolution experiment done as well, but not noted here.	Add 1 mL water to dry solids from wash (save 0.12 g slurry for analysis); add 0.39 mL 10M NaOH (effectively 3M NaOH); mix 6 hr at 100°C; centrifuge; decant; add 1 mL 3M NaOH to solids; mix 5.5 hr at 100°C; centrifuge; decant; add 5 mL water to solids; mix 0.5 hr at RT; centrifuge; decant; add 1 mL water to solids; mix 0.5 hr at RT; take 0.6 g slurry sample for particle analysis; centrifuge; decant; dry solids at 50°C to obtain 0.07 g dry sludge. 7.3 mL sludge leach solution.
241-C-112	PNL-9387	FY93	Lab scale caustic leaching	A composite of 2 segments obtained in the 1992 Core 36 diluted with 3 parts water (by wt.).	TBP U extraction process operated at U Plant (wastes treated w/ FeCN to scavenge 137-Cs from supernatant solution); 1C; Al &/or Zr cladding wastes; IX from B Plant Cs recovery process. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> TBP and FeCN-scavenged waste. <u>Secondary:</u> 1C. <sup>2</sup> <u>Primary:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>5</sup>	—	Mix 2 g C36 composite w/ 6 mL 0.1M NaOH. Mix 2.3 g slurry for 1 hr at 100°C; centrifuge; decant; add 2mL 0.1M NaOH; mix 1 hr at 100°C; centrifuge; decant; add 5 mL water; mix 0.5 hr at RT; centrifuge; decant (total 8.2 mL sludge wash solution); dry at 50°C (0.35 g dry sludge).	4.5E-3 g Al/g sludge	—	34%	—	Sludge washing and acid dissolution experiment done as well, but not reported here.	Rehydrate solids from wash w/ 1 mL water (save 0.11 g slurry for analysis); add 0.39 mL 10M NaOH (~3M NaOH solution); mix 5 hr at 100°C; centrifuge; decant; add 1 mL 3M NaOH; mix 6.3 hr at 100°C; centrifuge; decant; add 5 mL water; mix 0.5 hr at RT; centrifuge; decant; add 1 mL water; mix 0.5 hr at RT; subsample 0.06 g slurry for particle analysis; centrifuge; decant (7.3 mL total sludge leach solution); dry solids at 50°C (0.2 g dry sludge).

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-S-101	PNNL-12026	FY98	Lab scale caustic leaching; multiple samples taken at two temperatures (70 and 95°C) and two [OH <sup>-</sup> ] (1 and 3 M)	—	Primary: High-level REDOX process waste. Secondary: EB. Non-Complexed Waste. <sup>1</sup>	222-S lab ID S96T005965; Jar # 11720; Composite made from segments 5 through 9 of core number 137.	Wash 4 times at ambient temperature. 110.61 g sludge portion in 250 mL centrifuge bottle with 103 g 0.01 M NaOH/NaNO <sub>2</sub> . Agitate for 24 hr end-over-end. Centrifuge at 4500 G; decant liquid; weigh wet solids. This followed by 2 washes at 97°C. Agitation times for washes were 1) 24 hr; 2) 16 hr; 3) 143 hr; 4) 21 hr; 5) >1 hr; 6) >1hr. Wt solution added were: 1) 102.89 g; 2) 102.28 g; 3) 100.43 g; 4) 103.21 g; 5) 120.73 g; and 6) 119.10 g. Wt of wet solids was: 1) 91.58 g; 2) 83.09 g; 3) 78.85 g; 4) 75.04 g; 5) 68.2 g; and 6) 66.78 g.	9.41 wt%; 9.41E+4 ug/g	1.04E+5 ug/g; 9.27E+6 ug Al	1.4E+3 ug/mL; 1.14E+6 ug Al	11% removed	—	4 leaching conditions with 4 different time samples for each. Leaching conditions were: <b>1)</b> 30 mL 1M NaOH per g initial sludge solids at 70°C; <b>2)</b> 14.9 mL 1M NaOH per g initial sludge solids at 95°C; <b>3)</b> 8.5 mL 3M NaOH per g initial sludge solids at 70°C; <b>4)</b> 4.5 mL 3M NaOH per g initial sludge solids at 95°C. Times for samples were at 5, 24, 72, and 168 hr. After leaching, the samples were centrifuged for 3.5 min. Centrifuged liquid was decanted and kept at leaching temp. The leached solids were washed 3 times with 25 mL 0.01 M NaNO <sub>2</sub> .
241-S-101	PNNL-11636	FY97	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> Primary: High-Level REDOX. Secondary: EB. <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: IX. Other: mixture of several miscellaneous wastes. <sup>5</sup>	222-S Lab ID S96T003486; Jar # 10519; sludge	Wash 3 times by adding 10 ml 0.01 M NaOH to 3.120 g slurry; mix 1 hr at 100°C; cool; centrifuge; decant; repeat w/ solids portion.	147000 ug/g	87338 ug	11578 ug	101%	—	Add 6.87 ml 10 M NaOH to 18.705 g slurry; mix 5 hr at 100°C; cool; let sit overnight; centrifuge ~20-ml portion and transfer solids back to vessel.
241-S-101	ORNL/TM-13500	1998	Lab scale caustic leaching	—	High chromium concentration waste. (High Al sample). Waste type is REDOX waste. Non-Complexed Waste. <sup>1</sup>	—	—	Original Sludge had 86300 ug/g (in dry, untreated sludge, 127000 ug/g Al).	—	—	—	—	One leach test performed with the following conditions: 1.90 g sludge; 3.99M (21 mL) NaOH; 93°C; 65hr; 31.8 wt% moisture; 16 mL liquid/g solid.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-S-101	ORNL/TM-13655	1998	Lab scale caustic leaching: multiple samples taken at two temperatures (70 and 95°C) and two [OH <sup>-</sup> ] (1 and 3 M).	—	Group 1 waste based on the SORWT model. Largest fraction of HLW is from REDOX process (formed sludge accumulation); second largest is EB (formed extremely hard salt cake).  Non-Complexed Waste. <sup>1</sup>	WHC 222-S lab # S96T005965, jar # 11720	All solids washed together 6 times. Wash #1 add 102.98 g inhibited water (0.01 M NaOH/NaNO <sub>2</sub> ) to 110.61 g sludge, mix at RT for 24 hr, centrifuge at 4500 rpm for 20 min, decant, weigh wet solids; #2 add 102.28 g inhibited water, mix at RT for 16 hr, centrifuge, decant, weigh solids; #3 add 100.43 g inhibited water, mix at RT for 143 hr, centrifuge, decant, weigh solids; #4 add 103.21 g inhibited water, mix at RT for 21 hr, centrifuge, decant, weigh solids; #5 add 120.73 g inhibited water, mix at 97°C for 1+ hr, centrifuge, decant, weigh solids; #6 add 119.10 g inhibited water, mix at 97°C for 1+ hr, centrifuge, decant, weigh solids.	From Table 2.1: 2.37E+5 kg Al/2.67E+6 kg total sludge mass. From Table 4.1: 9.41E+4 ug Al/g untreated solids (calculated by sum of wash solution + washed solids divided by total mass of S-101 sample used).	From Table 4.1: 1.04E+5 ug Al/g washed solids; 9.27E+6 ug Al total.	From Table 4.1: 1.40E+3 ug Al/mL wash solution; 1.14E+6 ug Al total.	11% removed	Weights of wet solids from 6 washes: 1) 91.58 g; 2) 83.09 g; 3) 78.85 g; 4) 75.04 g; 5) 68.2 g; and 6) 66.78 g.	Four leach conditions were conducted with a total of 4 samples per condition. The four leach conditions were: 1) 30 ml NaOH per g initial sludge solids with 1 M NaOH at 70°C; 2) 14.9 ml NaOH per g initial sludge solids with 1 M NaOH at 95°C; 3) 8.5 ml NaOH per g initial sludge solids with 3 M NaOH at 70°C; and 4) 4.5 ml NaOH per g initial sludge solids with 3 M NaOH at 95°C. The calculated amounts of NaOH were increased by 50% in the actual experiments to account for uncertainties associated with the calculations. The actual amounts used are shown here. Each of these conditions had 4 samples performed for run times of 5, 24, 72, and 168 hours.
241-S-104	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>  Primary waste type was high-level REDOX waste. <sup>5</sup>	Composite of segments 4 and 5 from core 43 (222-S Jar #6588)	Add 34.2 g water to 7.9 g S104-1, mix and sample. Using 1.12 g slurry: Wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to slurry; mix 0.5 hr at RT; centrifuge; decant; repeat with solids portion. Dry remaining solids at 80°C (0.09 g solid remains).	—	—	—	—	Retrieval Wash prior to caustic leach: add 99.2 g water and 1.5 g 1M NaOH/1M NaNO <sub>2</sub> to 6.1 g dry solids in slurry. Mix 1h at 100°C, cool, measure settling, centrifuge, decant.	Add 9.3 g of 10 M NaOH to 17mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.
241-S-104	LAUR 95-2070	1995	Lab scale caustic leaching	A composite sample consisting of dry tan material.	Primary waste type was high level waste from the REDOX process.  Non-Complexed Waste. <sup>1</sup>	—	Add 25.8 mL water to 6.4603 g wet sludge. With 1.1157 g slurry add NaOH/NaNO <sub>2</sub> to 0.01M (5 mL), mix at RT for 0.5 hr, centrifuge, decant, repeat 2 more times, dry solids at 80°C (0.0822 g solids; 36.8 wt% insoluble solids).	—	—	—	—	Detection limit = 4.25 ug/mL AND 124.85 ug/g.	Due to experimental problem, sample is 6.4427g sludge + 25.8 mL water (5.5302 g dry). Retrieval Wash: Add 70 mL water and 1 mL NaOH/NaNO <sub>2</sub> to 0.01 M, mix 1 hr at 100°C, cool, measure settling, centrifuge, and decant. Volume settled solids ~ 58.9 mL, volume centrifuged solids ~ 12.1 mL). Caustic Leach 1: Add 5.4 mL 10 M NaOH to ~3M solution, mix 5 hr at 100°C, cool, measure settling, centrifuge, and decant. Volume settled solids ~ 19.7 mL, volume centrifuged solids ~ 10.3 mL.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-S-104	PNNL--11089	FY95	Sludge washing and caustic leaching	—	Primarily REDOX process HLW.	Core # 43	Add water to sludge sample to get ~2.3 wt% solids, as well as inhibited water (NaOH/NaNO <sub>2</sub> ) to 0.01 M. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	3% recovered	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 3.8M. Mix at 100°C for 5 hr. Cool, centrifuge, decant.
241-S-104	PNNL-11636	FY97	Lab scale caustic leaching.	—	Non-Complexed Waste. <sup>1</sup> Primarily high-level REDOX waste. <sup>2</sup>	—	Wash 3 times by adding 10 ml 0.01 M NaOH to 9.01 g slurry; mix 1 hr at 100°C; cool; centrifuge; decant; and repeat w/ solids portion.	153000 ug/g	307063.5 ug	5871 ug	128%	Solution density = 1.18 g/mL. 1.685 g sludge (1.595 g solids) initially in B1; 0.807 g washed solids in final analysis. Sample B1 has 9.01 g slurry (see Fig. 3.1).	Add 13.3 ml 10 M NaOH and 10 ml 0.01 M NaOH to 54.938 g slurry; mix 5 hr at 100°C; cool; centrifuge; take a 0.5ml sample; mix for 70 hr at 100°C; cool; centrifuge; take a 0.5ml sample; mix 136 hr at 100°C; cool; centrifuge; decant.
241-S-104	ORNL/TM-13500	1998	Lab scale caustic leaching at three temperatures (67, 70, and 80°C) and two [OH] (3.8, 3.99, and 6.33 M)	—	REDOX waste characterized by average aluminum concentration and high chromium concentration. Non-Complexed Waste. <sup>1</sup>	—	—	1-6) 140000 ug/g	—	—	—	—	<b>6 leach tests performed:</b> <b>1)</b> 3.01 g sludge; 3.8M (23 mL) NaOH; 67°C; 4 hr; 15.6 wt% moisture; 9 mL liquid/g solid. <b>2)</b> 3.02 g sludge; 3.8M (23 mL) NaOH; 67°C; 24 hr; 15.6 wt% moisture; 9 mL liquid/g solid. <b>3)</b> 3.06 g sludge; 3.99M (15 mL) NaOH; 70°C; 21 hr; 15.6 wt% moisture; 6 mL liquid/g solid. <b>4)</b> 1.49 g sludge; 3.99M (15 mL) NaOH; 70°C; 21 hr; 15.6 wt% moisture; 12 mL liquid/g solid. <b>5)</b> 1.51 g sludge; 6.33M (15 mL) NaOH; 70°C; 21 hr; 15.6 wt% moisture; 12 mL liquid/g solid. <b>6)</b> 1.10 g sludge; 6.33M (30 mL) NaOH; 80°C; 126 hr; 15.6 wt% moisture; 32 mL liquid/g solid.
241-S-107	PNNL-11278	FY96	Lab scale caustic leaching	—	<u>Primary:</u> High-level REDOX process waste. <u>Secondary:</u> EB. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> High-Level REDOX; <u>Secondary:</u> EB. <sup>2</sup>	Cores 105, 110, and 111: Composite of three core composites (222-S LABCORE sample numbers S95T003158, S95T003159, and S95T003164).	Mix 8.393 g sample with 15.5 g water. With 4.75 g slurry add 10 mL 0.01 M NaOH. Mix at 100°C for 1 hr, cool, centrifuge, and decant. Repeat for a total of 3 washes. Dry solids after wash #3 at 105°C. Had 1.668 g sludge initially in sample, 0.622 g of washed solids, and 37 wt% insoluble solids in sludge.	20.5 wt%; <u>Summation:</u> 2.02E+5 ug/g; <u>Direct:</u> 2.05E+5 ug/g	303000 ug/g; 188466 ug (92 wt%)	522 ug/mL; 17017 ug (8 wt%)	8% removed	—	Weight of sludge solids in sample was 3.4 g. Add 8.5 mL 10 M NaOH and water to a total volume of 37 mL (1.8 wt% solids), mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant. Measured 29.8 mL decanted liquid, 4.2 mL interstitial liquid, and 2.1 M OH.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-S-107	PNNL-12010	FY98	Lab scale caustic leaching; performed extended caustic leach at 80°C.	—	Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> High-level REDOX waste. <u>Secondary:</u> EB. <u>Tertiary:</u> CW. <u>Other:</u> IX/mixture of several miscellaneous wastes. <sup>5</sup>	11 Containers from 222-S Laboratory combined (using 205 g DI water and mixer) to make homogeneous sludge sample. Average solids wt% = 67.5; Average sludge bulk density = 1.815 g/mL.	Mix 1005.4 g sample with 3128.4 g inhibited water for 30 min at 100°C. Lost 125 g due to evaporation and 99 g from taking 4 slurry samples. Add 404 g DI water and perform settling test (8.25 cm/hr at 13.6 wt% solids & 80°C; lost 940 g to evaporation and 57 g to 3 liquid samples). Refluidize, using 3080.4 g inhibited water and 700 g DI, and perform second settling test (16.66 cm/hr at 7.7 wt% solids & 80°C; lost 598 g to evaporation, 42 g to 3 liquid samples, decanted 4537 g supernate).	—	—	799 ug/g, 4.74E+6 ug Al (2.5%)	—	Performed many settling tests and particle size analyses. Data available within document.	Add 501.1 g 10M NaOH and 969 g DI to sludge with a target of 2M NaOH in solution & 5 wt% insoluble solids after leach; actual free OH <sup>-</sup> = 1.98M & 10.9 wt% insoluble solids. Mix at 100°C for 5 hr (lost 139 g due to evaporation and 92 g removed as 4 slurry samples). Cool to 80°C and transfer to settler. Add 4552 g inhibited water for settling test (3.17 cm/hr at 14.9 wt% solids & 80°C; lost 662 g to evaporation and 40 g to 3 liquid samples). Decant supernate and add DI to fluidize mixture for transfer to sludge-receipt tank.
241-S-110	PNNL-13702	FY01	Lab scale caustic leaching; multiple samples taken at three temperatures (60, 80, and 100°C) and two [OH <sup>-</sup> ] (~1, ~3, and ~5 M)	—	Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> High-level REDOX waste. <u>Secondary:</u> EB. <u>Tertiary:</u> mixture of several miscellaneous wastes. <sup>5</sup>	Composite of two core samples. From core 240, riser 14: Sample ID S98T001898 (30 g of segment 9) and S98T001904 (30.7 g of segment 10). From core 241, riser 6: S98T001978 (segment 2, 30.2 g), S98T001984 (segment 3, 30g), S98T001994 (segment 4, 30.1 g), S98T002014 (segment 7, 30g), and S98T002026 (segment 8, 30.1 g).	Transfer 211 g composite to 500 mL HDPE bottle with 0.01M NaOH, adding to total vol. of 400 mL. Shake overnight. Stand 2 hr. Split between 2 centrifuge bottles and centrifuge 20 min at 1200G. Decant wash liquor and save. Transfer solids to 500 mL bottle and repeat for second wash. For third wash, conduct in centrifuge bottles. Add 0.01M NaOH to each centrifuge bottle to yield 175 mL total volume. Shake overnight & centrifuge. Decant wash liquor and combine all together.	—	Total mass of washed solids = 74.96 g. 3.25E+5 ug Al/g. 24362000 ug Al	Total mass of combined wash solution = 764.7 g. 1410 ug Al/g, 1077863 ug Al.	4% removed	—	Perform 9 caustic leach studies at varying temperatures & hydroxide concentrations, sampling at multiple times.  [Mass slurry (g), mass solids (g), Temp (°C), NaOH (M)]: <b>1)</b> 15.4g, 1.189g, 60°C, 1M. <b>2)</b> 15.2g, 1.173g, 60°C, 2.8M. <b>3)</b> 15g, 1.158g, 60°C, 4.8M. <b>4)</b> 15.1g, 1.166g, 80°C, 0.9M. <b>5)</b> 15g, 1.158g, 80°C, 2.7M. <b>6)</b> 15.1g, 1.166g, 80°C, 4.6M. <b>7)</b> 15g, 1.158g, 100°C, 0.8M. <b>8)</b> 15.2g, 1.173g, 100°C, 2.7M. <b>9)</b> 15.1g, 1.166g, 100°C, 4.6M.  10M NaOH was added to each aliquot of washed solids (11 mL for 1M NaOH, 31 mL for 3M NaOH, and 51 mL for 5M NaOH tests). Leaching mixtures diluted to 100 mL with DI (ratio of ~80 mL solution per g washed solids). Samples mixed, with leachate samples taken at 4, 8, 24, 72, and 168 hr. Stirring stopped and solids settled at T to take samples using 0.45 um preheated filters.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-S-110	PNNL-14018	FY01	Lab scale caustic plus oxidant leaching	—	Primarily REDOX waste, with secondary stream from EB and tertiary stream composed as a mixture of several miscellaneous wastes. From cluster analysis, contains saltcake from 242A campaign (1976-1980 and 1981-1988), saltcake from the 242-B operation (1951-1953), saltcake from the 242-S campaign (1973-1976), saltcake from self condensation, REDOX waste, and saltcake from the 242-S campaign (1977-1980).  Non-Complexed Waste. <sup>1</sup>	Composite of two core samples. From core 240, riser 14: Sample ID S98T001898 (30 g from segment 9) and S98T001904 (30.7 g from segment 10). From core 241, riser 6: S98T001978 (segment 2, 30.2 g), S98T001984 (segment 3, 30g), S98T001994 (segment 4, 30.1), S98T002014 (segment 7, 30g), and S98T002026 (segment 8, 30.1 g).	Transfer 211 g composite to 500 mL HDPE bottle. Contact 3 times with fresh portions of 0.01M NaOH, with the supernate removed after each contact. Final slurry prepared by adding final portion of 0.01M NaOH to washed solids. Two weighed aliquots were removed and dried at 105°C to constant wt. (7.72 wt% insoluble solids).	—	325000 ug Al/g dried solids	—	—	Study mostly concerning Cr removal using various oxidants, temperatures, and OH <sup>-</sup> concentrations.	<b>Oxidative Leach:</b> Separate composite washed sample into 16 aliquots, each containing nominally 15 g slurry and ~ 1 g insoluble solids, transferred to 125 mL polymethylpentene bottles. To samples, stock solutions of oxidant, 10M NaOH and DI water were added to meet target experimental conditions and ~ 100 mL slurry volume. A total of 16 different experimental conditions were analyzed.  [oxidant, NaOH, T, [Cr]/[Oxidant] <sub>final</sub> ]:  1) None, 0.1M, 30°C, N/A. 2) None, 3M, 30°C, N/A. 3) None, 0.1M, 85°C, N/A. 4) None, 3M, 85°C, N/A. 5) NaMnO <sub>4</sub> , 0.1M, 30°C, 1.5. 6) NaMnO <sub>4</sub> , 3M, 30°C, 1.5. 7) NaMnO <sub>4</sub> , 0.1M, 80°C, 1.5. 8) NaMnO <sub>4</sub> , 3M, 80°C, 1.5. 9) K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 0.1M, 30°C, 3. 10) K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 3M, 30°C, 3. 11) K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 0.1M, 80°C, 3. 12) K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 3M, 80°C, 3. 13) NaFeO <sub>4</sub> , 1.1M, 30°C, 1.5. 14) NaFeO <sub>4</sub> , 3M, 30°C, 1.5. 15) NaFeO <sub>4</sub> , 1.1M, 80°C, 1.5. 16) NaFeO <sub>4</sub> , 3M, 80°C, 1.5.  Total leach time = 48 hr.
241-S-111	PNNL-11636	FY97	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> High-Level REDOX, <u>Secondary:</u> EB. <sup>2,5</sup>	222-S Lab ID S96T005106; Jar # 11150; sludge received Dec. 5, 1996.	Wash 3 times by adding 20 ml water to 9.365 g slurry; mix; centrifuge; take 0.2 mL sample; mix 1 hr at 100°C; cool; centrifuge; decant; * add 30 mL 0.01 M NaOH; mix 1 hr at 100°C; cool; centrifuge; decant; repeat from * with solids portion.	In untreated sludge - 160215 ug/g wet sludge.	—	141897 ug. 9% Al in initial wash step before leaching.	—	—	Using solids from wash step, add 5.5 mL 10 M NaOH and 13.5 mL water; mix 5 hr at 100°C; cool; centrifuge; take 0.1 mL sample; add 0.53 mL 10 M NaOH; mix 5 hr at 100°C; cool; centrifuge; take 0.5 mL supernate and 0.1 mL aliquot; mix 133 hr at 100°C; cool; centrifuge; decant.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-SX-101	PNWD-3512 (WTP-RPT-117)	Sample shipped to PNWD in July 2003	Lab scale caustic leaching	Composite of black slurry sludge-containing segments	Dilute Complexed Waste. <sup>1</sup>	Approximately 75g from Core 225, Sample ID S03T001373, Jar 13998.	Transfer portion into separate 50 mL centrifugation cone. Wash 2 times with 0.01 M NaOH so total volume was 50 mL. Centrifuge and separate solid/liquid. Transfer solid to 50 mL centrifuge bottles and contact approximately 5 times (until bulk of color removed) with fresh 0.01 M NaOH and a solution-to-solid ratio of approximately 5:1. Decant supernate after each contact and discard. Prepare final slurry, adding a portion of 0.01 M NaOH to washed, centrifuged solids. Remove two weighed aliquots of stirred suspension and dry to constant weight at 105°C.	—	229000 ug/g dried solid.	—	—	Could not find initial analyses of elements in untreated sample.	Use samples SX-101-3 and SX-101-4 with slurry weights of 5.402 and 4.823 g, respectively. Add 4.8 ml 10M NaOH and 8.2 ml DI to each. Heat at 85 ± 5°C for 8 hr. Cool to RT overnight. Centrifuge and decant supernate to 30 mL plastic vials. Add 0.1M NaOH at 3:1 vol:vol. ratio to remaining solids. Mix, separate, and decant to same container as initial leachate solution. Continued washings until supernatant was colorless. Filter samples (0.2 um syringe filter), and place 1 ml aliquot in container w/ 10 mL 1M HNO <sub>3</sub> for ICP-AES.
241-SX-108	PNNL-11278	FY96	Lab scale caustic leaching	—	Primarily high-level REDOX process waste: Non-Complexed Waste. <sup>1</sup> Primarily high level REDOX. <sup>2</sup>	Auger Sample: Upper half of auger sample (222-S LABCORE sample #S95T002574).	Mix 23.47 g sample with 47.32 g water. With 4.55 g slurry add 10 mL 0.01 M NaOH. Mix at 100°C for 1 hr, cool, centrifuge, and decant. Repeat for a total of 3 washes. Dry solids after washes at 105°C. 1.506 g sludge initially in sample, 0.618 g washed solids and 41 wt% insoluble solids in sludge.	9 wt%; Summation: 1.3E+5 ug/g; Direct: 9.02E+4 ug/g	287000 ug/g; 177366 ug (94 wt%)	372 ug/mL; 11830 ug (6 wt%)	6% removed; Total mass = 189196 ug; 144% recovery.	—	Wt. Sludge Solids in sample was 20.22 g. Add 39 mL 10 M NaOH and water to a total volume of 151 mL (4.1 wt% solids), mix at 100°C for 5 hr, cool, measure settling, and decant. 97 mL decanted liquid, 46 mL interstitial liquid, and 2.7 M OH.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure	
241-SX-108	PNNL-11908	FY98	Lab scale caustic plus oxidant leaching	—	SOWRT Group IV. Primary waste type is high-level REDOX process waste.  Non-Complexed Waste. <sup>1</sup>  Primary waste type was high-level REDOX waste. <sup>5</sup>	—	Add 0.01 M NaOH to 28.63 g sludge to a total volume of 100 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Wash 2 more times, adding 0.01M NaOH to residual solids to total volume of 100 mL, mixing for 1 hr at RT, centrifuging and discarding supernate. <b>Split residual solids to A and B.</b> For A: Add 3M NaOH to residual solids to total volume of 125 mL. Heat for 1 week at 100°C. Centrifuge and discard supernate. Wash 3 times by adding 0.01M NaOH to residual solids to a total volume of 100mL, mixing for 1 hr at RT, centrifuging and discarding supernate. For A: Add 0.01 M NaOH to residual solids to a total volume of 50 mL. Transfer 2 mL of suspension to obtain weight of insoluble solids (0.0132 g) by drying at 105°C. Transfer ~25 mL portions to two bottles for testing. For B: With residual solids in suspension, transfer 2 mL suspension and dry at 105°C to obtain wt of insoluble solids (0.0889 g). Transfer remaining suspension to two ~25 mL portions for testing.	—	270000 ug Al/g dry solids	—	—	—	—	<b>Four oxidative leach tests performed:</b> 1) add solid NaMnO <sub>4</sub> to sludge suspended in 0.1M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 2) Add solid NaMnO <sub>4</sub> to sludge suspended in 3M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 3) Contact sludge, suspended in 0.1M NaOH, with oxygen. 4) Contact sludge, suspended in 3 M NaOH, with excess oxygen. Samples were stirred at RT for 24 hr, then heat to 80°C for remainder of experiment. DI was added to replenish evaporated water. Test samples taken at time 0 hr, with intermittent samples taken throughout the remainder of the experiment. Test slurries were centrifuged and supernate decanted after tests completed.
241-SX-113	LAUR 97-2889	FY97	Lab scale caustic leaching	As-received: opaque and tan in color; treated: translucent and red in color.	Non-Complexed Waste. <sup>1</sup>  Primary: High level REDOX, Secondary: Diatomaceous earth. <sup>2,5</sup>	222-S Lab ID S96T001524, jar #9146	Mix 5.81 g wet sludge with 11.6 mL water, then use 4.8 g sample for wash. Wash 3 times by adding 10 mL 0.01M NaOH; mix 1 hr at 100°C; cool; centrifuge; decant. Had 29.8 mL supernate and 48.5 wt% insoluble solids after dry at 105°C.	2.1 wt%.	21419 ug/g, 16135 ug	2.1 ug/mL, 62.58 ug	Sum: 2.74E-2; Direct: 2.12E-2; 129.3% mass recovery.	—	Mix 5.81 g wet sludge with 11.6 mL water; subsample to get 3.25 g wet sludge/1.63 g dry sludge in sample; Add 13.1 mL water & 6.7 mL 10 M NaOH (~5 wt% solids & ~2M NaOH); mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant. Had 19.52 mL supernate with 2.27 M free OH <sup>-</sup> , 14.7 mL settled solids, and 5.9 mL centrifuged solids.	



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-SX-113	ORNL/TM-13500	1998	Lab scale caustic leaching: two leach tests at 70 and 80°C and 6.33 M NaOH.	—	REDOX waste characterized as a low Al, low Cr-bearing sludge.  Non-Complexed Waste. <sup>1</sup>	—	—	166000 ug/g; Original Sludge: 7320 ug/g (14000 ug/g Table 4.23)	—	—	—	—	Two leach tests performed: 1) 1.46 g sludge; 6.33M (15 mL) NaOH; 70°C; 21hr; 47.9 wt% moisture; 20 mL liquid/g solid. 2) 1.32 g sludge; 6.33M (30 mL) NaOH; 80°C; 126hr; 47.9 wt% moisture; 44 mL liquid/g solid.
241-T-104	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>  Primary waste was 1C. <sup>2,5</sup>	Composite of segments 2 through 4 from core 46 (222-S Jar #6555)	Add 39.1 g water to 13.4 g T104-1, mix, and sample. Using 1.07 g slurry, wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to slurry; mix 0.5 hr at RT; centrifuge; decant; repeat with solids portion. Dry remaining solids at 80°C (0.035 g solid remained).	—	—	—	—	Retrieval Wash prior to caustic leach: add 37.2 g water and 0.97 g 1M NaOH/1M NaNO <sub>2</sub> to 4.18 g dry solids in slurry. Mix 1 hr at 100°C, cool, measure settling, centrifuge, decant.	Add 5.7g water and 3g 10 M NaOH to 7.3mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.
241-T-104	PNNL-11089	FY95	Sludge washing and caustic leaching	—	Primarily 1C waste.	Core # 46	Add water to sludge sample to get ~2.3 wt% solids, as well as inhibited water (NaOH/NaNO <sub>2</sub> ) to 0.01 M. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	uncorrected/ corrected recovery = 1/1%	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 0.3M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.
241-T-104	ORNL/TM-13660	Report issued in 1998	Sludge washing and caustic leaching at RT (~22°C), ~60°C, and ~95°C	—	—	—	Add 10 mL inhibited water to initial sludge sample (6.09g for RT; 4.4g for 60°C, and 6.18g for 95°C) and place on shaker at appropriate temperature. Mix for 65 min and centrifuge for 8 min.	1.82 ug Al/g solids	—	T=RT: 11.5 ug/mL. T=60°C: 18.5 ug/mL. T=95°C: 19.3ug/mL.	—	Solids from wash observed to have three distinct layers and a volume of ~4 mL. Liquid was yellow. Solution was filtered through a 0.45 um filter, sealed, and stored overnight.	Add ~10 mL 3M NaOH to samples. Mix 4 hr 45 min. Cool slightly and centrifuge. Decant liquid and filter through 0.45 um filter. Solids volume for samples 1 (RT) = ~3.5 mL; sample 2 (60°C) = ~ 3 mL; and sample 3 (95°C) = ~ 3 mL. Samples were sealed and stored overnight.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-T-104	LAUR 95-2070	1995	Lab scale caustic leaching	<u>Untreated:</u> yellowish in color and showed both yellow and white particles under a stereoscope; <u>Treated:</u> brownish solution.	1C waste Non-Complexed Waste. <sup>1</sup>	—	Add NaOH/NaNO <sub>2</sub> to 0.01M solution, mix at 100°C for 1 hr, settle and decant.	—	29 ug/mL (0.94%)	—	—	Detection limit for sludge was 3.137 ug/g.	Add NaOH to 3.2M, mix at 100°C for 5 hr, settle and decant.
241-T-107	LAUR 95-2070	1995	Lab scale caustic leaching	As received sample was completely dry and composed of dark material and white granules. Both treated and as-received samples contained reddish/reddish-brown solutions with settleable particles.	Three significant types of waste: 1C, PUREX CW, and TBP from the TBP U-extraction process at U Plant. Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> 1C. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary:</u> TBP/FeCN-scavenged waste. <u>Secondary:</u> 1C. <u>Tertiary:</u> CW. <u>Other:</u> IX. <sup>3</sup>	—	Add NaOH/NaNO <sub>2</sub> to 0.01M, mix at 100°C for 1 hr, settle and decant.	—	150 ug/mL (3.56%)	—	—	—	To solids, add NaOH to 3.2M, mix at 100°C for 5 hr, settle and decant.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-T-110	PNNL-13956	FY02	Lab scale caustic leaching: nine leach tests at three temperatures (60, 80, & 100°C) and three [OH] (~1, ~3, and ~4M) with samples taken over time.	—	Non-Complexed Waste. <sup>1</sup>  Primary: 2C. Secondary: lanthanum fluoride decontamination waste. <sup>5</sup>	<p>Composite of two cores (180 and 181).</p> <p>For Core 180 [Sample ID, segment #, g added]:  <u>S97T000215</u>, 1, 10.3g;  <u>S97T000255</u>, 2, 10g; <u>S97T000225</u>, 3, 10g;  <u>S97T000227</u>, 4, 10.1g;  <u>S97T000229</u>, 6, 10g; <u>S97T000258</u>, 7, 10.2g; and <u>S97T00060</u>, 8, 10g.</p> <p>For Core 181 [Sample ID, segment #, g added]:  <u>S97T000126</u>, 1, 10.3g;  <u>S97T000135</u>, 2, 10.1g;  <u>S97T000138</u>, 3, 10g; <u>S97T000139</u>, 4, 9.9g;  <u>S97T000167</u>, 5, 10g; <u>S97T000177</u>, 6, 9.9g;  <u>S97T000161</u>, 7, 9.9g; and <u>S97T000194</u>, 8, 10.2g.</p> <p>Total mass = 150.9 grams.</p>	Transferred 150 g composite to 1L HDPE bottle; 0.01M NaOH added to total volume of 500 mL and agitated with mechanical stirrer for ~1hr. Settle for 2 hr. Remove supernate with pipette (~250 mL). Stir settled solids and divide equally between two 200 mL centrifuge bottles. Dilute both with 0.01 M NaOH and magnetically stir ~1 hr. Centrifuge, decant, combine decant fluid with previous wash liquid. Add 0.01M NaOH to solids to total volume of 200 mL and repeat wash. Combine supernate (985.8 g). Combine solids using DI (508.2 g).	—	1048 ug/g; 44226 ug Al	0	—	—	<p><b>Nine aliquots with varying leach conditions [g slurry, g solids, temp (°C), M NaOH]:</b></p> <ol style="list-style-type: none"> <li>1) 30.1g, 2.498g, 60°C, 0.59M.</li> <li>2) 30.1g, 2.498g, 60°C, 2.5M.</li> <li>3) 30.1g, 2.498g, 60°C, 3.8M.</li> <li>4) 30.2g, 2.507g, 80°C, 0.55M.</li> <li>5) 30.3g, 2.515g, 80°C, 2.7M.</li> <li>6) 30.3g, 2.515g, 80°C, 4.3M.</li> <li>7) 30.2g, 2.507g, 100°C, 0.62M.</li> <li>8) 30.2g, 2.507g, 100°C, 2.5M.</li> <li>9) 30.3g, 2.515g, 100°C, 4.3M.</li> </ol> <p>Add 10M NaOH to each aliquot: 5.5 mL to yield 1M NaOH, 15.5 mL to yield 3M NaOH, and 25.5 mL to yield 5M NaOH. Leaching mixtures diluted with 50 mL DI. Target ratio of ~20 mL solution per gram washed solids. Mix, adding DI to maintain liquid level. Sample at 4, 8, 24, 72, and 168 hr using preheated transfer pipette and 0.45 um nylon syringe filter, filtering ~1 mL leachate solution. 0.5 mL aliquot acidified for analysis, with remaining volume transferred back to reaction vessel. After 168 hr, additional samples taken for analysis. At conclusion of test, vessels centrifuged for 5 min at ~1200 G, decanting supernate.</p>



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-T-111	PNL-10712	FY95	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup> Primary: 2C. Secondary: Lanthanum fluoride decontamination waste. <sup>2,5</sup>	Composite of segments 1 and 3 from core 33 (222-S Jar #6591).	Add 80.7 g water to 20.1 g T111-3, mix, and sample. Using 1.07 g slurry: Wash 3 times by adding 5 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to slurry; mix 0.5 hr at RT; centrifuge; decant; and repeat with solids portion. Dry remaining solids at 80°C (0.029 g solid remains).	—	—	—	—	Retrieval Wash prior to caustic leach: add 33.9 g water and 1.2 g 1M NaOH/1M NaNO <sub>2</sub> to 4.27 g dry solids in slurry. Mix 1h at 100°C, cool, measure settling, centrifuge, and decant.	Add 7.8g 10 M NaOH to 14mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.
241-T-111	PNNL-11089	FY95	Sludge washing and caustic leaching	—	Primarily 2C, with secondary waste from lanthanum fluoride decontamination waste from the BiPO <sub>4</sub> process.	Core # 33	Add water to sludge sample to get ~2.3 wt% solids, as well as inhibited water (NaOH/NaNO <sub>2</sub> ) to 0.01 M. Mix at 100°C for 1 hr. Cool, centrifuge, and decant.	—	—	—	—	—	Caustic leach of solids: Add NaOH to get ~8 wt% solids and a final NaOH of 3.5M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.
241-TY-104	LAUR 96-2839	FY95	Lab scale caustic leaching	wet reddish-brown paste	TBP waste from U-extraction process at U Plant; 1C which had been FeCN scavenged; decontamination waste wash solution from equipment decontamination efforts at T Plant (mostly dilute NaNO <sub>2</sub> solution). Non-Complexed Waste. <sup>1</sup> Primary: TBP, Secondary: 1C and FeCN-scavenged waste. <sup>2</sup> Primary: TBP. Secondary: 1C/FeCN-scavenged waste. Tertiary: decontamination waste. Other: mixture of several miscellaneous wastes/high-level REDOX waste. <sup>5</sup>	—	Mix 11.4065 g wet sludge with 45.6 mL water; with 1mL of this sample, wash 3 times by adding 5mL 0.01 M NaOH/0.01 M NaNO <sub>2</sub> ; mix for 0.5 hr at RT; centrifuge; decant; and repeat with solids. Dry solids at 80°C to get 0.0723 g or 33.6 wt % insoluble solids.	4.3 wt % Al	—	—	9% removed by simple wash.	Mix 11.4441 g wet sludge with 45 mL water (4.8916 g dry). Retrieval wash adding 32.4 ml water and 0.78 mL 0.01 M NaOH/NaNO <sub>2</sub> , mix 1 hr at 100°C, cool, measure settling, centrifuge, decant and send solids to caustic leach 1.	Add 4 mL 10 M NaOH and 3.9 mL water to solids (18 mL settled solids/4.7 mL centrifuged solids); mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-U-108	PNNL-11908	FY98	Lab scale caustic plus oxidant leaching.	—	SOWRT Group VI. Primary waste type is EB, with secondary waste type being CW.  Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> EB. <u>Secondary:</u> CW. <u>Tertiary:</u> mixture of several miscellaneous wastes. <sup>5</sup>	—	Add 0.01 M NaOH to 50 g sludge to a total volume of 250 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01 M NaOH to residual solids to a total volume of 150 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01 M NaOH to residual solids to total volume of 150 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01M NaOH to residual solids to total volume of 50 mL. Transfer 2 mL and dry at 105°C to obtain dry insoluble solids weight (0.5007 g). Transfer ~4.5 mL portions to 6 bottles for further testing.	—	42600 ug Al/g dry solids	—	—	—	<b>Six oxidative leach tests performed:</b> 1) Add solid NaMnO <sub>4</sub> to sludge suspended in 0.1M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 2) Add solid NaMnO <sub>4</sub> to sludge suspended in 3M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 3) Contact sludge, suspended in 0.1M NaOH, with O <sub>2</sub> . 4) Contact sludge, suspended in 3 M NaOH, with excess O <sub>2</sub> . 5) Bubble Ar gas through sludge suspended in 0.1M NaOH. 6) Bubble Ar gas through sludge suspended in 3M NaOH. Samples were stirred at RT for 24 hr, then heat to 80°C for remainder of experiment. DI was added to replenish evaporated water. Test samples taken at time 0 hr, with intermittent samples taken throughout the remainder of the experiment. Test slurries were centrifuged and supernate decanted after tests completed.
241-U-108	PNNL-14019	2001	Lab scale caustic plus oxidant leaching	—	Tank waste group 17.  Non-Complexed Waste. <sup>1</sup>	<b>Composite of seven 25 g samples from three cores [222-S Lab sample ID, segment #]:</b> Core 141: [S96T002249, 1], [S96T002237, 4A], [S96T002602, 6]. Core 145: [S96T002870, 5], [S96T002907, 9]. Core 146: [S96T002950, 3A], [S96T003142, 9].	Transfer composite to 200 mL HDPE centrifuge bottles and contacted 4 times with fresh 0.01M NaOH at initial solution: solid ratio of 3:1 to 4:1. After each contact, decant and discard supernate. After final wash, prepared slurry by adding final portion of 0.01M NaOH to washed solids. Three weighed aliquots of well-stirred suspension removed and dried to constant weight at 105°C. 47.9 g slurry remained.	—	3.63 g (7.75 wt%) insoluble solids; 126440 ug Al/g dried solids	—	—	—	<b>Six oxidative leaching tests performed:</b> Each aliquot contained nominally 7 g slurry and ~0.5g insoluble solids in 60 mL polypropylene bottles. Stock solutions of oxidant, 10M NaOH, and DI were added as needed to meet experimental conditions and 50 mL total slurry volume. Initial experimental conditions were: 1) 0.1M NaOH, 30°C. 2) 3M NaOH, 85°C. 3) 0.1M NaOH, 30°C, 1 [Cr]/[NaMnO <sub>4</sub> ]. 4) 3M NaOH, 85°C, 1.01 [Cr]/[NaMnO <sub>4</sub> ]. 5) 0.2 to 0.3M NaOH, 30°C, 3.85 [Cr]/[ONOO]. 6) 3M NaOH, 85°C, 2.56 [Cr]/[ONOO]. Samples were taken at 2, 4, 6, and 24 hr after initial contact with leach solution. Aliquots filtered through 0.2 um filter and diluted with 0.1M NaOH prior to analysis.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-U-109	PNNL-11908	FY98	Lab scale caustic plus oxidant leaching.	—	SOWRT Group VI. Primary waste type is EB, with secondary waste type being CW.  Non-Complexed Waste. <sup>1</sup>  <u>Primary:</u> EB. <u>Secondary:</u> CW. <u>Tertiary:</u> high-level REDOX waste. <sup>5</sup>	—	Add 0.01 M NaOH to 50 g sludge to a total volume of 250 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01 M NaOH to residual solids to a total volume of 150 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01 M NaOH to residual solids to total volume of 150 mL. Mix for 1 hr at RT. Centrifuge and discard supernate. Add 0.01M NaOH to residual solids to total volume of 50 mL. Transfer 2 mL and dry at 105°C to obtain dry insoluble solids weight (0.3163 g). Transfer ~7 mL portions to 6 bottles for further testing.	—	75100 ug Al/g dry solids.	—	—	—	<b>Six oxidative leach tests performed:</b> 1) Add solid NaMnO <sub>4</sub> to sludge suspended in 0.1M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 2) Add solid NaMnO <sub>4</sub> to sludge suspended in 3M NaOH to obtain 1:1 MnO <sub>4</sub> to Cr stoichiometry. 3) Contact sludge, suspended in 0.1M NaOH, with O <sub>2</sub> . 4) Contact sludge, suspended in 3 M NaOH, with excess O <sub>2</sub> . 5) Bubble Ar gas through sludge suspended in 0.1M NaOH. 6) Bubble Ar gas through sludge suspended in 3M NaOH. Samples were stirred at RT for 24 hr, then heat to 80°C for remainder of experiment. DI was added to replenish evaporated water. Test samples taken at time 0 hr, with intermittent samples taken throughout the remainder of the experiment. Test slurries were centrifuged and supernate decanted after tests completed.
241-U-110	PNL-9387	FY93	Lab scale caustic leaching	Slurry of 2.038 g sample + 5.306 g 0.1 M NaOH	Non-Complexed Waste. <sup>1</sup> <u>Primary:</u> 1C. <u>Secondary:</u> CW. <sup>2</sup> <u>Primary:</u> 1C. <u>Secondary:</u> CW. <u>Tertiary:</u> high-level REDOX waste. <u>Other:</u> Laboratory waste from 222-S building. <sup>5</sup>	—	—	1.8 g Al/g sludge.	—	—	—	—	Mix 2.038 g dry tank sludge with 5.306 g 0.1M NaOH, using 1.189 g for leach procedure. Add 0.5 mL water & 1.5 mL 10M NaOH; stir & heat for 1 hr at 100°C; cool; centrifuge; decant; add 3mL 5M NaOH; stir & heat 5.5 hr at 100°C; cool; centrifuge; and decant.
241-U-110	PNL-10078	FY94	Lab scale caustic leaching	—	Neutralized 1C waste. Other wastes include REDOX process HLW, CW, and lab waste from the 222-S building (waste has high Al content).  Non-Complexed Waste. <sup>1</sup>	Core 14	Add 2mL 0.1M NaOH to 0.57 g dry sludge, mix at RT for 1 hr, centrifuge, decant; add 2mL water to solids, mix at RT for 0.5hr, centrifuge, and decant.	~20 wt%; 2.1E-1 g Al/g dry sludge	—	1%	—	Dissolution of solids using nitric acid done after final wash following second leach.	Add 4mL 3M NaOH/2M Na <sub>2</sub> CO <sub>3</sub> to solids, mix at 100°C for 5 hr, centrifuge, and decant.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
241-U-110	PNNL-11779	FY97	Lab scale caustic leaching	—	Non-Complexed Waste. <sup>1</sup>	Composite of material taken from seven different core samplings of the tank.	Thorough wash using 0.01M NaOH to remove any water-soluble components. A sample was dried to constant weight at 105°C for analysis.	—	273000 ug Al/g dry sludge solids.	—	—	—	Using 12.858 g slurry (1.12 g solids), add 10 mL 10M NaOH and 5 mL water. Mix at 100°C for 85 hr. Cool, centrifuge, and decant.
Group 1 Bismuth Phosphate Sludge (1C and 2C)	PNNL-17992 (WTP-RPT-166)	N/A 2006-2009	—	As-received sample colors: dry white crystals, grey pastes, brownish yellow sludge, yellow supernate.	Composite of 31 samples. 93% from B-104, 4% from BX-112, and 3% from T-104. Solids passed through 3.2 mm sieve. Additional 742.97 g DI added during compositing.	—	Wash 3 times by mixing 60mL aliquots of 0.01M NaOH with solids for 15 minutes, centrifuging at ~1200G for 15 min, and decanting supernate between subsequent washes. After each wash step, the centrifuged solids were stratified in three layers: white dense layer, darker middle layer, and light brown or tan top layer.	—	By KOH Fusion: 26350 ug Al/g. By Acid Digestion: 28450 ug Al/g.	By Acid Digestion: Supernatant = <3.77 ug/mL. Wash Composite <3.75 ug/mL.	—	Washed solids subdivided into eight ~9.9 g washed samples for caustic leaching (~0.95 g undissolved solids). Washed and centrifuged solids mixed with 60 mL DI (10g solids in 87 g slurry or 11.5 wt% undissolved solids).	A total of 8 samples were leached at different free hydroxide concentrations (1 to 3 M NaOH) and temperatures (40, 60 and 80°C). Solution samples were obtained at 0, 1, 2, 4, 8, and 24 hours, with obtained leachate filtered through a 0.45 um syringe filter. After final samples removed, the slurries were removed from heat and allowed to cool to ambient temperature (~22°C). Slurries were centrifuged and half the leachate was decanted.
Group 2 Bismuth Phosphate Saltcake (BY and T saltcake)	PNNL-17992 (WTP-RPT-166)	N/A 2006-2009	—	—	Composite of 41 samples from 13 tanks. 7.1% from BX-110, 11.6% from BX-111, 5.9% from BY-104, 4% from BY-105, 10.4% from BY-107, 24.9% from BY-108, 11% from BY-109, 8.2% from BY-110, 2.4% from BY-112, 1% from T-108, 1.8% from T-109, 4% from TX-104, and 7.6% from TX-113. Total of 1966 g solids passed through 3.2 mm sieve. Additional 2973.6 g of water added. Homogenized sample settled over the weekend to allow separation of settled solids for sampling.	—	Remove a 34.5 g aliquot of homogenized slurry and transfer to 200 mL centrifuge bottle (0.29 g dry water-insoluble solids/g slurry). Centrifuge at ~1200G for 15 min. Wash 3 times by mixing 45mL aliquots of 0.01M NaOH with solids for 15 minutes with an overhead mixer, centrifuge at ~1200G for 15 min, and decanting supernate between subsequent washes.	—	By KOH Fusion: 112500 ug/g. By Acid Digestion: 122500 ug/g.	By Acid Digestion: Supernatant = 2030 ug/mL. Wash Composite 322 ug/mL.	13.1% in supernate, 1.1% in wash solution, and 85.8% remained with solids.	Add 100 mL DI to washed solids and subdivide into seven ~14 g washed samples for caustic leaching (~1 g undissolved solids). Average Al in solids prior to leaching = 149500 ug/g.	A total of 7 samples were leached at different free hydroxide concentrations (1 to 5 M NaOH) and temperatures (60, 80, and 100°C). Solution samples were obtained at 0, 1, 2, 4, 8, and 24 hours, with obtained leachate filtered through a 0.45 um syringe filter. After final samples removed, the slurries were removed from heat and allowed to cool to ambient temperature (~22°C). Slurries were centrifuged and half the leachate was decanted.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
Group 3 PUREX Cladding Waste Sludge	PNNL-18054 (WTP-RPT-167)	N/A 2006- 2009	1:100 solids to solution ratio caustic leaching at various T, [OH], and [NaNO <sub>3</sub> ] with periodic sampling from 0 to 48 hours.	Dark and viscous consistency	15 samples from 6 tanks: 241-C-103 (14.06 g, 3%), 241-B-109 (54.09 g, 11%), 241-C-105 (72.09 g, 14%), 241-B-108 (57.39 g, 11%), 241-C-104 (52.47 g, 10%), 241-BY-109 (259.14 g, 51%). Added Water = 438 g.	222-S IDs available: 6440, 11486, 9765, 19798, 11505, 11506, 11507, 11244, 11245, 11356, 11366, 13043, 13044, 19098, and 19134.	Composite sieved through stainless steel strainer and mixed at 29°C for one hr using overhead mixer to homogenize. Wash 3 times using approximately 17.4 mL 0.01M NaOH. Centrifuge and decant supernate to composite wash collection.	98.1 wt% Al. In initial slurry there were 311500 ug Al/g solids and 2035 ug Al/mL aqueous phase.	297500 ug/g (Fusion), 325500 ug/g (Acid Digest). Water washed insoluble solids were ~30 wt% Al.	Supernate = 2035 ug/mL. Wash composite = 136 ug/mL.	—	Wash prior to caustic leach: Centrifuge slurry sample and remove supernate to get ~15 mL solids. Add ~45 mL 0.01M NaOH and shake on vortex mixer for 15 minutes. Centrifuge for 30 min and remove supernate. Repeat for a total of 3 washes, centrifuging the last washed solids for only 20 minutes.	Add 74.6 mL DI to solids to obtain slurry (13.2 g solids in 103.8 g slurry) 14.1 wt% undissolved solids. Mix and transfer ~7.1 g slurry to thirteen 125 mL high density polyethylene bottles, each with ~1 g undissolved solids. Leaching was performed at different free OH concentrations (1, 3, and 5 M), T (60, 80, and 100°C), and NaNO <sub>3</sub> concentrations (1 and 5 M). Additional DI was added to bring the total volume to 100 mL. Samples were taken at times of 0, 1, 2, 4, 8, 24, and 48 hours, allowing the solids to settle for 5 to 10 minutes and filtering through a 0.45 um syringe filter prior to sampling.
Group 4 REDOX Cladding Waste Sludge	PNNL-18054 (WTP-RPT-167)	N/A 2006- 2009	1:100 solids to solution ratio caustic leaching at various T, [OH], and [NaNO <sub>3</sub> ] with periodic sampling from 0 to 48 hours.	Dry white powders, gray solids, and dark tar-like sludge samples with yellow supernate liquid on some samples	14 samples from 5 tanks: 241-U-105 (191.68g, 34%), 241-U-201 (75.04 g, 13%), 241-U-202 (185.37 g, 32%), 241-U-203 (24.24 g, 4%), and 241-U-204 (95.69 g, 17%). Added Water = 94 g.	222-S IDs available: 19476, 9702, 9711, 16961, 6882, 13462, 19154, 6911, 6916, 13486, 15011, 19169, 13072, and 15020.	Composite sieved through stainless steel strainer and mixed at 29°C for one hr using overhead mixer to homogenize. Wash 3 times using between 10.2 and 14.2 mL 0.01M NaOH. Centrifuge and decant supernate to composite wash collection. 2 samples washed = 67.5 mL wash collection.	In initial slurry there were 320000 ug Al/g solids and 505 ug Al/mL aqueous phase.	296500 ug/g (Fusion), 343500 ug/g (Acid Digest). Water washed insoluble solids were made up of ~32 wt% Al. Solids had 99.6 wt% of Al.	Supernate = 505 ug/mL (0.29 wt%). Wash composite = 60.2 ug/mL (0.13 wt%).	—	Wash prior to caustic leach: Centrifuge slurry sample and remove supernate to get ~15 mL solids. Add ~45 mL 0.01M NaOH and shake on vortex mixer for 15 minutes. Centrifuge for 30 min and remove supernate. Repeat for a total of 3 washes, centrifuging the last washed solids for only 20 minutes.	Add 68.5 mL DI to solids to obtain slurry (14 g solids in 116.5 g slurry) 12 wt% undissolved solids. Mix and transfer ~8 g slurry to thirteen 125 mL high density polyethylene bottles, each with ~1 g undissolved solids. Leaching was performed at different free OH concentrations (1, 3, and 5 M), T (60, 80, and 100°C), and NaNO <sub>3</sub> concentrations (1 and 5 M). Additional DI was added to bring the total volume to 100 mL. Samples were taken at times of 0, 1, 2, 4, 8, 24, and 48 hours, allowing the solids to settle for 5 to 10 minutes and filtering through a 0.45 um syringe filter prior to sampling.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
Group 5 REDOX	PNNL-17368 (WTP-RPT-157)	N/A 2006-2008	Caustic leach of homogenized sample	gray solids with yellow supernate	1283 g from 241-S-101, 397 g from 241-S-110, 213 g from 241-S-107, and 78 g from 241-SX-103. Composite of 39 different archived samples. Solids passed through 1/8 inch stainless steel sieve, with ~ 4 L DI added to solids to homogenize.	—	Wash 3 times with ~ 10 mL 0.01 M NaOH, centrifuge and decant supernate between subsequent washes.	388250 ug/g	326500 ug/g (97%)	2595 ug/mL (3%) in supernatant and 704 ug/mL (0.02%) in wash composite.	—	—	A total of 13 samples were leached at different free OH concentrations (1 to 5 M NaOH), T (80, 90, and 100°C), and NaNO <sub>3</sub> concentrations (1 to 5 M NaNO <sub>3</sub> ). Solution samples were obtained at 1, 4, 8, 24, 72, and 170 hours.
Group 6 S-Saltcake	PNNL-17368 (WTP-RPT-157)	N/A 2006-2008	Caustic leach of homogenized sample.	dark brown settled solids, bright yellow supernate.	965.3 g from 241-SX-106, 227.3 g from 241-SY-103, 151.7 g from 241-SX-105, 131.5 g from 241-SX-102, 76.2 g from 241-S-111, 55.1 g from 241-U-108, 53.4 g from 241-U-103, 30.3 g from 241-S-106. Composite of 28 different archived samples. Solids passed through 1/8 inch stainless steel sieve to remove large particles (- 5.7 g). Added DI to make total volume ~3 L.	—	Wash 3 times with ~ 5 mL 0.01 M NaOH, centrifuge and decant supernate between subsequent washes.	—	187000 ug/g (60%)	7590 ug/mL (38%) in supernatant and 1580 ug/mL (2%) in wash composite	—	Initial washing of solids prior to caustic leaching: With ~35 mL centrifuged solids, add ~105 mL 0.01M NaOH. Mix 5 minutes by shaking. Centrifuge at ~610 G for 15 min and decant supernate. Repeat for a total of 3 washes.	Add 100 mL 3M NaOH to washed solids and allow leaching at ~100°C for 8 hours. Solids were suspended using a magnetic stir bar. After 8 hours, removed slurry from heat and cooled to ambient temperatures (overnight). Centrifuge slurry at ~610 G for 15 min and remove leachate.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
Group 7 TBP Waste Sludge	PNNL-18119 (WTP-RPT-169)	N/A 2007-2009	—	Centrifuged solids were dark brown-red with pale yellow supernate.	Composite of 33 samples from 2 tanks. 92% (30 samples) from BX-109 and 8% (3 samples) from B-106. Total of 1479.16 g from original samples transferred to composite. A total of 779.7 g DI added during compositing. This was passed through a stainless steel strainer to ensure no large pieces of foreign material in composite.	222-S sample IDs from 241-B-106: 7417, 7424, and 16913. 222-S sample IDs from 241-BX-109: 6907, 6921, 6922, 6927, 6930, 6931, 6932, 6933, 6934, 6935, 7153, 7154, 7157, 7158, 7372, 7378, 9334, 9346, 10116, 11840, 13092, 13445, 13473, 13515, 13516, 13517, 13522, 13523, 16916, and 19302.	Wash two samples 3 times with 20 to 22 mL 0.01 M NaOH. Centrifuge and decant supernate between subsequent washes.	—	16000 ug/g (Fusion), 18550 ug/g (Acid Digest). Water washed insoluble solids were made up of ~ 97 wt% of the total Al.	Supernate = <0.73 ug/mL (<0.01 wt%) Al. Wash 1 = 8.96 ug/mL Al. Wash 2 = 28 ug/mL Al. Wash 3 (filtered) = 46.3 ug/mL Al. Combined wash solution contained 3.4 wt % of the total Al.	—	Wash prior to caustic leach: With 98.4 g sample (12.1 g insoluble solids), centrifuge 15 min and remove supernate to get ~15 mL solids. Add ~45 mL 0.01M NaOH and shake for 15 min. Centrifuge for 15 min and remove supernate. Repeat for a total of 3 washes, centrifuging the last washed solids for 65 minutes, adding 0.5mL 19M NaOH and centrifuging an additional 15 min to remove floating solids. Sample bottle was knocked over after mixing ~10 min, losing 2/3 of sample. Therefore, an additional 73.3 g aliquot was washed in a similar manner as described above to make up for the loss.	Additional DI water was added to thin samples (~84 g DI). A total of 10 samples (~9.4 g slurry in 125 mL HDPE bottles with ~1 g UDS) were leached at different free OH <sup>-</sup> concentrations (0.25, 1, and 3 M NaOH) and T (40, 60, and 80°C). Solution samples were obtained at 1, 4, 8, and 24 hours, allowing the samples to settle for ~5 to 10 min before sampling.



Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
Group 8 FeCN Waste Sludge	PNNL-18120 (WTP-RPT-170)	N/A 2007-2009	Cell Unit Filtration, caustic leaching and wash of Group 8 archived solids.	Solids ranged from gray dry crystals, gray pastes, white solids, and brown sludge. Aqueous phase was yellowish.	Composite of 30 samples from 5 tanks. 45% from BY-110, 22% from BY-108, 21% from BY-105, 10% from BY-104, and 2% from BY-106. Total of 1358.8 g from original samples transferred to composite. A total of ~960 g DI added during compositing. This was passed through a sieve (collect objects > 3mm diameter) to ensure no large pieces of foreign material in composite. No significant sample was trapped in sieve.	—	Samples were composited and sieved through a 3mm sieve. An additional ~960 g DI was added during compositing, with the solution mixed 1hr 5min before sub-sampling. Wash two samples 3 times with 6.6 to 9.1 mL 0.01 M NaOH. Centrifuge and decant supernate between subsequent washes and composite the decanted wash solution (46.8 mL total).	—	88350 ug/g (Fusion), 90950 ug/g (Acid Digest). Water washed insoluble solids were made up of 9 wt% Al, with 89.4 wt% of the total Al remaining after wash.	Supernate (acid digest) = 1430 ug/mL (8.5 wt%) Al. Composite wash (acid digest) = 159 ug/mL (2.1 wt%) Al.	—	High-Solids slurry composition (After filtration using acid digestion): Dry solids = 39000 ug Al/g. Supernate = 1470 ug Al/mL. Dry solids = 85000 ug Al/g.	Caustic leach after filtration to 13 wt% undissolved solids. Slurry had an initial mass of 3.9 kg, undissolved solids concentration of 230 g, and initial volume of 3.1 L. Caustic was added to the tank prior to beginning of heat and mix. Heating of slurry from 26 to 60°C took 2.5 hr. Leaching was performed at 60°C for 8 hr in 3.7M OH, with samples taken at times - 1.5 hr, 0 hr, 2 hr, 4 hr, 8 hr, and final.
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant	PNNL-18007 (WTP-RPT-171)	N/A 2007-2009	Only initial caustic leach of mixed Group 5 and 6 composite		Group 5 solids contained 33 wt% Al, with ~90 wt% Al in the form of boehmite. The individual tank waste material sources, compositions, and physical properties are described in PNNL-17368 (WTP-RPT-157) above.	—	—	Group 5 supernate had 2595 ug Al/mL with 326500 ug Al/g washed solids on a dry mass basis. Group 6 supernate had 7590 ug Al/g with 187000 ug Al/g washed solids on a dry mass basis. Al in initial solids = 312500 ug/g. Al in initial supernate = 5246 ug/mL.	—	—	—	From total slurry, 16 g was removed for analysis, with the remainder centrifuged. The supernate (106 g) was also removed.	The remaining concentrated sludge (~18 wt% undissolved solids) was combined with 84 mL of 9.5M NaOH, stirred and heated to ~100°C for 12 hr. Water was added to account for evaporation. After leaching, the solution was cooled to ambient temperature.

Table A1-1. SSTs and DSTs Wash and Leach Information for Aluminum, Part 1. (38 sheets)

Tank	Lab Study Reference(s)	Year of Sampling Event	Test type	Sample color	Waste Sources	Sample ID	NaOH Wash Procedure	Initial Al in Untreated Solids	Final Al in Washed solids	Al measured in Wash Solution	% recovery <sup>2</sup>	comments	Caustic Leach #1 Procedure
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant	PNNL-17965 (WTP-RPT-172)	N/A 2007-2009	Section 5: Group 6/5 Aluminum Leaching Studies	—	<u>Group 5 Caustic Leach Test:</u> 1.8 L Group 5 composite (18 wt% undissolved solids) diluted with 2.1 L simulant supernate solution based upon permeate composition reported in PNNL-17368 (WTP-RPT-157). <u>Group 5 + Group 6 Caustic Leach Test:</u> 1157 g Group 5 slurry + 613 g Group 6 slurry.	—	The group 5+6 composite was mixed and a 62.4 g sample was transferred to a 200 mL bottle, centrifuged for 15 min, and decanted. Volume centrifuged solids ~10 mL. Approximately 30 mL 0.01M NaOH was added to wash the solids. This was mixed for 15 min, centrifuged for 15 min, and decanted. This was repeated for a total of 3 washes. After being washed, an additional 75.6 mL DI was added to obtain ~91.4 g slurry.	—	—	—	—	—	Four ~21 g slurry samples were transferred to 125 mL bottles. These were caustic leached at 100°C with the following M free OH concentrations: #1 = 3.18M, #2 = 5.44M, #3 = 4.86M, and #4 = 7.34M OH. Total volume was adjusted to 100 mL using DI. Sample #3 was knocked over. Samples were taken at times of 0, 1, 2, 4, 8, and 24 hrs.
Mixture of Group 3 and Group 4 composites	PNNL-18048 (WTP-RPT-181)	N/A 2007-2009	Caustic leaching /washing after cell unit filtration.	—	490 mL Group 3 composite (598 g at 28.8 wt% UDS) mixed with 540 mL Group 4 composite (710 g at 29 wt% UDS) with 2.88 L simulant supernate added to dilute blended slurry to 9 wt% UDS. Filtered solids were subsequently caustic leached.	—	—	122,200 mg Al Total. 54,500 mg Al from Group 3 and 67,700 mg Al from Group 4 waste.	—	—	Solids estimated at 94 g Al in 390 g undissolved solids.	—	Initial caustic leach slurry: Initial mass = 4.7 kg with 300 g undissolved solids and initial volume of 3.5L and final free hydroxide concentration of 5.3M. Leaching was performed at 100°C (heat up took 5.3 hours) for 8 hours and cooled to ambient temperature over 12 hours. Samples were taken at 0, 4, and 8 hours.

**References:** <sup>1</sup> WHC-EP-0625. <sup>2</sup> PNNL-11636. <sup>3</sup> PNNL-13394 (NP = measurement not performed). <sup>4</sup> PNWD-3300 (WTP-RPT-076). <sup>5</sup> PNL-9814.



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
DSTs	—	—	—	—	—	—	—	—	—
241-AN-104	PNNL-11636	Add 7.5 mL of 10 M NaOH to solids + 16.5 ml water; mix 5 hr at 100°C; cool; centrifuge; decant.	Add 10 mL of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to remaining solids; mix 0.5 hr at RT; centrifuge; decant; repeat for total of 3 wash steps. After 3rd wash, remove two 0.2 g slurry samples before centrifuge & decant.	156,816 (96%)	5,606 (3%)	406 (0%)	500 (0%)	163,327 (100% removal based upon iron normalized procedure; 154% mass recovery)	—
241-AZ-101	PNWD-3206 (WTP-RPT-043)	—	Following leaching, rinse three times with 1200 g of 0.01M NaOH at 25°C and then dewater. Repeat.	70% Al removed in wash, leach, and rinse. In slurry after leach: 79850 ug Al/g slurry. In leach permeate: 14600 ug Al/mL.	—	In slurry after rinse: 100,075 ug Al/g slurry. Rinse 1 permeate: 7,900 ug Al/mL. Rinse 2 permeate: 3,990 ug Al/mL. Rinse 3 permeate: 2,060 ug Al/mL.	25% Al in residual	99% Al recovery	—
241-AZ-101 + 241-AZ-102	PNNL-11580	Wash #1: to solids, add 152.3 ml of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> , mix 0.5 hr at RT, measure settling, and decant. Wash #2: to solids, add 152.7 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> , mix 0.5 hr at RT, measure settling, and decant. Wash #3: to solids, add 150.4 ml 0.01 M NaOH/0.01 M NaNO <sub>2</sub> , mix 0.5 hr at RT, sample slurry (amounts not recorded), measure settling, and decant.	Through caustic leach #5: 62% Al removed (71% corrected value).	Caustic leach #6: 14% Al removed (8 % corrected value).	4% Al (0% corrected value)	21% Al in dried residue (21% corrected value)	0.0604 g Al/g dried sludge (113 % mass recovery)	—	1st caustic leach: 222.2 ug Al/mL leach solution. 86.8 mL leach solution. 2nd caustic leach: 5797 ug Al/ml leach solution. 80.6 mL leach solution. 3rd caustic leach: 2343 ug Al/mL leach solution. 189.2 mL leach solution. 4th caustic leach: 2580 ug Al/mL leach solution. 189 mL leach solution. 5th caustic leach: 2769 ug Al/mL leach solution. 187.9 mL leach solution. 6th caustic leach: 864.5 ug Al/mL leach solution. 184.5 mL leach solution. Final Wash Solution: 76.89 ug Al/mL leach solution. 460.5 mL leach solution. Dried Residue: 30325 ug Al/g dried residue.
241-AZ-102	75764-PCS95-086	—	—	—	—	—	—	—	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-AZ-102	PNWD-3045 (BNFL-RPT-038)	—	Following leaching, rinse twice with 0.01M NaOH (1,390g and 1,400 g for leach 1 and 2) for 8 hr at 85°C and then dewater. Repeat.	1.04E+4 ug Al/mL. Leach efficiency = 59.3%.	Intermediate sample: 1.01E+5 ug Al/g dry solids	Wash permeate #1: 4.91E+3 ug Al/mL, 1.9% efficiency. Wash permeate #2: 2.18E+3 ug Al/mL, <0.003% efficiency.	1.07E+5 ug Al/g dry solids. 36.3% Al in residue.	120% Al recovery	—
241-SY-102	PNWD-3512 (WTP-RPT-117)	3 M NaOH leach of samples SY-102-5 and SY-102-6 (slurry weights of 15.46 and 16.174 g, respectively). Add 4.8 ml 10M NaOH and 8.2 ml DI to each. Heat at 85 ± 5°C for 8 hr. Cool to RT overnight. Centrifuge and decant supernate to 30 mL plastic vials. Add 0.1M NaOH at 3:1 vol:vol ratio to remaining solids. Mix, separate, and decant to same container as initial leachate solution. Continue washings until supernate was colorless. Filter samples (0.2 um syringe filter), and place 1 ml aliquot in container w/ 10 mL of 1M HNO <sub>3</sub> for ICP-AES.	Tank #/Pre NaOH Leach/Post NaOH Leach/Oxidative Leach M [NaOH] <sub>initial</sub> /Oxidative Leach T, °C/ Oxidative Leach M [NaMnO <sub>4</sub> ] <sub>initial</sub> /[Cr]: Tank#1/No/No/3/85/1.1. Tank#2/No/No/5/85/5. Tank#3/Yes/No/0.25/25/1.1. Tank#4/Yes/No/0.25/85/1.1. Tank#5/No/Yes/0.25/25/1.1. Tank#6/No/Yes/0.25/85/1.1.	—	—	no data	no data	% removal values for 6 bottles range from 76 - 94% Al removal (86, 88, 76, 78, 94, 89 % removal for bottles #1-6, respectively)	[Sample ID, Core #, Jar #, Phase, grams Added]; [S03T001382, 286, 19506, S, 44.7]; [S03T001386, 286, 19561, SL, 48.1]; [S03T001376, 284, 19606, S, 25.8]; [S03T001377, 284, 19607, S, 45.9]; [S03T001375, 284, 19411, S, 13.0]; [S03T001406, 286, 18982, S, 6.9]; [S03T001381, 286, 18714, S, 9.0]; [S03T001380, 286, 18599, S, 34.1]; [S03T001374, 284, 18586, S, 34.1]; [S03T001379, 284, 18528, L, 135.5]; [S03T001385, 286, 19203, L, 142.8]; [S03T001378, 284, 19213, L, 171.4]; [S03T001405, 284, 19538, L, 173.4]; [S03T001384, 286, 19209, L, 166.3]; [S03T001404, 286, 19211, L, 168.6]. Here, S = Solid, SL = Slurry, and L = Liquid phase.
241-SY-103	PNL-10712	Solids did not settle. Add 4.1 g 3M NaOH to 1.1 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 3.5 g of 1 M NaOH/1 M NaNO <sub>2</sub> to 1.3 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 3.3g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 3.5g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	48% Al found, 43350 ug Al/mL measured.	25% Al found, 15300 ug Al/mL measured.	7% Al found, 1615 ug Al/mL measured.	Measured an Al concentration of 83,000 ug/g; 10% Al in residue.	Direct analysis: 4.7E-02 g Al/g sludge; Summation method: 3.84E-02 g Al/g sludge; 82% Al recovery.	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-SY-103	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 2.8M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	uncorrected/ corrected recovery = 48/78% Al.	Uncorrected/ corrected Al recovery = 25/2 %.	Uncorrected/ corrected Al recovery = 7/1%.	Total wt% Al = 4.7; Total % Al removed = 90%; percent undissolved Al in residue = 10%.	—	—
SSTs	—	—	—	—	—	—	—	—	—
241-B-101	PNNL-12026	After leach test, remove vessel from heating block and cool to RT; centrifuge 15 min; decant; wash solids 3 times with 10 mL 0.01 M NaOH/NaNO <sub>2</sub> ; dry at 105°C.	—	At 60°C: 1M NaOH removed 42% Al (Fe normalized = 43% Al); 3M NaOH removed 51% Al (Fe normalized = 51% Al). At 100°C: 1 M NaOH removed 46% Al (Fe normalized = 49% Al); 3M NaOH removed 50% Al (Fe normalized = 43% Al).	—	—	—	—	—
241-B-104	LAUR 96-2839	Add 25.2 mL of 10M NaOH to solids and ~68.1 ml water to get ~1 wt% solids and final NaOH of ~3M; 11.2 mL settled solids/ 4.8 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 18.2 mL inhibited water to solids (12 mL settled solids/ 3.7 mL centrifuged solids); mix 0.5 hr at RT; measure settling; centrifuge; decant. Repeat 2 times with solids portion, sampling slurry (0.64 mL) before measuring settling in wash #3. Dry solids at 105°C.	5.27 ug Al/mL; 73.62 ug Al (0.5% Al removed).	97.79 ug Al/mL; 8045.17 ug Al (56.4% Al removed).	15.45 ug Al/mL; 808.5 ug Al (5.7% Al removed).	10210.13 ug Al/g; 5327.64 ug Al (37.4 % Al removed); 63% [Table S.1] Al removed during sludge wash/caustic leach).	Direct analysis: 2.47E-3 g Al/g sludge; Summation method: 2.53E-3 g Al/g sludge. 97.7% Al recovery.	—
241-B-106	LAUR 97-2889	Add 43.1 mL water and 18.9 mL 10M NaOH to get ~1 wt% solids & ~3M NaOH. Mix 5 hr at 100°C; cool; measure settling; centrifuge; decant. Supernate: 59.88 mL with 3.26 M free OH <sup>-</sup> , 9.58 mL settled solids, & 4.11 mL centrifuged solids.	Wash 3 times: With solids, add 13.22 mL inhibited water; mix 0.5 hr at RT; measure settling; centrifuge; decant. Sample slurry (0.6 g) after 3rd mix. Dry solids at 105°C after 3rd decant to obtain 0.5 g solids.	744.9 ug Al/mL, 19426.99 ug Al (74% Al removed).	49.4 ug Al/mL, 2958.07 ug Al (11.3% Al removed).	5.5 ug Al/mL, 215.88 ug Al (0.8% Al removed).	7304.117 ug Al/g, 3652.06 ug Al (13.9% Al removed).	Summation: 7.45E-3 g Al/g; Direct: 7.29E-3 g Al/g; 102.1% Al mass recovery; 26253 ug Al; 86% Al removed.	—
241-B-110	PNL-9387	ACID LEACH: Add 3 mL 2M HNO <sub>3</sub> & 0.2 mL 10M HF to solids; mix 1 hr at 100°C; analyze solution - all solids dissolved.	—	—	Al in caustic leach was less than or equal to 26%.	—	Leached sludge had greater than or equal to 47% Al.	—	—
241-B-110	Internal Letter 9404238	Add 11.8 mL water and 5 mL of 10M NaOH to solids. Mix, using a magnetic stirrer, for 5 hr at 100°C. Cool, then settle and decant liquid.	Wash 5 times with water for 0.5 hr at RT (12 mL; 15 mL; 13 mL; 13 mL; and 12 mL). Settle solids and decant liquid portion between washes.	—	Total of 18-19% Al removed during caustic leach and final wash steps.	—	—	—	—

**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

<b>Tank</b>	<b>Lab Study Reference(s)</b>	<b>Caustic Leach #2 Procedure</b>	<b>Wash of solids after Caustic leach steps</b>	<b>Mass (ug) Al removed during 1st leach step</b>	<b>Mass (ug) Al removed during 2nd leach step</b>	<b>Mass (ug) Al removed in final wash solution</b>	<b>Mass (ug) Al in Leached Solids</b>	<b>Total mass Al (ug)</b>	<b>Comments</b>
241-B-111	PNL-10712	Add 1.44 g of 3M NaOH to 5.2 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Solids did not settle. Add 5 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 5.2 mL centrifuged solids; mix 5 hr at RT; measure settling; centrifuge; decant; and repeat. Add 4.9 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	99% Al removed (1% Al found, 17.85 ug Al/mL).	Remaining 1% Al (16 ug Al/mL detected).	99% Al removed (measured 8.5 ug Al/mL).	Measured concentration of 18000 ug Al/g.	Direct analysis: 3E-03 g Al/g sludge; Summation method: 4.11E-03 g Al/g sludge; 137% Al recovered.	—
241-B-111	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 4.3M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	1% Al removed.	—	1% Al removed.	Total wt% Al = 0.3; Total % Al removed = 2%; 98% Al undissolved.	—	—
241-B-201	PNL-10078	<b>Dry cores:</b> Add 5mL of 1M K <sub>2</sub> CO <sub>3</sub> to solids, mix at 100°C for 5 hr, centrifuge, decant. Add 5mL 1M K <sub>2</sub> CO <sub>3</sub> to solids, mix at 100°C for 5 hr, centrifuge, decant. <b>Wet core 26:</b> Add 1mL 1M K <sub>2</sub> CO <sub>3</sub> to solids, mix at 100°C for 5 hr, centrifuge, decant and repeat.	Add 5 mL water to solids, mix at RT for 0.5 hr, centrifuge, decant; dry at 80°C.	<b>Core 26:</b> 16% Al dissolved. <b>Core 27:</b> 38-39% Al dissolved.	<b>Core 26:</b> 0-1% Al dissolved. <b>Core 27:</b> 5% Al dissolved.	—	<b>Core 26:</b> 82-84% Al dissolved. <b>Core 27:</b> 55-56% Al dissolved.	<b>Core 26 composite:</b> direct analysis measured 8.2E-3 g Al/g dry sludge; summation showed 7.6E-3 to 7.7E-3 g Al/g dry sludge. <b>Core 27 composite:</b> direct analysis measured 6.5E-3 g Al/g dry sludge; summation showed 4.0E-3 g Al/g dry sludge.	—
241-B-202	LAUR 95-2070	Add 2.5mL 3M NaOH to solids, mix at 100°C for 5 hr, cool, measure settling, centrifuge, decant (Vol. settled solids ~6.5mL; Vol. centrifuged solids ~4.6mL).	Wash 3 times by adding 11.5 mL inhibited water to solids and mix at RT for 0.5 hr, measure settling, centrifuge and decant. For wash #3, sample slurry (0.9mL) after mix phase. Dry solids at 80°C.	102 ug Al/mL (5.24% Al removed).	119 ug Al/mL (5.47% Al removed).	8.5 ug Al/mL (5.48% Al removed).	3874 ug Al/g sludge (80.92% Al remaining).	Direct Analysis: 2.6E-3 g Al/g sludge; Summation: 2.4E-3 g Al/g sludge (92.14% Al recovery).	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-BX-103	LAUR 97-2889	Add 155.2 mL water & 67.5 mL 10M NaOH to achieve 1.7 wt% solids & ~3M NaOH. Mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant. Supernate: 212.44 mL with 3.17 M free OH, 5.2 mL settled solids, 4 mL centrifuged solids	Wash 3 times by adding 72 mL inhibited water to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Subsample (0.61 g) after mix of wash #3. Dry solids at 105°C. (0.855 g solids & 212.44 mL supernate).	17082 ug Al/mL, 1136294.6 ug Al (93.12% Al removed).	365.3 ug Al/mL, 77604.33 ug Al (6.36% Al removed).	11.66 ug Al/mL, 2471.69 ug Al (0.2% Al removed).	4542.526 ug Al/g, 3882.04 ug Al (0.32% Al remaining).	Concentration in initial solids: Summation = 2.33E-1 g Al/g; Direct analysis = 2.1E-1 g Al/g. 110.91% Al mass recovery. 1220252.7 ug Al.	—
241-BX-105	LAUR 95-2070	Add 47.5 mL of 3M NaOH, mix 5 hr at 100°C, cool, measure settling, centrifuge, and decant. Volume settled solids <1 mL; Volume centrifuged solids <1 mL, 47.16 mL liquid from decant.	Wash 3 times by adding 50.5 mL inhibited water to solids and mix at RT for 0.5 hr, measure settling, centrifuge, and decant. For wash #3, sample slurry (0.3 mL) after mix phase. Dry solids at 80°C (0.0352 g).	<u>Found in retrieval wash prior to leach:</u> 255 ug/mL (1.64 %); Residual = 35 mg Al/g sludge (0.04%). <u>From Leach #1:</u> 48500 ug Al/mL (61.02% Al removed).	23800 ug Al/mL (35.21% Al removed).	442 ug Al/mL (2.09% Al removed).	34632 ug Al/g sludge.	Direct analysis: 3.05E-01 g Al/g sludge; Summation method: 3.41E-01 g Al/g sludge; 111.71% Al recovery.	—
241-BX-107	PNL-10712	Add 10.5g of 3 M NaOH to 6.1 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 10.9 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 4.0 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 11.6 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 11.7 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	49% Al, 7225 ug Al/mL.	11% Al, 2720 ug Al/mL.	7% Al, 272 ug Al/mL.	41000 ug Al/g, 32% Al in residue.	Direct analysis: 3.5E-02 g Al/g sludge; Summation method: 2.83E-02 g Al/g sludge; 81% Al recovery.	—
241-BX-107	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 3.6M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat two more times.	49% Al uncorrected; 75% Al corrected value.	11% Al uncorrected; -5% Al corrected value.	7% Al uncorrected; -2% Al corrected value.	Total wt% Al = 3.5; Total % Al removed = 68%; Al in undissolved residue = 32% (31% corrected value).	—	—

**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-BX-109	Laur 96-2839	Add 11 mL 10M NaOH and ~23ml water to solids to get ~1 wt% solids and final NaOH of ~3M (6.2 mL settled solids; 1.5 mL centrifuged solids). Mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 7.1 mL inhibited water to solids (3.9 mL settled solids/1.6 mL centrifuged solids); mix 0.5 hr at RT; measure settling; centrifuge; decant. Repeat 2 times with solids portion, sampling slurry (0.7 mL) before measuring settling in wash #3. Dry solids at 105°C.	148.413 ug Al/mL; 1959.44 ug Al; 74.7% Al.	17.423 ug Al/mL; 569.55 ug Al; 21.6% Al.	1.387 ug Al/mL; 27.91 ug Al; 1.0% Al.	20875 ug Al/g; 7031 ug Al; 2.7% Al in solids; 97% Al removed.	Direct analysis: 1.11E-3 g Al/g sludge; Summation method: 1.38E-3 g Al/g sludge. 80.6% Al recovery.	
241-BX-110	PNNL-12026	After leach test, remove vessel from heating block and centrifuge 5 min. Replace on heating block & transfer solution to clean bottle. Wash solids 3 times with 15 mL 0.01 M NaOH, stirring 30 min (minimum), centrifuge, and decant. Dry at 105°C.	<u>For 60°C studies:</u> 95% Al removal at 1 M NaOH (94% Fe normalized), 99% Al removal at 3M NaOH (99% Fe normalized). <u>For 80°C studies:</u> 98% Al removal at 1 M NaOH (97% Fe normalized), 97% Al removal at 3M NaOH (96% Fe normalized).	<u>For 95°C studies:</u> 99% Al removal at 1 M NaOH (99% Fe normalized), 99% Al removal at 3M NaOH (98% Fe normalized).	—	—	—	—	—
241-BX-112	PNNL-12026	After leach test, leachate solution transferred from settled solids. Settled solids centrifuged & supernate decanted & combined w/ leachate solution. Wash solids 3 times with 15 mL 0.01 M NaOH stirring 30 min (minimum), centrifuge, and decant. Dry at 105°C.	<u>For 60°C studies:</u> 62% Al removal at 1 M NaOH (64% Fe normalized), 68% Al removal at 3M NaOH (75% Fe normalized). <u>For 80°C studies:</u> 54% Al removal at 1 M NaOH (59% Fe normalized), 63% Al removal at 3M NaOH (69% Fe normalized).	<u>For 100°C studies:</u> 51% Al removal at 1 M NaOH (57% Fe normalized), 59% Al removal at 3M NaOH (59% Fe normalized).	—	—	—	—	—
241-BY-104	PNNL-11278	Add 10.3 mL 10 M NaOH and water to a total volume of 32 mL (2 wt% solids) to solids, mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant (31.5 mL decanted liquid, 1.0 mL interstitial liquid, 3.3M OH <sup>-</sup> ).	Wash 3 times by adding 24 mL 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. Sample 0.4 mL slurry after mix of wash 3. Dry solids at 105°C and get 0.768 g solids (adjusted for slurry removed from wash 3 step).	10595 ug Al/mL; 225674 ug Al; 32% Al.	606 ug Al/mL; 4862 ug Al; 1% Al.	14.6 ug Al/mL; 426 ug Al; 0% Al.	14900 ug Al/mL; 11443 ug Al; 2% Al.	695880 ug Al; Summation: 1.75E+4 ug Al/g sludge; Direct: 1.88E+4 ug Al/g sludge; Mass Recovery: 93% Al.	98 % removed by leaching 2 times with caustic and washing 3 times with 0.01 M NaOH/NaNO <sub>2</sub> solution. Initial Wash Solution: 2425 ug Al/mL; 453475 ug Al; 65%.
241-BY-108	PNNL-11636	Add 46.85 mL 10 M NaOH and water to solids to total volume of ~170 mL; mix 5 hr at 100°C; cool; gravity settle; decant.	Add 34.3 mL of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to remaining solids; mix 0.5 hr at RT; gravity settle; decant and repeat for total of 3 wash steps. After 3 <sup>rd</sup> wash, remove two 0.2 g slurry samples before gravity settle & decant.	40222 ug Al (30% Al).	50276 ug Al (37% Al).	6290 ug Al (5% Al).	39336 ug Al (29% Al).	136124 ug Al (126% Al recovery).	—



**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

<u>Tank</u>	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-BY-110	PNNL-11278	Add 13 mL 10 M NaOH and water to a total volume of 43 mL (1.1 wt% solids). To solids, mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant (39.3 mL decanted liquid, 2.0 mL interstitial liquid, 3.2M OH <sup>-</sup> ).	Wash 3 times by adding 9 mL 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. Sample 0.4 mL slurry after mix of wash 3. Dry solids at 105°C = 0.548 g solids (adjusted for slurry removed from wash 3 step).	9048 ug Al/mL; 164674 ug Al; 94% Al.	454 ug Al/mL; 3414 ug Al; 2% Al.	38.6 ug Al/mL; 177 ug Al; 0% AL.	13850 ug Al/mL; 7590 ug Al; 4% Al.	175854 ug Al; Summation: 4.39E+4 ug Al/g sludge. Direct: 3.37E+4 ug Al/g sludge. Mass Recovery: 130% Al. Total of 96% Al removed by leaching 2 times with caustic and washing 3 times with 0.01 M NaOH/NaNO <sub>2</sub> solution.	—
241-C-102	PNNL-12026	Remove reaction vessel and allow to cool to RT, centrifuge 15 minutes, decant liquid, wash solids 3 times with 10mL 0.01 M NaOH/NaNO <sub>2</sub> , dry at 105°C.	At time of 5hr: #1=5185 ug Al/mL; #2=8524 ug Al/mL; #3=12030 ug Al/mL; #4=24160 ug Al/mL.	At time of 72hr: #1=6273 ug Al/mL, 115419 ug Al; #2=9406 ug Al/mL, 115694 ug Al; #3=16260 ug Al/mL, 635766 ug Al; #4=30620 ug Al/mL, 532788 ug Al.	#1=745 ug Al/mL, 22344 ug Al; #2=535 ug Al/mL, 16059 ug Al; #3=2066 ug Al/mL, 62393 ug Al; #4=3356 ug Al/mL, 101026 ug Al.	#1=139345 ug Al/g, 373891 ug Al; #2=180464 ug Al/g, 530202 ug Al; #3=26042 ug Al/g, 36321 ug Al; #4=27600 ug Al/g, 34949 ug Al.	Mass recovery: #1=84% Al; #2=99% Al; #3=118% Al; #4=117% Al. Summation method: % Al removed by washing + leaching: #1=27, #2=20, #3=95, and #4=95.	—	—
241-C-103	PNL-10712	Add 7.98 g 3 M NaOH to 6.4 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 6.5 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 7.0 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 6.6 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 6.5 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	16% Al found, 25500 ug Al/mL measured.	23% Al found, 30600 ug Al/mL measured.	9% Al found, 4505 ug Al/mL measured.	Measured concentration of 105,000 ug Al/g, 52% Al in residue.	Direct analysis: 1.4E-01 g Al/g sludge; Summation method: 1.58E-01 g Al/g sludge; 113% Al recovery.	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-C-103	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 1.0M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	Uncorrected/corrected recovery = 16/48% Al.	Uncorrected/corrected recovery = 23/29% Al.	Uncorrected/corrected recovery = 9/-3% Al.	Total wt% Al = 14; Total % Al removed = 48%; Undissolved residue (uncorrected/corrected) = 52/26% Al.	—	—
241-C-103	ORNL TM-13655	—	—	—	—	—	—	—	—
241-C-104	LAUR 97-2889	Add 154.1 mL water and 66.8 mL 10M NaOH to solids to get ~1 wt% solids & ~3M NaOH. Mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant. Supernate: 211.3 mL with 3.16 M free OH <sup>-</sup> , 13.89 mL settled solids, and 7.12 mL centrifuged solids.	Wash 3 times by adding 45.3 mL inhibited water to solids, mixing for 0.5 hr at RT; measure settling; centrifuge; and decant. Sample slurry (0.59 g) after 3 <sup>rd</sup> mix cycle. Dry solids at 105°C (0.5 g).	6068.4 ug Al/mL, 221132.5 ug Al (75.5% Al).	288.6 ug Al/mL, 60981.18 ug Al (20.8% Al).	17.16 ug Al/mL, 22242.98 ug Al (0.8% Al).	5000 ug Al/g, 8548 ug Al (2.9% Al).	292904.66 ug Al; Summation method: 6.95E-2 g Al/g; Direct analysis: 6.32E-2 g Al/g; 110% Al mass recovery.	—
241-C-104	PNWD-3027 (BNFL-RPT-021)	—	Transfer most of filtered solids from leach back into HDPE bottle using spatula and 0.01 M NaOH. Total slurry volume made to ~100 mL using additional 0.01M NaOH (total slurry wt ~123 g). Heat & stir at 85°C for 21 hr. Filter hot, yielding 92.45 g washing solution and 33.35 g wet solids. Repeat wash of wet solids, heating at 85°C for 22.5 hr. Yield 88.31 g washing liquid and 33.92 g wet solids. Analyze composite of two wash solutions. Filtered solids evaporated at 80°C, then dried overnight at 105°C to yield 7.6051 g dried leached solids.	Direct = 45900 ug Al/g; Adjusted due to 0.06% loss from evaporation = 45,873 ug Al/g; Amount = 453,3915 ug Al.	—	Direct = 2065 ug Al/g; Adjusted due to 0.23% loss from evaporation = 2060 ug Al/g; Amount = 372,430 ug Al.	Mean from KOH Fusion analysis = 32600 ug Al/g dry solids; mean from Na <sub>2</sub> O <sub>2</sub> Fusion analysis = 35,900 ug Al/g dry solids; Amount = 260,475 ug Al.	From original sample, assume 127,892 ug Al; with 34,250 ug Al remaining in leached solids. 95% Al removed.	Solubility versus temperature study done, with temperatures of 30°C for 18 hr, increased to 40°C and held for 24 hr, then increased to 50°C and stirred for 21 hours. Samples taken after each time. Used 0.1M NaOH to fluidize sample.
241-C-104	PNWD-3024 (BNFL-RPT-030)	—	Wash twice using 1405.22 g and 1512.18 g 0.01M NaOH for 8 hrs at 85°C (0.9 and 0.3M NaOH concentrations). De-water by filtration.	Caustic leach permeate: 15600 ug Al/mL (4.3E+7ug Al). Caustic leach permeate #1: 7770 ug Al/mL (1.2E+7 ug Al). Permeate #2: 2650 ug Al/mL (3.8E+6 ug Al). 90.8% Al removed.	—	Wash #1: <0.003% Al removed. Wash #2: <0.0006% Al removed	Final slurry sample: 36700 ug Al/mL (7.7E+6 ug Al). 6.8% Al in residue.	98% Al recovery	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-C-104	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M sodium hydroxide and 0.01 M sodium nitrate), using ~15 mL for each rinse.	<b>Leachate:</b> 1) 441 ug Al/g; 2) 559 ug Al/g; 3) 2440 ug Al/g	—	<b>Rinse:</b> 1) 17.3 ug Al/g; 2) 16.5 ug Al/g; 3) 83.4 ug Al/g	<b>Residual:</b> 1) 22800 ug Al/g; 2) 19900 ug Al/g; 3) 3920 ug Al/g	<b>% based on residue:</b> 1) 28.73% Al; 2) 39.73% Al; 3) 90.23% Al. <b>% based on leachate &amp; rinse:</b> 1) 19.36% Al; 2) 19.24% Al; 3) 83.16% Al. <b>% Recovery:</b> 1) 90.63% Al; 2) 79.51% Al; 3) 92.92% Al. <b>% Removed:</b> 1) 28.7% Al; 2) 39.7% Al; 3) 90.2% Al.	—
241-C-105	LAUR 97-2889	Add 82.6 mL water and 38 mL 10M NaOH to solids (1.6 wt% solids and ~3M NaOH in final solution); mix 5 hr at 100°C; cool; measure settling; centrifuge; decant. Supernate: 114.9 mL w/ 3.13 M free OH, 1.57 mL settled solids, and 1.36 mL centrifuged solids.	Wash 3 times by adding 39 mL inhibited water to solids; mix 0.5 hr at RT; measure settling; centrifuge; decant. Sample 0.6 g slurry after mix of wash 3. Dry solids at 105°C (0.175 g solids, 114.5 mL supernate from decant).	19410.3 ug Al/mL, 681301.53 ug Al; 83.7% Al.	1066 ug Al/mL, 122440.76 ug Al; 15% Al.	14.63 ug Al/mL, 1674.99 ug Al; 0.2% Al.	48192.771 ug Al/g, 5414.46 ug Al; 1% Al.	813831.74 ug Al.	—
241-C-105	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M NaOH and 0.01 M NaNO <sub>2</sub> ), using ~15 mL for each rinse.	<b>Leachate:</b> 1) 18100 ug Al/g.	—	<b>Rinse:</b> 1) 160 ug Al/g.	<b>Residual:</b> 1) 18700 ug Al/g.	<b>% based on residue:</b> 1) 97.34% Al. <b>% based on leachate &amp; rinse:</b> 1) 93.16% Al. <b>% Recovery:</b> 1) 95.82% Al. <b>% Removed:</b> 1) 97% Al.	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-C-106	PNNL-11381	With solids, add 8 mL 10 M NaOH and water to a total volume of 35 mL (42 g slurry; 3.9 wt% solids). Mix for 5 hr at 100°C. Cool, settle, and decant off liquid. Liquid: 27.6 mL with 3M OH. Solids: 13.5 g slurry with 10.5 mL interstitial liquid.	With solids after 2 <sup>nd</sup> caustic leach, wash 3 times by adding 45 mL 0.01M NaOH/NaNO <sub>2</sub> to solids portion. Mix 0.5 hr at RT. Settle. Decant off liquid. Take a sample (1.02 g) of the slurry after mixing during 3rd wash step. Total liquid: 133 mL. Total solids: 10.3 g wet. Dry solids at 105°C to obtain dry solids wt of 1.651 g (adjusted for samples removed from 3 <sup>rd</sup> wash step).	2048 ug Al/mL; 84378 ug Al (39% Al).	845 ug Al/mL; 12534 ug Al (6% Al).	86.8 ug Al/mL; 2672 ug Al (1% Al).	69100 ug Al/g; 114084 ug Al (53% Al).	213667 ug Al. Concentration in initial sludge solids as determined in caustic leaching test: Summation: 4.49E+04 ug Al/g. Direct: 4.85E+04 ug Al/g. Percent recovery = 93% Al.	—
241-C-106	PNNL-11432	Slurry from 1 <sup>st</sup> caustic leach (~6.4 wt% solids & 3.2M NaOH) mixed while heating to 100°C. Slurry was then mixed at 100°C for 5 hr, then transfer to sludge settler. Gravity settled at 85°C. Three axial supernate samples taken, 2832 mL supernate decanted. Add 1252 g inhibited water to resuspend solids + 1912 g inhibited water to rinse waste to sludge receipt tank.	<b>WASH 1:</b> Slurry in sludge receipt tank (~5846 g) cooled to ambient T & transferred to sludge settler (6.9 wt% solids) for gravity settling test (4900 mL). Three axial supernate samples taken. Supernate decanted (~3550 mL). 575 mL inhibited water added to resuspend solids, with additional 3050 mL inhibited water used to transfer to sludge receipt tank (~5662 g). <b>WASH 2:</b> Sludge mixed at ambient T & transferred to sludge settler (7.2 wt% solids; 4200 mL). Settled at ambient T; three supernate samples taken; supernate decanted (3800 mL). Inhibited water (773 mL) added to resuspend solids (foaming observed), with additional 3050 mL added to transfer and rinse solids to sludge receipt tank. <b>WASH 3:</b> Sludge (5530 g) mixed at ambient T & transferred to sludge settler (7.5 wt% solids; 4718 mL). Foam layer present at beginning of settling test. Settled at ambient T; 3 supernate samples taken; supernate decanted (3800 mL). <b>Sludge Removal:</b> DI water (2225 mL) added to resuspend solids (Total volume 3135 mL) and transfer to sludge receipt tank (3511 g; 14.8 wt% solids). Take 5 slurry samples (total wt. = 364 g), with remaining material transferred to 2 two-liter bottles and sent to privatization contractors.	30% removed; 3.1E+3 ug/mL; 1.44E+7 ug Al.	<0.1% removed; 1388 ug/mL; 335024 ug Al.	<b>First Water Wash:</b> 438 ug/mL; 0 ug Al. <b>Second Water Wash:</b> 150 ug/mL; 1.5E+5 ug Al. <b>Third Water Wash:</b> 55 ug/mL; 1.1E+5 ug Al.	4.91E+4 ug/g; 2.64E+7 ug Al (~69% Al in residue).	Mass Recovery: Direct Analysis: 4.78E+7 ug; Summation method: 4.64E+7 ug; 97% recovery.	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-C-106	PNWD-3013 (BNFL-RPT-017)	—	Most of filtered solids transferred back to high density polyethylene bottle using spatula and aliquots of 0.01 M NaOH, with final slurry volume of ~100 mL (114.88 g). Heat and stir at 85°C for 23 hr. Filter hot to yield 91.73 g washing solution. Repeat washing and heating at 85°C for 28 hr, collecting 123.95 g washing liquid. Composite of two wash solutions prepared for analysis. After final wash, filtered solids transferred to pre-weighed glass jar with DI water. Excess water evaporated at 80°C, with solids dried overnight at 105°C, yielding 5.8149 g dried leached solids.  Appearance: white solid collected around walls of jar, while brown solid remained in the bottom of the jar.  Similar to wash, 1.9464 g solids were stuck to magnetic stir bar, and had to be dissolved in HCl/HNO <sub>3</sub> .	22% removed by leaching.	—	—	—	—	<b>Solubility versus Temperature Results:</b> Blank: < detection limit. 30°C: 286 and 147 ug/g. 40°C: 67.9 and 333 ug/g. 50°C: 442 and 66.2 ug/g.
241-C-107	LAUR 96-2839	Add 9.8 mL 3M NaOH to solids (no settled solids/ 4.5 mL centrifuged solids); mix 5 hr at 100°C; cool; measure settling; centrifuge; decant.	Add 9.6 mL inhibited water to solids (no settled solids/ 4.7 mL centrifuged solids); mix 0.5 hr at RT; measure settling; centrifuge; decant; repeat twice with solids portion, sampling slurry (0.3 mL) before measuring settling in wash #3. Dry solids at 80°C.	25500 ug/mL (45.51%).	9350 ug/mL (21.11%).	61020 ug/mL (6.91%).	61307.9 ug/g (24.28 % in residue; 76% removed during sludge wash and caustic leach).	Direct analysis: 1.05E-1 g/g sludge; Sum method: 9.03E-2 g/g sludge. 86.32 % recovery.	—
241-C-107	PNNL-11278	Add 22.8 g 3 M NaOH to solids, mix at 100°C for 5 hr, cool, measure settling, centrifuge, decant (19.5 mL decanted liquid, 2.8 mL interstitial liquid, and 2.96 M OH).	Wash 3 times: 1 <sup>st</sup> wash: add 24.2 g 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. 2 <sup>nd</sup> and 3 <sup>rd</sup> wash: Add 19.1 g 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. Sample 0.22 g slurry after mix of wash 3. Dry solids at 80°C = 2.17 g solids (adjusted for slurry removed from wash 3 step).	12852 ug/mL; 253000 ug; 64%.	2997 ug/mL; 28271 ug; 7%.	550 ug/mL; 21930 ug; 6%.	41061 ug/mL; 89102 ug; 22%.	397543 ug; Summation: 8.68E-2 g Al/g sludge; Direct: 9.48E-2 g Al/g sludge; Mass Recovery: 109%.	78 % removed by leaching twice with caustic and washing 3 times with 0.01 M NaOH/NaNO <sub>2</sub> solution. Retrieval Solution: 44 ug/mL; 5240 ug; 1%.

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-C-107	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M sodium hydroxide and 0.01 M sodium nitrate), using ~15 mL inhibited water for each rinse.	Leachate: 7060 ug/g.	—	Rinse: 319 ug/g.	Residual: 20900 ug/g.	82% based on residue; 79.71% based on leachate & rinse; 97.71% recovery; 82% removed.	—
241-C-108	LAUR 95-2070	Add 16.1 mL 3M NaOH, mix 5 hr at 100°C, cool, measure settling, centrifuge, and decant. Volume settled solids ~20.8 mL, volume centrifuged solids ~6.2 mL, 15.78 mL liquid from decant.	Wash 3 times by adding 15.9 mL inhibited water to solids and mixing at RT for 0.5 hr, measure settling, centrifuge, and decant. For wash #3, sample slurry (0.3 mL) after mix phase. Dry solids at 80°C (1.1869 g).	39100 ug/mL (49.51 %).	21250 ug/mL (28.14%).	3230 ug/mL (12.87%).	64779 ug Al/g sludge.	Direct analysis: 1.51E-01 g/g sludge; Summation method: 1.8E-01 g/g sludge; 119.1% recovery.	Found in retrieval wash prior to leach: 280.5 ug/mL (3.03 %); Residual = 65 mg Al/g sludge (6.45%).
241-C-109	PNL-9387	—	—	—	73% from leach.	—	19% in leached sludge.	—	—
241-C-112	PNL-9387	—	—	—	51% found in leach solution.	—	15% in leached sludge.	—	—
241-S-101	PNNL-12026	N/A	—	—	—	Summation Method [leach (wash + leach)]: 1) 66% (70%); 2) 87% (88%); 3) 59% (63%); 4) 89% (90%). Fe Normalized: 1) 71%; 2) 75%; 3) 57%; 4) 70%.	—	—	<b>Washed Solids:</b> 1) 1.04E+5 ug/g; 5.18E+5 ug Al; 2) 1.04E+5 ug/g; 5.29E+5 ug Al; 3) 1.04E+5 ug/g; 7.1E+5 ug; 4) 1.04E+5 ug/g; 5.2E+5 ug. <b>5 hr:</b> 1) 86.3 ug/mL; 2) 1.62E+3 ug/mL; 3) 334 ug/mL; 4) 4.67E+3 ug/mL. <b>24 hr:</b> 1) 501 ug/mL; 2) 2.64E+3 ug/mL; 3) 1.17E+3 ug/mL; 4) 9.95E+3 ug/mL. <b>72 hr:</b> 1) 860 ug/mL; 2) 3.61E+3 ug/mL; 3) 1.17E+3 ug/mL; 4) 1.09E+4 ug/mL. <b>168 hr:</b> 1) 1.26E+5 ug/mL; 2.39E+5 ug Al. 2) 4.95E+3 ug/mL; 4.27E+5 ug Al. 3) 3.1E+3 ug/mL; 3.62E+5 ug. 4) 1.34E+4 ug/mL; 4.05E+5 ug. <b>Final Wash Solution:</b> 1) 44.9 ug/mL; 3.52E+3 ug. 2) 75.3ug/mL; 5.74E+3 ug. 3) 469 ug/mL; 3.76E+4 ug. 4) 104 ug/mL; 8.09E+3 ug. <b>Leached Solids:</b> 1) 3.32E+4 ug/g; 1.26E+5 ug. 2) 2.84E+4 ug/g; 6.54E+4 ug. 3) 4.98E+4 ug/g; 2.8E+5 ug. 4) 3.48E+4 ug/g; 4.98E+4 ug. <b>Mass Recovery (%):</b> 1) 71%; 2) 94%; 3) 96%; 4) 89%.



**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-S-101	PNNL-11636	Add 34 mL 10 M NaOH and 80 mL water to solids/sludge from previous step; mix 5 hr at 100°C; cool; gravity settle; take a 1ml sample; mix 95 hr at 100°C; cool; settle; and decant.	Add 24 mL of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to remaining solids; mix 0.5 hr at RT; centrifuge; decant; and repeat for total of 3 wash steps. After 3 <sup>rd</sup> wash, remove two 0.194 g slurry samples before centrifuge & decant.	72742 (11%).	488485 (76%).	54553 (9%).	25645 (4%).	641425 (109% recovery).	—
241-S-101	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M NaOH and 0.01 M NaNO <sub>2</sub> ), using ~15 mL inhibited water for each rinse.	Leachate: 7050 ug/mL.	—	Rinse: 106 ug/mL.	Residual: 8760 ug/g.	97.76% based on residue; 97.29% based on leachate & rinse; 99.54% Recovery; 98% Removed.	—
241-S-101	ORNL TM-13655	—	Centrifuge leached solids for 20 min at 4500 rpm, decant liquid, and weigh solids. Wash 3 times with 25 mL inhibited water. Place sample in vortex mixer until all solids suspended in solution, weigh sample, centrifuge for 5 min at 4500 rpm, decant liquid, and repeat twice. After last wash, sample centrifuged for 20 min at 4500 rpm. Weights of solids and combined water washes were determined.	<p><b>1)</b> 1M NaOH, 70°C: 5hr = 86.3 ug Al/mL; 24 hr = 501 ug Al/mL; 72 hr = 860 ug Al/mL; 168 hr = 1260 ug Al/mL. (2.39E+5 total ug Al).</p> <p><b>2)</b> 1M NaOH, 95°C: 5hr = 1.62E+3 ug Al/mL; 24 hr = 2.64E+3 ug Al/mL; 72 hr = 3.61E+3 ug Al/mL; 168 hr = 4.95E+3 ug Al/mL. (4.27E+5 total ug Al).</p> <p><b>3)</b> 3M NaOH, 70°C: 5hr = 334 ug Al/mL; 24 hr = 1.17E+3 ug Al/mL; 72 hr = 3.10E+3 ug Al/mL; 168 hr = 5.68E+3 ug Al/mL. (3.62E+5 total ug Al).</p> <p><b>4)</b> 3M NaOH, 95°C: 5hr = 4.67E+3 ug Al/mL; 24 hr = 9.95E+3 ug Al/mL; 72 hr = 1.09E+4 ug Al/mL; 168 hr = 1.34E+4 ug Al/mL. (4.05E+5 total ug Al).</p>	<p><b>1)</b> 168 hr = 44.9 ug Al/mL (3.52E+3 ug total Al).</p> <p><b>2)</b> 168 hr = 75.3 ug Al/mL (5.74E+3 ug total Al).</p> <p><b>3)</b> 168 hr = 469 ug Al/mL (3.76E+4 ug total Al).</p> <p><b>4)</b> 168 hr = 104 ug Al/mL (8.09E+3 ug total Al).</p>	<p><b>1)</b> 168 hr = 3.32E+4 ug Al/g solids (1.26E+5 ug total Al).</p> <p><b>2)</b> 168 hr = 2.84E+4 ug Al/g solids (6.54E+4 ug total Al).</p> <p><b>3)</b> 168 hr = 4.98E+4 ug Al/g solids (2.80E+5 ug total Al).</p> <p><b>4)</b> 168 hr = 3.48E+4 ug Al/g solids (4.98E+4 ug total Al).</p>	—	—	The water content in sample of washed sludge was 72.8%. This was assumed to be the same for each aliquot used for leaching. The density of the composite wash solution (26.043 g/25 mL filtered composite) was used to determine that the original sludge sample contained 33.4 g (30 wt%) water-soluble solids, 23.17 (21 wt%) water-insoluble solids, and 54.04 g (49 wt%) water.



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-S-104	PNL-10712	Solids did not settle. Add 13.6 g 3 M NaOH to 10 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Solids did not settle. Add 12.3 g 1 M NaOH/1 M NaNO <sub>2</sub> to 11 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 11.5g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 11.5g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 11.3 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	10% found, 18700 ug/mL measured.	12% found, 13600 ug/mL measured.	13% found, 3060 ug/mL measured.	Measured concentration of 330000 ug/g, 62% in residue.	Direct analysis: 1.5E-01 g/g sludge; Summation method: 1.77E-01 g/g sludge; 118% recovery.	—
241-S-104	LAUR 95-2070	Add 6.6 mL 3M NaOH, mix 5 hr at 100°C, cool, measure settling, centrifuge, and decant. Volume settled solids ~16.1 mL; volume centrifuged solids ~9.1 mL.	Wash 3 times by adding 7.8 mL inhibited water to solids and mix at RT for 0.5 hr, measure settling, centrifuge, and decant. For wash #3, sample slurry (0.3 mL) after mix phase. Dry solids at 80°C (2.0985 g).	16150 ug/mL (9.04%).	17850 ug/mL (10.39%).	4590 ug/mL (9.67%).	328947 ug Al/g sludge.	Direct analysis: 1.67E-01 g/g sludge; Summation method: 1.86E-01 g/g sludge; 111.46% recovery.	Found in retrieval wash prior to leach: 417 ug/mL (3.7%); Residual = 329 mg Al/g sludge (67.2%).
241-S-104	PNNL--11089	Add NaOH to get ~8 wt% solids and a final NaOH of 3.0M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	Uncorrected/corrected recovery = 10/25%.	Uncorrected/corrected recovery = 12/10 %.	Uncorrected/corrected recovery = 13/2%.	Total of 15wt% Al; Total of 38% Al removed; percent undissolved residue (uncorrected/corrected) = 62/59%.	—	—
241-S-104	PNNL-11636	Add 12 ml 10 M NaOH and 10 ml water to give 121 g slurry; mix 76 hr at 100°C; cool; centrifuge; and decant.	Add 50 mL of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to remaining solids; mix 0.5 hr at RT; centrifuge; decant; and repeat for total of 3 wash steps. After 3 <sup>rd</sup> wash, remove two (0.501 g and 0.201 g) slurry samples before centrifuge & decant.	539570 ug (95% removal; mass value corrected for mass of material present in samples taken at 5 & 75 hr; no change between 75 & 211 hr).	15429 ug (3% removal).	9654 ug (2% removal).	5530 ug (1%).	570183 ug.	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-S-104	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M NaOH and 0.01 M NaNO <sub>2</sub> ), using ~15 mL for each rinse.	<b>Leachate:</b> 1) 1200 ug/mL; 2) 1870 ug/mL; 3) 2750 ug/mL; 4) 1620 ug/mL; 5) 1340 ug/g; 6) 3830 ug/g.	—	<b>Rinse:</b> 1) 162 ug/mL; 2) 292 ug/mL; 3) 68.8 ug/mL; 4) 33.8 ug/mL; 5) 46.7 ug/g; 6) 45.5 ug/g.	<b>Residual:</b> 1) 77300 ug/g; 2) 63900 ug/g; 3) 50000 ug/g; 4) 40100 ug/g; 5) 66600 ug/g; 6) 12600 ug/g.	<b>% based on residue:</b> 1) 34.51%; 2) 50.28%; 3) 20.4%; 4) 20.8%; 5) 26.6%; 6) 96.32%. <b>% based on leachate &amp; rinse:</b> 1) 7.64%; 2) 12.17%; 3) 11.2%; 4) 13.22%; 5) 14.98%; 6) 91.66%. <b>% Recovery:</b> 1) 73.13%; 2) 61.89%; 3) 90.8%; 4) 92.42%; 5) 88.3%; 6) 95.35%	—
241-S-107	PNNL-11278	Add 55 mL 10 M NaOH and water to solids for a total volume of 185 mL (0.35 wt% solids). Mix at 100°C for 5 hr, cool, measure settling, centrifuge, and decant. Obtain 171 mL decanted liquid, 5.9 mL interstitial liquid, and 2.8 M OH.	Wash 3 times by adding 37 mL 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. Sample 0.4 mL slurry after mix of wash 3. Dry solids at 105°C to get 0.702 g solids (adjusted for slurry removed from wash 3 step).	11725 ug/mL; 398650 ug; 56%.	949 ug/mL; 118728 ug; 17%.	83.2 ug/mL; 3220 ug; 0%.	272000 ug/mL; 190944 ug; 27%.	711542 ug; <b>Summation:</b> 2.09E+5 ug/g; <b>Direct:</b> 2.05E+5 ug/g; <b>Mass Recovery:</b> 102%.	73 % removed by leaching twice with caustic and washing 3 times with 0.01 M NaOH/NaNO <sub>2</sub> solution.



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-S-107	PNNL-12010	Add 3670 g 3M NaOH (target 3M NaOH final concentration & 5 wt% insoluble solids) and 540 g DI to sludge. Heat at 100°C for 5.75 hr (take slurry samples; free OH <sup>-</sup> ~ 2.86 M). Mix 5 min at 80°C and transfer to settler (lost 603 g due to evaporation). Add 437 g DI for settling tests (7.13 cm/hr at 7.2 wt% solids & 80°C. Lost 599 g due to evaporation, 47 g for 3 liquid samples, and decanted 4343 g supernate).	<p><b>First water wash:</b> Add inhibited water (4552.65 g) to compacted sludge. Mix 2 hr at 50°C (lost ~ 166 g from evaporation and 86 g for 4 slurry samples). Settle (15.33 cm/hr at 4.3 wt% solids &amp; 50°C; lost 265 g from evaporation, 43 g for 3 liquid samples, and 4073 g of decanted supernate).</p> <p><b>Second water wash:</b> Add 4552 g inhibited water; mix 1.5 hr at 50°C (lost 139 g due to evaporation). Settle (15.12 cm/hr at 4.3 wt% solids &amp; 50°C; lost 340 g due to evaporation, 43 g for 3 liquid samples, and 4059 g of decanted supernate).</p> <p><b>Third water wash:</b> Add 4552 g inhibited water; mix 42 min at 50°C (lost 603 g due to evaporation). Settle (12.2 cm/hr at 4.3 wt% solids &amp; 50°C. Lost 342 g due to evaporation, 41 g for 3 liquid samples, and 2731 g of decanted supernate). Mix sludge 4 min at 50°C. Add 459 g DI and settle (6.19 cm/hr at 8.7 wt% solids &amp; 50°C. Lost 92 g for 4 slurry samples, 485 g due to evaporation, 49 g for 3 liquid samples, and 995 g of decanted supernate).</p>	1.34E+4 ug/g; 4.12E+7 ug Al (21.8%).	8.65E+3 ug/g, 3.34E+7 ug Al (17.6%).	<p><b>First water wash:</b> 2.64E+3 ug/g, 1.19E+7 ug Al (6.3%).</p> <p><b>Second water wash:</b> 724 ug/g, 9.09E+4 ug Al (0.048%).</p> <p><b>Third water wash:</b> 199 ug/g, 5.49E+4 ug Al (0.029%).</p>	3.14E+5 ug/g, 9.81E+7 ug Al (51.8% left in residue).	Direct: 2E+8 ug. Summation: 1.89E+8. 95% recovery.	<p><b>Concentrations (ug Al/mL; ug Al; % removal) for extended caustic leach study:</b>  <u>Slurry at 6.5 hr:</u> 2.48E+3; 4.23E+6; 19%.  <u>Slurry at 14 hr:</u> 3.86E+3; 6.62E+6; 29.7%.  <u>Supernate at 22 hr:</u> 5.58E+3; 9.47E+6; 42.5%. <u>Slurry at 38.5 hr:</u> 7.39E+3; 1.19E+7; 53.5%. <u>Supernate at 46.5 hr:</u> 8.04E+3; 1.26E+7; 56.7%. <u>Slurry at 54.5 hr:</u> 8.03E+3; 1.27E+7; 57%. <u>Slurry at 64 hr:</u> 9.04E+3; 1.42E+7; 63.5%. <u>Supernate at 72 hr:</u> 8.95E+3; 1.39E+7; 62.3%.  <u>Supernate at 94.5 hr:</u> 1.04E+4; 1.52E+7; 68.2%. <u>Supernate at 118.5 hr:</u> 1.02E+4; 1.5E+7; 67.4%. <u>Supernate at 237 hr:</u> 1.48E+4; 1.57E+7; 70.6%.</p> <p><b>Extended Caustic Leach:</b> Mix 4190 g 4.32 M NaOH with 5759 g starting slurry at 80°C (lost 4027 g slurry). (1843 g slurry; 2.94M NaOH; &amp; 4 wt% solids). <b>Day 1:</b> Add 219 g DI. Take 4 slurry samples at 0 hr (66 g). One slurry sample at 6.5 hr (16 g). One slurry sample at 14 hr (22 g). One supernate sample at 22 hr (21 g). Estimate 236 g water evaporation. <b>Day 2:</b> Add 149 g DI. Take one slurry sample at 30 hr (18 g). One slurry sample at 38 hr (26 g). One supernate sample at 46 hr (15 g). Estimate 250 g water evaporation. <b>Day 3:</b> Add 281 g DI. Take one slurry sample at 54 hr (23 g). One slurry sample at 62 hr (21 g). One supernate sample at 70 hr (12 g). Estimate 181 g water evaporation. <b>Day 4:</b> Add 102 g DI. Take one slurry sample at 94 hr (12 g). Estimate 194 g water evaporation. <b>Day 5:</b> Add 204 g DI. Take one supernate sample at 118 hr (18 g). Estimate 163 g water evaporation. <b>Settling Test at 166 hr:</b> Add 484 g DI (5.62 cm/hr at 1.9 wt% solids &amp; 80°C). Take 3 supernate samples at 237 hr (36 g). Take 4 slurry samples at 237 hr (69 g). Estimate 122 g water evaporation.</p>



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-S-110	PNNL-13702	—	—	<ol style="list-style-type: none"> <li>1) 1775 ug/g, 155819 ug Al.</li> <li>2) 1881 ug/g, 184459 ug Al.</li> <li>3) 1911 ug/g, 188989 ug Al.</li> <li>4) 3029 ug/g, 272961 ug Al.</li> <li>5) 3771 ug/g, 374041 ug Al.</li> <li>6) 3729 ug/g, 393569 ug Al.</li> <li>7) 4059 ug/g, 383902 ug Al.</li> <li>8) 4158 ug/g, 415665 ug Al.</li> <li>9) 3967 ug/g, 422563 ug Al.</li> </ol>	<p><b>% Al removed at 168 hr:</b></p> <ol style="list-style-type: none"> <li>1) 39%.</li> <li>2) 47%.</li> <li>3) 50%.</li> <li>4) 69%.</li> <li>5) 91%.</li> <li>6) 96%.</li> <li>7) 91%.</li> <li>8) 100%.</li> <li>9) 100%.</li> </ol>	<ol style="list-style-type: none"> <li>1) 178 ug/g, 16167 ug Al.</li> <li>2) 139 ug/g, 12549 ug Al.</li> <li>3) 203 ug/g, 18882 ug Al.</li> <li>4) 137 ug/g, 11833 ug Al.</li> <li>5) 186 ug/g, 16285 ug.</li> <li>6) 178 ug/g, 15379 ug.</li> <li>7) 139 ug/g, 12130 ug.</li> <li>8) 9 ug/g, 762 ug.</li> <li>9) 134 ug/g, 11862 ug.</li> </ol>	<ol style="list-style-type: none"> <li>1) 331800 ug/g, 265108 ug Al.</li> <li>2) 334800 ug/g, 224651 ug Al.</li> <li>3) 343800 ug/g, 205592 ug Al.</li> <li>4) 299800 ug/g, 126815 ug Al.</li> <li>5) 192800 ug/g, 39910 ug Al.</li> <li>6) 122800 ug/g, 17560 ug Al.</li> <li>7) 176800 ug/g, 37128 ug Al.</li> <li>8) 13100 ug/g, 1402 ug Al.</li> <li>9) 8840 ug/g, 875 ug Al.</li> </ol>	<ol style="list-style-type: none"> <li>1) 437094 ug (113% recovery).</li> <li>2) 421659 ug (111%).</li> <li>3) 413463 ug (110%).</li> <li>4) 411609 ug (109%).</li> <li>5) 430235 ug (114%).</li> <li>6) 426508 ug (113%).</li> <li>7) 433159 ug (115%).</li> <li>8) 417829 ug (110%).</li> <li>9) 435301 ug (115%).</li> </ol>	<p><b>Al concentration (ug/g) at t (hr) = 4, 8, 24, 72, 168a, 168b, and final wash:</b></p> <ol style="list-style-type: none"> <li>1) 542, 715, 919, 1262, 1811, 1739, 178.</li> <li>2) 673, 778, 932, 1267, 1892, 1870, 139.</li> <li>3) 626, 701, 830, 1201, 1926, 1896, 203.</li> <li>4) 758, 1042, 1699, 2502, 3003, 3056, 137.</li> <li>5) 870, 1029, 1794, 2907, 3757, 3786, 186.</li> <li>6) 855, 1032, 1880, 2911, 3788, 3670, 178.</li> <li>7) 1253, 1823, 2781, 3550, 4087, 4031, 139.</li> <li>8) 1428, 2059, 3186, 4038, 4167, 4150, 138.</li> <li>9) 1462, 2239, 3499, 3951, 3891, 4043, 134.</li> </ol>
241-S-110	PNNL-14018	—	Centrifuge leached slurries at 3000 rpm for a minimum of 5 min. Decant supernate from solids. Wash solids 3 times with 0.1M NaOH. Centrifuge samples after each wash, with supernate combined with final leachate. Residual solids dried to a constant weight at 105°C.	<p>% Al removed after leaching 48 hr:</p> <ol style="list-style-type: none"> <li>1) 1%.</li> <li>2) 7%.</li> <li>3) 11%.</li> <li>4) 62%.</li> <li>5) 3%.</li> <li>6) 15%.</li> <li>7) 12%.</li> <li>8) 49%.</li> <li>9) 2%.</li> <li>10) 8%.</li> <li>11) 8%.</li> <li>12) 41%.</li> <li>13) 6%.</li> <li>14) 9%.</li> <li>15) 51%.</li> <li>16) 51%.</li> </ol>	—	—	<p>ug Al/g leached sludge:</p> <ol style="list-style-type: none"> <li>1) 350000.</li> <li>2) 333000.</li> <li>3) 310000.</li> <li>4) 332000.</li> <li>5) 332000.</li> <li>6) 349000.</li> <li>7) 330000.</li> <li>8) 293000.</li> <li>9) 338000.</li> <li>10) 355000.</li> <li>11) 358000.</li> <li>12) 337000.</li> <li>13) 305000.</li> <li>14) 304000.</li> <li>15) 273000.</li> <li>16) 281000.</li> </ol>	—	—
241-S-111	PNNL-11636	Add 28 mL 10 M NaOH and 64 mL water to solids from previous leach step; mix 60 hr at 100°C; cool; gravity settle; and decant.	Add 19 mL of 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to remaining solids; mix 0.5 hr at RT; centrifuge; decant; repeat for total of 3 wash steps. After 3rd wash, remove two (0.50 g and 0.20 g) slurry samples before centrifuge & decant.	717046 (48%)	541896 ug (36%)	96448 ug (6%)	3131 ug (0%)	1500417	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-SX-101	PNWD-3512 (WTP-RPT-117)	3 M NaOH leach of samples SX-101-5 and SX-101-6 [slurry weights of 3.851g and (1.301/9.963) g, respectively (see Table 2.6 in reference)]. Add 4.8 ml 10M NaOH and 8.2 ml DI to each. Heat at 85 ± 5°C for 8 hr. Cool to RT overnight. Centrifuge and decant supernate to 30 mL plastic vials. Add 0.1M NaOH at 3:1 vol:vol ratio to remaining solids. Mix, separate, and decant to same container as initial leachate solution. Continue washings until supernate was colorless. Filter samples (0.2 um syringe filter), and place 1 ml aliquot in container w/ 10 mL of 1M HNO <sub>3</sub> for ICP-AES.	Sample #/Prior NaOH Leach/Post NaOH Leach/Oxidative Leach [NaOH] <sub>initial</sub> M/Oxidative Leach T °C/Oxidative Leach ([NaMnO <sub>4</sub> ] <sub>initial</sub> /[Cr]) Tank#1/No/No/3M/85°C/1.1. Tank#2/No/No/5M/85°C/5. Tank#3/Yes/No/0.25M/25°C/1.1. Tank#4/Yes/No/0.25M/85°C/1.1. Tank#5/No/Yes/0.25M/25°C/1.1. Tank#6/No/Yes/0.25M/85°C/1.1.	—	—	—	—	% removal values for 6 bottles range from 83-97% Al removal 1 = 96%, 2 = 96%, 3 = 83%, 4 = 89%, 5 = 97%, 6 = 97%.	—
241-SX-108	PNNL-11278	Add 60 mL 10 M NaOH and water to a total volume of 240 mL (2.5 wt% solids) to solids, mix at 100°C for 5 hr, cool, measure settling, and decant resulting in 191 mL decanted liquid, 51 mL interstitial liquid, and 2.9 M OH.	Wash 3 times by adding 151 mL 0.01 M NaOH/NaNO <sub>2</sub> to solids, mix at RT for 0.5 hr, measure settling, centrifuge, and decant. Sample 0.4 mL slurry after mix of wash 3. Dry solids at 105°C = 7.19 g solids (adjusted for slurry removed from wash 3 step).	5369 ug/mL; 767767 ug; 28%.	1080 ug/mL; 14386 ug; 1%.	104 ug/mL; 0 ug; 0%.	270000 ug/mL; 194130 ug (table shows 1941300 ug, which may be incorrect); 71%.	2723453 ug; <u>Summation:</u> 1.34E+5 ug/g; <u>Direct:</u> 9.02E+4 ug/g; <u>Mass Recovery:</u> 149%.	29 % removed by leaching twice with caustic and washing 3 times with 0.01 M NaOH/NaNO <sub>2</sub> solution.
241-SX-108	PNNL-11908	—	Wash solids 3 times with 0.1M NaOH. After each wash, centrifuge and transfer supernate to final leachate container. The washed residual solids were dried to a constant wt at 105°C.	1) 70%. 2) 93%. 3) 63%. 4) 89%.	—	—	0.1M oxidative leached solids - initial: 22900 ug Al/g solids. 3M oxidative leached solids - initial: 235000 ug Al/g solids. 1) 13300 ug/g. 2) 50500 ug/g. 3) 10900 ug/g. 4) 116000 ug/g residual Al.	—	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-SX-113	Laur 97-2889	Add 93 mL water & 40.4 mL 10 M NaOH (~1 wt% solids & ~3M NaOH); mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant. Final solution had 126.4 mL supernate with 2.99 M free OH, 17.3 mL settled solids, & 6 mL centrifuged solids.	Wash 3 times by adding 27.5 mL inhibited water to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Subsample after mixing for wash #3 (0.62 g). Dry solids at 105°C.	623.35 ug/mL. 12167.79 ug (27.3%).	208 ug/mL. 26289.12 ug (58.9%).	17.05 ug/mL. 1371.67 ug (3.1%).	30438 ug/g. 4812.21 ug (10.8%).	44641 ug.	—
241-SX-113	ORNL TM-13500	—	After mixing, the samples were centrifuged for 15-20 min at 2500 rpm. The wet sludge was rinsed three times with inhibited water (0.01 M NaOH and 0.01 M NaNO <sub>2</sub> ), using ~15 mL inhibited water for each rinse.	<u>Leachate:</u> 1) 307 ug/g; 2) 215 ug/g.	—	<u>Rinse:</u> 1) 10 ug/g; 2) 6.61 ug/g.	<u>Residual:</u> 1) 5080 ug/g; 2) 2820 ug/g.	<u>% based on residue:</u> 1) 51.04%; 2) 78.69%. <u>% based on leachate &amp; rinse:</u> 1) 62.12%; 2) 81.75%. <u>% Recovery:</u> 1) 111.08%; 2) 103.06%. <u>% Removed:</u> 1) 51%; 2) 78.7%.	—
241-T-104	PNL-10712	Add 11.4 g 3 M NaOH to 4.8 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 3.9 g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to 11 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 3.9g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 4.0g 0.01 M NaOH/0.01 M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	24%, 5015 ug/mL measured.	26%, 4845 ug/mL measured.	11%, 1615 ug/mL measured.	Measured concentration of 33000 ug/g; 38% Al in residue.	Direct analysis: 4.7E-02 g/g sludge; Summation method: 4.29E-02 g/g sludge; 91% recovery.	—
241-T-104	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 2.2M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	Uncorrected/corrected recovery = 24/37%.	Uncorrected/corrected recovery = 26/24%.	Uncorrected/corrected recovery = 11/2%.	Total wt% Al = 4.7%; Total % Al removed = 62%; Percent undissolved residue (uncorrected/corrected) = 38/35%.	—	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-T-104	ORNL TM-13660	Add ~10mL of 3M NaOH to samples. Mix for 5 hr 30 min. Cool. Centrifuge for ~ 10 min. Decant and filter.	Wash 3 times by adding inhibited water and place on shaker at RT for 30 min. Remove samples from shaker, centrifuge, decant liquid and filter. Repeat process.	RT: 7320 ug/mL. T=60: 5160 ug/mL. T=95: 5870 ug/mL.	RT: 1720 ug/mL. T=60: 1040 ug/mL. T=95: 1380 ug/mL.	RT: 220, 52.8, and 24.4 ug/mL. T=60: 106, 18.4, and 8.48 ug/mL. T=95: 174, 28.4, and 10.7 ug/mL.	—	—	—
241-T-104	LAUR 95-2070	Add 3M NaOH to solids, mix at 100°C for 5 hr, settle and decant.	Add inhibited water to solids, mix at RT for 0.5 hr, settle/decant, and repeat for total of 3 washes. Centrifuge, decant, and dry at 80°C.	11000 ug/mL (38.62%).	4000 ug/mL (14.9%).	420 ug/mL (9.16%).	78125 ug/g (36.38% in residual).	Direct analysis: 5.35E-2 g Al/g sludge; Summation: 5.44E-2 g Al/g sludge (101.77 % recovery).	Residual: 78 mg Al/g sludge (36%).
241-T-107	LAUR 95-2070	To solids, add 3M NaOH, mix at 100°C for 5 hr, settle and decant.	Wash solids 3 times with inhibited water at RT for 0.5hr, settle and decant. After wash, centrifuge, decant, and dry solids at 80°C.	14000 ug/mL (62.71%).	1900 ug/mL (10.22%).	78 ug/mL (1.46%).	50691 ug/g in leached sludge (22.06% in residue).	Direct analysis: 5.67E-2 g Al/g sludge; Summation: 6.17E-2 g Al/g sludge (108.79% recovery).	Residual: 51 mg Al/g sludge (22%).
241-T-110	PNNL-13956	—	Leached solids were washed 3 times with 30 mL portions of 0.01M NaOH, and then dried at 105°C.	<b>ug Al/g, ug Al, and % Al removed after 1 week leaching (168 hr):</b> 1) [4], [186], 27%. 2) [8], [371], 42%. 3) [13], [703], 59%. 4) [6], [245], 53%. 5) [13], [544], 67%. 6) [14], [653], 83%. 7) [6], [255], 50%. 8) [12.4], [613], 75%. 9) 16, 808, 77%.  (Values in [] are within 10 times the detection limit and uncertainty is >15%).	—	<b>ug/g and ug Al removed after 1 week leaching (168 hr):</b> 1) [2], [214]. 2) -, -. 3) [6], [528]. 4) [6], [590]. 5) [7], [677]. 6) [9], [822]. 7) [6], [587]. 8) [7.2], [661]. 9) [4], [333].  (Values in [] are within 10 times the detection limit and uncertainty is >15%).	<b>ug/g and ug Al removed after 1 week leaching (168 hr):</b> 1) 800, 1090. 2) 426, 520. 3) [740], [868]. 4) 540, 734. 5) 500, 602. 6) 290, 305. 7) 630, 834. 8) 380, 433. 9) 295, 343.  (Values in [] are within 10 times the detection limit and uncertainty is >15%).	1) 1490 ug Al. 2) 890 ug. 3) [2098] ug. 4) 1569 ug. 5) [1823] ug. 6) [1781] ug. 7) 1676 ug. 8) 1707 ug. 9) 1484 ug.  (Values in [] are within 10 times the detection limit and uncertainty is >15%).	<b>Al concentration (ug/g) as a function of time [4, 8, 24, 72, 168, 168, Final Wash]:</b> 1) [5.4], [5.6], [5.4], [5], [5.1], [3.6], [2.3]. 2) [11.3], [12.1], [11.9], [9], [8.5], [8], [0]. 3) [10.7], [12.6], [13.5], [13.4], [15.2], [11.3], [5.8]. 4) [4.1], [4.3], [3.9], [3.7], [8.3], [4.2], [6.3]. 5) [11.6], [12.5], [11.5], [12.4], [14.3], [12.1], [7.3]. 6) [11.7], [12.1], [12.8], [13.1], [15], [13.5], [9]. 7) [4.6], [5.2], [5], [4.5], [8.8], [2.7], [6.4]. 8) [19.1], [12.6], [13.1], [13.9], [13.1], [11.6], [7.2]. 9) [10.4], [14.4], [14.1], 14.8, 17.9, [14.1], [3.7].  (Values in [] are within 10 times the detection limit and uncertainty is >15%).

**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

<u>Tank</u>	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-T-111	PNL-10712	Solids did not settle. Add 11.7 g 3 M NaOH to 8.5 mL centrifuged solids; mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Solids did not settle. Add 12.7g 0.01M NaOH/0.01M NaNO <sub>2</sub> to 6.2 mL centrifuged solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 13g 0.01M NaOH/0.01M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; measure settling; centrifuge; and decant. Add 12.9g 0.01M NaOH/0.01M NaNO <sub>2</sub> to solids; mix 0.5 hr at RT; sample slurry; measure settling; centrifuge; and decant.	4%; 79.9 ug/mL measured.	5%; 78.2 ug/mL measured.	4%; 17 ug/mL measured.	Measured concentration of 5700 ug/g; 87% in residue.	Direct analysis: 4.9E-03 g/g sludge. Summation method: 3.68E-03 g/g sludge. 75% recovery.	—
241-T-111	PNNL-11089	Add NaOH to get ~8 wt% solids and a final NaOH of 3.4M. Mix at 100°C for 5 hr. Cool, centrifuge, and decant.	Add inhibited water. Mix at RT for 0.5 hr (target 8 wt% solids). Centrifuge, decant, and repeat twice more.	Uncorrected/ corrected recovery = 4/8%.	Uncorrected/ corrected recovery = 5/5 %.	Uncorrected/ corrected recovery = 4/1%.	Total wt% Al = 0.49. Total % Al removed = 13%. Percent undissolved residue (uncorrected/ corrected) = 87/86%	—	—
241-TY-104	LAUR 96-2839	Add 12.6 mL 3M NaOH to solids (no settled solids/ 4.6 mL centrifuged solids); mix 5 hr at 100°C; cool; measure settling; centrifuge; and decant.	Add 8.1 mL inhibited water to solids (no settled solids/ 4.5 mL centrifuged solids); mix 0.5 hr at RT; measure settling; centrifuge; decant; and repeat twice with solids portion, sampling slurry (0.3 mL) before settling in wash #3. Dry solids at 80°C.	7480 ug/mL (27.67%).	2890 ug/mL (18.23%).	688.5 ug/mL (8.23%).	42857.14 ug/g. 36.74% Al in residue; 63% Al removed during sludge wash/caustic leach.	Direct analysis: 4.28E-2 g/g sludge; Sum method: 4.03E-2 g/g sludge. 94.09 % recovery.	—
241-U-108	PNNL-11908	—	Wash solids 3 times with 0.1M NaOH. After each wash, centrifuge and transfer supernate to final leachate container. The washed residual solids were dried to a constant wt at 105°C.	1) 6.5%, 2) 96.4%, 3) 12%, 4) 94.7%, 5) 10%, 6) 81%.	—	—	202000 ug Al/g initial sludge. <u>ug Al/g final leached sludge:</u> 1)183500. 2)18100. 3) 149000. 4) 66500. 5) 179000. 6) 81500.	—	—

**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

<u>Tank</u>	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-U-108	PNNL-14019	—	After 24 hr, centrifuge slurries at 3000 rpm for 5 min (minimum). Decant supernate. Wash residual solids 2-3 times with 0.01M NaOH. After each wash, centrifuge samples and combine supernate with final leachate. Final leachate was passed through a 0.2 um syringe filter prior to analysis. Washed residual solids were dried to constant weight at 105°C and analyzed by KOH fusion in Ni crucible followed by dissolution into nitric acid.	<u>ug/g Al in treated sludge, % Al removed from oxidative alkaline leaching of washed sludge solids:</u> 1) 102630, 11%. 2) 51300, 73%. 3) 92830, 25%. 4) 23630, 86%. 5) 140630, 17%. 6) 63330, 75%.	—	—	—	—	—
241-U-109	PNNL--11908	—	Wash solids 3 times with 0.1M NaOH. After each wash, centrifuge and transfer supernate to final leachate container. The washed residual solids were dried to a constant wt at 105°C.	1) 9.2%. 2) 77%. 3) 17%. 4) 80%. 5) 24%. 6) 54%.	—	—	86800 ug Al/g initial sludge.  <u>ug Al/g final leached sludge:</u> 1)76600. 2)22300. 3) 90200. 4) 82900. 5) 77150. 6) 49500.	—	—
241-U-110	PNL-9387	<u>Residual sludge was washed with 2mL 1M K<sub>2</sub>CO<sub>3</sub>; mix sludge with K<sub>2</sub>CO<sub>3</sub> for 2 hr at RT; centrifuge; decant; and repeat. Add 2 mL 1M K<sub>2</sub>CO<sub>3</sub> to sludge; heat 5 hr at 100°C; cool; centrifuge; decant; and repeat. Wash sludge with 5 mL water at RT.</u>	Acid leaching done after K <sub>2</sub> CO <sub>3</sub> digestion - not described here.	—	Total 79% Al in NaOH leach.	Total 4% Al in K <sub>2</sub> CO <sub>3</sub> digest.	12% Al in leached sludge.	—	—
241-U-110	PNL-10078	Add 4mL 3M NaOH/2M Na <sub>2</sub> CO <sub>3</sub> to solids, mix at 100°C for 5 hr, remove samples of liquid and solid, continue to mix at ~ 20°C for 5 hr with sonication, centrifuge, and decant when finished.	Add 3mL water to solids, mix at RT for 0.5 hr, centrifuge, decant, and repeat with solids.	37%.	46% (0.38M Al before sonication; 0.42M Al after sonication).	—	—	—	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
241-U-110	PNNL-11779	Add 23 mL 3.2M NaOH to solids. Mix at 100°C for 62 hr. Cool, centrifuge, and decant.	Wash solids 3 times using 20 mL 0.1M NaOH. Mix 0.25 hr at RT, centrifuge, decant, and repeat with solids portion. All supernate from wash/leach mixed together for analysis.	—	—	—	96% Al removed from caustic leach + wash.  3% removed from first Tiron leach + wash.  1% removed from second Tiron leach.  0.2% removed from third Tiron leach + wash.  0% in solid residue.	—	Subsequent Tiron wash and leach performed in three stages. Total removal from Tiron leach was the remaining 4.2 % of Al. <u>Tiron Leach 1:</u> Add 20 mL 0.1M NaOH, 2 g Tiron, and ~1 mL 10M NaOH to get pH 13. Mix 49 hr at RT, centrifuge and decant (21.1 mL solution). <u>Tiron Wash (3 times):</u> Using 10 mL aliquots of 0.1M NaOH, mix at RT for 0.25 hr, centrifuge, and decant (29.8 mL solution). <u>Tiron Leach 2:</u> Add 20 mL 0.1M NaOH, 2 g Tiron, and ~1 mL 10M NaOH to get pH 13. Mix at RT for 124 hr, centrifuge and decant (21.4 mL solution). <u>Tiron Leach 3:</u> Add 20 mL 0.1M NaOH, 2 g Tiron, and ~1 mL 10M NaOH to get pH 13. Mix at RT for 124 hr, centrifuge, and decant (mix w/ wash (3 times) solution from next step). <u>Wash (3 times):</u> Using 10 mL aliquots of 0.1M NaOH, mixing at RT for 0.25 hr, centrifuge, and decant (51.3 mL solution). <u>Dry solids</u> at 105°C to get 0.032 g dry solids.
Group 1: BiPO <sub>4</sub> Sludge (1C and 2C).	PNNL-17992 (WTP-RPT-166)	—	Three samples (G1-40-3a, -3b, and -3c, leached at 40°C and 3M NaOH) analyzed. Slurry one of the samples with 15mL 0.01 M NaOH and divide between remaining two samples. Rinse leaching bottle with 10 mL 0.01M NaOH and split wash solution between two samples. Mix solids on shaker table for 15 min and centrifuge for 5 min. Remove supernate. Wash both again for a total of 3 washes. Slurry remaining solids in ~2mL DI and sub-divide for analysis.	Sample #, T (°C), [OH] M, ug Al/mL at t=0hr, 1hr, 2hr, 4hr, 8hr, and 24hr: <u>#1</u> , 40°C, 1M, 123, 265, 278, 284, 275, 292. <u>#2</u> , 40°C, 3M, 222, 302, 312, 313, 320, 337. <u>#3</u> , 40°C, 3M, 151, 288, 295, 308, 300, 321. <u>#4</u> , 40°C, 3M, 132, 252, 284, 300, 306, 308. <u>#5</u> , 60°C, 1M, 83.8, 275, 292, 289, 296, 300. <u>#6</u> , 60°C, 3M, 131, 292, 328, 331, 338, 331. <u>#7</u> , 80°C, 1M, 107, 294, 304, 304, 312, 302. <u>#8</u> , 80°C, 3M, 123, 319, 329, 331, 337, 333.  Total wt% Al removed: <u>#1</u> =75.9%; <u>#2</u> =87.6%; <u>#3</u> =83.6%; <u>#4</u> =80.1%; <u>#5</u> =78.1%; <u>#6</u> =86%; <u>#7</u> =78.5%; <u>#8</u> =86.6%.	—	4.73 ug/mL.	~11500 ug/g.	—	—

Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
Group 2: BiPO <sub>4</sub> Saltcake (BY and T saltcake).	PNNL-17992 (WTP-RPT-166)	—	Three samples (G2-80-3a, -3b, and -3c, leached at 80°C and 3M NaOH) analyzed. Slurry all of the solids into one sample. Wash 3 times with 0.01M NaOH. Centrifuge and decant between washes.	<p>Sample #, T (°C), [OH] M, ug Al/mL at t=0hr, 1hr, 2hr, 4hr, 8hr, and 24hr:</p> <p><b>#1</b>, 60°C, 3M, 188, 736, 921, 1095, 1191, 1256. <b>#2</b>, 80°C, 1M, 90, 691, 866, 964, 1142, 1261. <b>#3</b>, 80°C, 3M, 144, 969, 1120, 1246, 1272, 1331. <b>#4</b>, 80°C, 3M, 138, 954, 1171, 1273, 1284, 1281. <b>#5</b>, 80°C, 3M, 132, 849, 1076, 1240, 1360, 1323. <b>#6</b>, 80°C, 5M, 276, 940, 1182, 1304, 1364, 1349. <b>#7</b>, 100°C, 3M, 111, 1324, 1451, 1472, 1908, 1515.</p> <p>Total wt% Al removed: <b>#1</b>=58%; <b>#2</b>=58%; <b>#3</b>=61%; <b>#4</b>=59%; <b>#5</b>=60%; <b>#6</b>=62%; <b>#7</b>=70%.</p>	—	103.5 ug/mL.	91450 ug/g.	—	—
Group 3: PUREX CW Sludge.	PNNL-18054 (WTP-RPT-167)	<p>Test Conditions in the form of Test #, [T (°C); M free [OH]; M [Na]; M [NO<sub>3</sub>]]:</p> <p><b>#1</b>, [60°C; 0.95M; 1.09M; -]. <b>#2</b>, [60°C; 3.03M; 3.22M; -]. <b>#3</b>, [60°C; 5.15M; 5.27M; -]. <b>#4</b>, [80°C; 0.93M; 1.06M; -]. <b>#5</b>, [80°C; 3.01M; 3.21M; -]. <b>#6</b>, [80°C; 3.17M; 3.21M; -]. <b>#7</b>, [80°C; 2.93M; 3.07M; -]. <b>#8</b>, [80°C; 2.88M; 3.95M; 1.05M]. <b>#9</b>, [80°C; 2.80M; 8.04M; 5.33M]. <b>#10</b>, [80°C; 4.93M; 4.87M; -]. <b>#11</b>, [100°C; 0.94M; 1.09M; -]. <b>#12</b>, [100°C; 3.12M; 3.44M; -]. <b>#13</b>, [100°C; 5.23M; 5.61M; -].</p>	Three samples leached at 80°C in 3M NaOH were composited into two samples using 15 mL 0.01M NaOH and splitting between two remaining samples. The two samples were then washed with 15 mL aliquots of 0.01M NaOH three times, shaking for 15 minutes prior to centrifuging and decanting supernate.	<p>Three methods for fraction removed were used:</p> <p>(1) Based on initial solids/leachate solution Al;  (2) Based on final solids/leachate solution; and  (3) Based on initial/final solids.</p> <p>For fraction (3) in the form [Test #; fraction removed]:  <b>#1</b>; 0.91. <b>#2</b>; 1.03. <b>#3</b>; 0.91. <b>#4</b>; 1.04. <b>#5</b>; 1.11. <b>#6</b>; 0.94. <b>#7</b>; 0.92. <b>#8</b>; 0.83. <b>#9</b>; 0.92. <b>#10</b>; 1.06. <b>#11</b>; 0.96. <b>#12</b>; 0.99. <b>#13</b>; 1.05.</p>	—	111.1 ug/mL.	<p>Average Al in solids before leaching = 299500 ug/g.  Average Al in solids after leaching = 27100 ug/g.</p>	<p>Average initial Al in solids = 297500 ug/g.</p>	<p>Al concentration (ug/mL) in solution during parametric leaching in the form [test #, ug Al/mL, at t = 0 hr, 1hr, 2hr, 4hr, 8hr, 24hr, and 48hr]:</p> <p><b>#1</b>, 86.2, 393, 756, 1246, 1862, 2559, 3056.  <b>#2</b>, 92.3, 764, 1384, 2022, 2543, 2740, 2778.  <b>#3</b>, 118, 1039, 2120, 3075, 3457, 3581, 3557.  <b>#4</b>, 133, 1356, 2329, 3557, 3493, 3538, 3484.  <b>#5</b>, 175, 2699, 3433, 3625, 3643, 3466, 3730.  <b>#6</b>, 241, 2473, 3033, 3109, 3216, 2691, 3159.  <b>#7</b>, 204, 2430, 3037, 2982, 3054, 3001, 3071.  <b>#8</b>, 232, 2485, 2792, 2839, 2871, 2801, 2790.  <b>#9</b>, 184, 3097, 3127, 3280, 3191, 2968, 3092.  <b>#10</b>, 237, 3426, 3612, 3748, 3606, 3726, 3538. <b>#11</b>, 64.4, 2964, 3073, 3177, 3186, 3237, 3202. <b>#12</b>, 143, 3059, 3144, 3193, 3252, 3254, 3297. <b>#13</b>, 112, 3379, 3432, 3406, 3438, 3482, 3503.</p>



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
Group 4: REDOX CW Sludge.	PNNL-18054 (WTP-RPT-167)	Test Conditions in the form of [T (°C); M free [OH]; M [Na]; M [NO <sub>3</sub> ]]: 60°C; 0.98M; 1.13M; -; 60°C; 3.04M; 3.2M; -; 60°C; 5.31M; 5.49M; -; 80°C; 0.96M; 1.14M; -; 80°C; 3.17M; 3.46M; -; 80°C; 3.14M; 3.36M; -; 80°C; 3.08M; 3.4M; -; 80°C; 3.01M; 4.41M; 1.39M. 80°C; 5.67M; 11.17M; 5.66M. 80°C; 5.28M; 6.3M3; -; 100°C; 1M; 1.17M; -; 100°C; 3.42M; 3.55M; -; 100°C; 5.53M; 5.47M; -.	Three samples leached at 80°C in 3M NaOH were composited into two samples using 15 mL 0.01M NaOH and splitting between two remaining samples. The two samples were then washed with 15 mL aliquots of 0.01M NaOH three times, shaking for 15 minutes prior to centrifuging and decanting supernate.	Two methods for fraction removed were used: (1) Based on final solids/leachate solution; and (2) Based on initial/final solids.  For fraction based on (2) in the form [Test #; fraction removed]: #1; 0.65. #2; 0.75. #3; 0.74. #4; 0.81. #5; 0.59. #6; 0.84. #7; 0.91. #8; 0.93. #9; 0.83. #10; 0.80.	—	180.3 ug/mL.	Average Al in solids before leaching = 332500 ug/g. Average Al in solids after leaching = [38000] ug/g.	Average initial Al in solids = 296500 ug/g.	Al concentration (ug/mL) in solution during parametric leaching in the form [test #, ug Al/mL at 0 hr, 1hr, 2hr, 4hr, 8hr, 24hr, and 48hr]: #1, 14.6, 488, 1073, 1806, 2473, 3020, 3150. #2, 55.7, 1070, 2138, 2968, 3342, 3362, 3568. #3, 57.9, 1866, 2883, 3497, 3669, 3586, 3688. #4, 112, 2355, 2949, 3434, 3439, 3376, 3425. #5, 153, 3390, 3494, 3657, 3558, 3473, 3710. #6, 115, 3057, 3381, 3435, 3363, 3546, 3552. #7, 99.3, 2948, 3736, 3500, 3508, 3477, 3560. #8, 276, 3172, 3290, 3327, 3377, 3067, 3381. #9, <1.79, 3686, 3588, 3509, 3533, 3297, 3600. #10, 122, 3294, 3435, 3407, 3499, 3324, 3534. #11, 71.578, 3741, 3792, 3865, 3791, 3889, 3910. #12, 86.1, 3058, 3094, 3117, 3128, 3295, 3317. #13, 125, 2981, 2943, 2993, 3050, 3003, 2961.
Group 5: REDOX.	PNNL-17368 (WTP-RPT-157)	Sample #, °C, [OH], [NO <sub>3</sub> ], M: #1, 80°C, 0.97M, 0M. #2, 80°C, 3.3M, 0M. #3, 80°C, 5.06M, 0M. #4, 90°C, 0.91M, 0M. #5, 90°C, 2.94M, 0M. #6, 90°C, 2.9M, 0M. #7, 90°C, 3.02M, 0M. #8, 90°C, 3.13M, 1.1M. #9, 90°C, 3.06M, 5.35M. #10, 90°C, 5.01M, 0M. #11, 100°C, 0.86M, 0M. #12, 100°C, 2.81M, 0M. #13, 100°C, 5.11M, 0M.	Three samples (G5-90-3a, -3b, and -3c) leached at 90°C and 3M NaOH with ~0.2 mL centrifuged solids each. Slurry one of the samples with 0.01 M NaOH and divide between remaining two samples. Wash both 3 times by adding ~5 mL 0.01 M NaOH and mixing in vortex mixer for 5 minutes. Centrifuge slurries for 5 minutes and remove supernate. Combine two samples using additional 0.01 M NaOH. Centrifuge and remove aqueous phase. Slurry solids with ~4 mL DI and sub-divide for analysis.	Sample #, ug Al/mL at t=1hr, 4hr, 8hr, 24hr, 72hr, and 170hr: #1, 188, 777, 1197, 2070, 3336, 4205. #2, 243, 993, 1437, 2792, 4443, 5501. #3, 214, 1099, 1567, 2894, 4877, 5219. #4, 393, 1065, 1524, 2521, 3822, 4871. #5, 541, 1256, 1910, 3385, 4926, 5343. #6, 477, 1114, 1651, 3095, 4446, 5366. #7, 473, 1077, 1691, 3155, 4608, 5328. #8, 561, 1281, 2040, 3477, 5016, 5105. #9, 379, 1107, 1837, 3319, 4494, 4959. #10, 658, 1408, 2146, 3752, 4631, 5145. #11, 559, 1654, 2358, 3681, 4837, 5344. #12, 519, 1767, 2842, 4515, 5350, 5421. #13, 734, 2478, 3605, 5022, 5452, 5595.	—	—	83675 ug/g.	Sample # = total wt% removed: #1=77%. #2=101%. #3=96%. #4=89%. #5=98%. #6=98%. #7=98%. #8=94%. #9=91%. #10=94%. #11=98%. #12=100%. #13=103%.	—



Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
Group 6: S-Saltcake.	PNNL-17368 (WTP-RPT-157)	—	Add 60 mL 0.01 M NaOH to leached centrifuged solids. Mix for 5 min by shaking. Centrifuge at ~610 G for 15 min and remove supernate. Repeat for a total of three washes. <b>Note: Oxidative leaching performed on final caustic leached solids. This information is not included here. They performed 17 tests analyzing the effects of temperature, M [OH], and Mn/Cr mole ratio on leaching of Cr.</b>	—	—	—	~20 mL; 113500 ug/g.	—	Total of 59% Al leached from the solids by caustic leaching (with 46% removed due to oxidative leaching).
Group 7: TBP Waste Sludge.	PNNL-18119 (WTP-RPT-169)	Sample #, T (°C), [OH] M: #1, 40°C, 1.02M. #2, 40°C, 3.12M. #3, 40°C, 3M. #4, 40°C, 3.19M. #5, 60°C, 0.24M. #6, 60°C, 1.01M. #7, 60°C, 3.02M. #8, 80°C, 0.25M. #9, 80°C, 1.05M. #10, 80°C, 3.05M.	Slurries were removed from heat and cooled to ambient temperature. They were then centrifuged and decanted. Triplicate solids (samples # 2, 3, and 4) were used for wash study after caustic leach. One of the samples was slurried in ~15 mL 0.01M NaOH and divided between the other two samples. Solids were mixed for 15 min, centrifuged for 5 min, and decanted. A total of 3 washes were performed using this technique. Additional 0.01M NaOH was used to transfer solids from one bottle to combine all solids in one bottle. This was centrifuged for 5 min and decanted.	—	—	Wash solution Al composition = 5.45 ug/mL.	Average Al in solids before leaching = 15100 ug/g. Average Al in solids after leaching = [6550] ug/g.	Average initial Al in solids = 16000 ug/g.	Also performed crossflow ultrafiltration testing with caustic leaching of solids, with the addition of archived samples from 241-A Y-102. Procedure not detailed here, but total Al removed from slurry due to 4 equal volume washes was 53 wt%. Aluminum concentration (ug/mL) in solution during parametric leaching in the form [Test #, ug/mL Al at 0 hr, 1hr, 2hr, 4hr, 8hr, and 24hr]: #1, 29.2, 55.1, 57.7, 59.9, 59.2, 71. #2, 42.5, 62.2, 62.9, 66.6, 70, 88.1. #3, 38.6, 57.9, 60.7, 60.2, 65.7, 79.3. #4, 36.9, 59.8, 60.8, 63.5, 65.7, 87.1. #5, 25, 53.8, 56.5, 57.7, 60.7, 63.6. #6, 32.4, 57.8, 60.9, 68.9, 84.9, 90.5. #7, 40.2, 62.7, 73.5, 87.6, 96.2, 97.3. #8, 22.5, 62.8, 66.1, 71.7, 84.6, 99.5. #9, 30.8, 74.7, 85.5, 90.6, 89.8, 90.3. #10, 39.7, 77, 86.5, 87.9, 89, 86.6.
Group 8: FeCN Waste Sludge.	PNNL-18120 (WTP-RPT-170)	—	Wash 4 times after dewatering leached slurry. Concentration of NaOH added for wash #1 was 0.47M NaOH, wash #2 was 0.16M NaOH, wash #3 was 0.049M NaOH, and wash #4 was 0.014M NaOH, with a total volume of 1.2 L wash solution and mass of 1.2 kg.	Concentration of Al (ug/mL) in supernate calculated based on mass balance was 697 ug Al/mL. At -1.5 hr (during heat-up after addition of caustic; 40°C) had 4580 ug Al/mL. At times of 0, 2, 4, and 8 hr (during mix after reaching leaching temperature of 60°C), had 4600, 4520, 4600, and 4650 ug Al/mL respectively. The final Al concentration (after cool down to 25°C) was 4570 ug Al/mL. Total of 60 wt% Al removed.	—	—	—	Initial Composition: Supernate had 1430 ug/mL Al, Washed Solids had 89650 ug/g Al.  Final composition of leached and washed solids had 50000 ug/g Al.	—

**Table A1-2. SSTs and DSTs Wash and Leach Information for Aluminum, Part 2. (27 sheets)**

Tank	Lab Study Reference(s)	Caustic Leach #2 Procedure	Wash of solids after Caustic leach steps	Mass (ug) Al removed during 1st leach step	Mass (ug) Al removed during 2nd leach step	Mass (ug) Al removed in final wash solution	Mass (ug) Al in Leached Solids	Total mass Al (ug)	Comments
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant	PNNL-18007 (WTP-RPT-171)	—	After decanting leachate (81.7 g), the solids were washed six times with 40 mL 0.01M NaOH. Wash solution was added to the solids and mixed for 15 min, centrifuged for 10 min, and decanted.	Caustic leach solution = 15180 ug Al/mL.	—	Combined wash solution = 2540 ug Al/mL.  Final contact solution = 280 ug Al/mL.	Caustic leached and washed solids = 288000 ug Al/g.	38% leached.	—
Mixture of Group 5 and Group 6 waste. 31.6 wt% Group 5 Slurry + 16.8 wt% Group 6 slurry + 51.6 wt% Group 6 supernatant	PNNL-17965 (WTP-RPT-172)	—	The solids from sample #2 was washed 3 times by adding 15 mL 0.01M NaOH, mixed on a shaker table for 15 min, centrifuged 5 min, and decanted. The solids after the third wash were slurried in ~2mL DI for analysis.	Initial Al in sample was 0.61 g. Total dissolution of Al in samples were: #1 = 0.4 g, #2 = 0.42 g, and #4 = 0.59 g Al removed.	—	164.1 ug/mL.	Sample #2: Average Al before leaching = 612,393 ug. Average Al after leaching 24 hr= 193,907 ug.	Total wt% Al removed: Sample #1 = 65 wt%; #2 = 68 wt%; and #4 = 97 wt%.	Aluminum concentration (ug Al/mL) as a function of leaching time. Times were 0, 1, 2, 4, 8 and 24 hours:  Sample #1 concentrations: [360.6, 1224, 1570, 2269, 3063, 4006 ug Al/mL].  Sample #2 concentrations: [583.7, 1396, 1639, 2301, 3413, 4185 ug Al/mL].  Sample #4 concentrations: [830.7, N/A, 2369, 3229, 4731, 5937 ug Al/mL].
Mixture of Group 3 and Group 4 composites	PNNL-18048 (WTP-RPT-181)	—	After caustic leach and cool down, slurry was dewatered, removing ~1.9L slurry supernate. After filtration testing (leach and dewatering), the slurry was washed 3 times with 1.2 L caustic at concentrations of 1.78M NaOH, 0.78M NaOH, and 0.3M NaOH. It was re-circulated in the filter for ~30 min and then dewatered after each wash. Final dewatering was stopped at 1L due to cavitation.	By the end of the heat ramp to 100°C, 94% of the Aluminum had been leached into solution. Aluminum concentration in the aqueous phase at start of heat up (32°C) = 650 ug/mL. At 1 hr heat up (38°C) = 7800 ug/mL. At 3 hr heat up (72°C) = 24000 ug/mL. At 0 hr leach (90°C) = 25000 ug/mL. At 4 hr leach (93°C) = 25000 ug/mL. At 8 hr leach (95°C) = 25000 ug/mL.	—	—	Al in caustic leached, dewatered slurry was 40 g. 38 g were in the liquid, with 1.7 g in the solids fraction.	Pre-leached solids were 98 wt% Al. Caustic leaching removed 94 wt% Al. After washing, 75 wt% Al was removed.	—

References: <sup>1</sup> WHC-EP-0625. <sup>2</sup> PNNL-11636. <sup>3</sup> PNNL-13394. <sup>4</sup> PNWD-3300 (WTP-RPT-076). <sup>5</sup> PNL-9814.

**A2.0 SINGLE- AND DOUBLE-SHELL TANKS WITHOUT LABORATORY WASH AND LEACH INFORMATION**

A total of 108 SSTs and 23 DSTs were found to have no information concerning wash and leach of aluminum from tank solids. Information was available concerning the waste types found in these tanks, as well as two papers which had information on the potential or known aluminum solid species found in the tank sludge. This information is provided in Table A2-1 below.

**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum. (8 sheets)**

Tank	Waste Sources
DSTs	
241-AN-101	Dilute Non-Complexed Waste. <sup>1</sup> A1-SltCk (liquid and solid) and NA (liquid and solid). <sup>2</sup>
241-AN-102	CC. <sup>1</sup> A2-SltSlr (solid) and NA (liquid). <sup>2</sup> Potential Al solid species in washed solids: boehmite, Zeolite, Na aluminate, diaspore. <sup>4</sup>
241-AN-103	Double Shell Slurry. <sup>1</sup> A2-SltSlr (liquid and solid). <sup>2</sup>
241-AN-105	Double Shell Slurry Feed. <sup>1</sup> A2-SltSlr (liquid and solid). <sup>2</sup>
241-AN-106	Concentrated Phosphate Waste. <sup>1</sup> NA (liquid, sludge, and SltCk). <sup>2</sup>
241-AN-107	CC. <sup>1</sup> A2-SltSlr (liquid and solid). <sup>2</sup>
241-AP-101	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid). <sup>2</sup>
241-AP-102	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid and sludge). <sup>2</sup>
241-AP-103	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid and SltCk). <sup>2</sup>
241-AP-104	Dilute Non-Complexed Waste. <sup>1</sup> A2-SltSlr (solid) and NA (liquid). <sup>2</sup>
241-AP-105	Double Shell Slurry Feed. <sup>1</sup> A2-SltSlr (solid) and NA (liquid). <sup>2</sup>
241-AP-106	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid). <sup>2</sup>
241-AP-107	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid). <sup>2</sup>
241-AP-108	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid and SltCk). <sup>2</sup>
241-AW-101	Double Shell Slurry Feed. <sup>1</sup> A2-SltSlr (liquid and solid). <sup>2</sup>
241-AW-102	Dilute Non-Complexed Waste. <sup>1</sup> NA (liquid and sludge). <sup>2</sup>
241-AW-103	PUREX Neutralized Cladding Removal Waste (TRU). <sup>1</sup> A1-SltCk (liquid and solid), CWZr2 (solid), and NA (liquid). <sup>2</sup>
241-AW-104	Dilute Non-Complexed Waste. <sup>1</sup> A2-SltSlr (liquid and solid), PL2 (liquid and solid), and NA (liquid). <sup>2</sup>



**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-AW-105	DN/PD. <sup>1</sup> CWZr2 (solid), PL2 (solid), and NA (liquid). <sup>2</sup> Aluminum containing phases identified in sludge: gibbsite and crystalline AlSi in as-received; crystalline AlSi in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>
241-AW-106	Dilute Non-Complexed Waste. <sup>1</sup> A2-SltSlr (solid) and NA (liquid). <sup>2</sup>
241-AY-101	Dilute Complexed Waste. <sup>1</sup> NA (liquid and sludge), NA Lower (liquid), and NA Upper (liquid). <sup>2</sup>
241-AY-102	Dilute Non-Complexed Waste. <sup>1</sup> BL (liquid and solid) and NA (liquid and sludge). <sup>2</sup>
241-SY-101	CC. <sup>1</sup> S2-SltSlr (solid) and NA (liquid). <sup>2</sup> Aluminum containing phases identified in sludge: NaAlO <sub>2</sub> and amorphous gibbsite in as-received; none in leached sludge (determined by TEM/SEM/XRD). <sup>3</sup>
SSTs	
241-A-101	Double Shell Slurry Feed. <sup>1</sup> A1-SltCk (liquid and solid) and P2 (solid). <sup>2</sup> Primary: double-shell slurry feed. Secondary: non-complexed waste. Tertiary: evaporator feed. <sup>5</sup>
241-A-102	Double Shell Slurry Feed. <sup>1</sup> A1-SltCk (liquid and solid). <sup>2</sup> Primary: double-shell slurry feed. Secondary: non-complexed waste. Tertiary: evaporator feed. <sup>5</sup>
241-A-103	Double Shell Slurry Feed. <sup>1</sup> A1-SltCk (liquid and solid) and AR (solid). <sup>2</sup> Primary: double-shell slurry feed. Secondary: non-complexed waste. Tertiary: evaporator feed. <sup>5</sup>
241-A-104	Non-Complexed Waste. <sup>1</sup> AR (solid) and P1 (solid). <sup>2</sup> Primary: SLUCE. Secondary: neutralized acid waste. Tertiary: H <sub>2</sub> O. Other: high-level waste from waste fractionation process at B plant starting in 1967. <sup>5</sup>
241-A-105	Non-Complexed Waste. <sup>1</sup> P2 (solid). <sup>2</sup> Primary: neutralized acid waste. Secondary: IX. <sup>5</sup>
241-A-106	Double Shell Slurry Feed. <sup>1</sup> A1-SltCk (solid), AR (solid), and SRR (solid). <sup>2</sup> Primary: CC. Secondary: non-complexed waste. Tertiary: evaporator feed. Other: high-level waste from waste fractionation process at B plant starting in 1967. <sup>5</sup>
241-AX-101	Double Shell Slurry Feed. <sup>1</sup> A1-SltCk (liquid and solid) and SRR (solid). <sup>2</sup> Primary: double-shell slurry feed. Secondary: non-complexed waste. Tertiary: evaporator feed. <sup>5</sup>
241-AX-102	Complexant Concentrate Waste. <sup>1</sup> A1-SltCk (solid) and B (solid). <sup>2</sup> Primary: CC. Secondary: double-shell slurry feed. Tertiary: evaporator feed. <sup>5</sup>
241-AX-103	Complexant Concentrate Waste. <sup>1</sup> A1-SltCk (liquid and solid) and P2 (solid). <sup>2</sup> Primary: CC. Secondary: double-shell slurry feed. Tertiary: evaporator feed. <sup>5</sup>
241-AX-104	Non-Complexed Waste. <sup>1</sup> P2 (solid). <sup>2</sup> Primary: evaporator feed. Secondary: non-complexed waste. Tertiary: neutralized acid waste. <sup>5</sup>
241-B-102	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid). <sup>2</sup> Primary: CW. Secondary: EB. Tertiary: B Plant low-level waste. Other: IX waste. <sup>5</sup>

**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-B-103	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid) and MW (solid). <sup>2</sup> Primary: CW. Secondary: EB. Tertiary: IX waste. Other: mixture of several miscellaneous wastes. <sup>5</sup>
241-B-105	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid), 1C (solid), and 2C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-B-107	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid), 1C (solid), and CWP2 (solid). <sup>2</sup> Primary: 1C. Secondary: EB. Tertiary: CW. Other: TBP. <sup>5</sup>
241-B-108	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid) and CWP2 (solid). <sup>2</sup> Primary: 1C. Secondary: EB. Tertiary: CW. Other: IX/TBP. <sup>5</sup>
241-B-109	Non-Complexed Waste. <sup>1</sup> B-SltCk (liquid and solid) and CWP2 (solid). <sup>2</sup> Primary: 1C. Secondary: EB. Tertiary: CW. Other: IX. <sup>5</sup>
241-B-112	BY-SltCk (liquid and solid) and 2C (solid). <sup>2</sup> Primary: 2C. Secondary: high-level B Plant waste. Tertiary: Fission Products Waste. Other: EB/in-tank solidification. <sup>5</sup>
241-B-203	224-2 (solid) and NA (liquid). <sup>2</sup> Primary: lanthanum fluoride decontamination waste. <sup>5</sup>
241-B-204	224-2 (solid) and NA (liquid). <sup>2</sup> Primary: lanthanum fluoride decontamination waste. <sup>5</sup>
241-BX-101	Non-Complexed Waste. <sup>1</sup> BL (solid), CWP2 (solid), and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: B plant low level waste. Other: IX. <sup>5</sup>
241-BX-102	Non-Complexed Waste. <sup>1</sup> CWP2 (solid), DE (solid), and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: B plant low level waste. Other: diatomaceous earth. <sup>5</sup>
241-BX-104	Non-Complexed Waste. <sup>1</sup> CWP2 (solid), CWR1 (liquid and solid), MW1 (solid), and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: IX. Other: high-level REDOX waste. <sup>5</sup>
241-BX-106	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid), CWP2 (solid), and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: EB/IX. Other: B plant low level waste. <sup>5</sup>
241-BX-108	1C (solid) and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: IC. Other: IX. <sup>5</sup>
241-BX-111	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and 1C (solid). <sup>2</sup> Primary: 1C. Secondary: EB/in-tank solidification. Tertiary: CW. Other: IX. <sup>5</sup>
241-BY-101	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and PFeCN (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: CW. Other: 1C. <sup>5</sup>
241-BY-102	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid). <sup>2</sup> Primary: TBP. Secondary: EB/in-tank solidification. Tertiary: CW. Other: 1C. <sup>5</sup>
241-BY-103	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and CWP2 (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: neutralized acid waste. Other: CW/OWW. <sup>5</sup>

**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-BY-105	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid), PFeCN (solid), and Portland Cement (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: CW. <sup>5</sup>
241-BY-106	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and PFeCN (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: CW. <sup>5</sup>
241-BY-107	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and PFeCN (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: CW. <sup>5</sup>
241-BY-109	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and CWP2 (solid). <sup>2</sup> Primary: TBP. Secondary: EB/in-tank solidification. Tertiary: CW. Other: metal waste. <sup>5</sup>
241-BY-111	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: OWW. Other: CW. <sup>5</sup>
241-BY-112	Non-Complexed Waste. <sup>1</sup> BY-SltCk (liquid and solid) and MW2 (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: EB/in-tank solidification. Tertiary: CW. <sup>5</sup>
241-C-101	Non-Complexed Waste. <sup>1</sup> CWP1 (solid) and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: CW. Tertiary: neutralized acid waste. Other: OWW. <sup>5</sup>
241-C-110	Dilute Complexed Waste. <sup>1</sup> IC (solid). <sup>2</sup> Primary: IC. Secondary: TBP. Tertiary: OWW. Other: EB/IX. <sup>5</sup>
241-C-111	Non-Complexed Waste. <sup>1</sup> IC (solid), CWP1 (solid), HS (solid), and TFeCN (solid). <sup>2</sup> Primary: TBP/FeCN-scavenged waste. Secondary: IC. Tertiary: CW. Other: hot semiworks waste. <sup>5</sup>
241-C-201	HS (solid) and NA (liquid). <sup>2</sup> Primarily hot semiworks waste. <sup>5</sup>
241-C-202	HS (solid) and NA (liquid). <sup>2</sup> Primarily hot semiworks waste. <sup>5</sup>
241-C-203	HS (solid) and NA (liquid). <sup>2</sup> Primarily hot semiworks waste. <sup>5</sup>
241-C-204	HS (solid) and NA (liquid). <sup>2</sup> Primarily hot semiworks waste. <sup>5</sup>
241-S-102	Double Shell Slurry Feed. <sup>1</sup> NA (SltCk) and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: double-shell slurry feed. <sup>5</sup>
241-S-103	Double Shell Slurry Feed. <sup>1</sup> S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: double-shell slurry feed. <sup>5</sup>
241-S-105	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid) and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>
241-S-106	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>



**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-S-108	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid) and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>
241-S-109	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid) and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>
241-S-112	Non-Complexed Waste. <sup>1</sup> R1 (solid) and NA (liquid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>
241-SX-102	Double Shell Slurry Feed. <sup>1</sup> S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: REDOX IX. <sup>5</sup>
241-SX-103	Non-Complexed Waste. <sup>1</sup> R-SltCk (liquid and solid), S1-SltCk (liquid and solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: CW. Other: OWW. <sup>5</sup>
241-SX-104	Double Shell Slurry Feed. <sup>1</sup> R-SltCk (liquid and solid), S1-SltCk (liquid and solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: REDOX IX. <sup>5</sup>
241-SX-105	Double Shell Slurry Feed. <sup>1</sup> S1-SltCk (liquid and solid), R1 (solid), and R2 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: REDOX IX. Other waste type: Hanford Laboratory Operations. <sup>5</sup>
241-SX-106	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid) and S2-SltSlr (liquid and solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: REDOX IX. Other: Laboratory waste from the 300 area and mixed waste. <sup>5</sup>
241-SX-107	Non-Complexed Waste. <sup>1</sup> R1 (solid) and R2 (solid). <sup>2</sup> Primarily high-level REDOX waste. <sup>5</sup>
241-SX-109	Non-Complexed Waste. <sup>1</sup> R-SltCk (solid), R1 (solid), and R2 (solid). <sup>2</sup> Primarily high-level REDOX waste. <sup>5</sup>
241-SX-110	Non-Complexed Waste. <sup>1</sup> R-SltCk (solid) and R2 (solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: REDOX IX. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-SX-111	Non-Complexed Waste. <sup>1</sup> R-SltCk (solid), R1 (solid), and R2 (solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: REDOX IX. <sup>5</sup>
241-SX-112	Non-Complexed Waste. <sup>1</sup> R1 (solid) and R2 (solid). <sup>2</sup> Primarily high-level REDOX waste. <sup>5</sup>
241-SX-114	Non-Complexed Waste. <sup>1</sup> R-SltCk (liquid and solid), R1 (solid), and R2 (solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: REDOX IX. Tertiary: EB. <sup>5</sup>
241-SX-115	Non-Complexed Waste. <sup>1</sup> R2 (solid). <sup>2</sup> Primarily high-level REDOX waste. <sup>5</sup>

**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-T-101	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and CWR2 (solid). <sup>2</sup> Primary: CW. Secondary: mixture of several miscellaneous wastes. Tertiary: TBP/FeCN-scavenged waste. Other: evaporator feed. <sup>5</sup>
241-T-102	Non-Complexed Waste. <sup>1</sup> CWP2 (solid), MW2 (solid), and CSR (liquid). <sup>2</sup> Primary: CW. Secondary: mixture of several miscellaneous wastes. Tertiary: IX. <sup>5</sup>
241-T-103	CWP2 (solid), CWR1 (solid), MW2 (solid), and CSR (liquid). <sup>2</sup> Primary: CW. Secondary: mixture of several miscellaneous wastes. <sup>5</sup>
241-T-105	Non-Complexed Waste. <sup>1</sup> 1C (solid), 2C (solid), and CWR1 (solid). <sup>2</sup> Primary: 1C. Secondary: CW. Tertiary: 2C. Other: B Plant low-level waste/IX. <sup>5</sup>
241-T-106	Non-Complexed Waste. <sup>1</sup> 1C (solid), CWR1 (solid), and CWR2 (solid). <sup>2</sup> Primary: 1C. Secondary: CW. Tertiary: 2C. Other: mixture of several miscellaneous wastes. <sup>5</sup>
241-T-108	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid) and 1C (solid). <sup>2</sup> Primary: 1C. Secondary: TBP. Tertiary: EB. Other: Hanford Laboratory Operations. <sup>5</sup>
241-T-109	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid). <sup>2</sup> Primary: TBP. Secondary: EB. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-T-112	224-2 (solid), 2C (solid), CSR (liquid), and DW (liquid). <sup>2</sup> Primary: 2C. Secondary: Lanthanum fluoride decontamination waste. Tertiary: decontamination waste. Other: mixture of several miscellaneous wastes. <sup>5</sup>
241-T-201	224-1 (liquid and solid). <sup>2</sup> Primarily lanthanum fluoride decontamination waste. <sup>5</sup>
241-T-202	224-2 (solid). <sup>2</sup> Primarily lanthanum fluoride decontamination waste. <sup>5</sup>
241-T-203	224-2 (solid). <sup>2</sup> Primarily lanthanum fluoride decontamination waste. <sup>5</sup>
241-T-204	224-2 (solid). <sup>2</sup> Primarily lanthanum fluoride decontamination waste. <sup>5</sup>
241-TX-101	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), MW2 (solid), R1 (solid), and Z (solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: mixture of several miscellaneous wastes. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-TX-102	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and MW2 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-TX-103	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid) and T2-SltCk (liquid and solid). <sup>2</sup> Primary: TBP. Secondary: EB. <sup>5</sup>
241-TX-104	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-TX-105	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and MW2 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>

**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-TX-106	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), MW2 (solid), and R1 (solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-TX-107	Non-Complexed Waste. <sup>1</sup> R-SltCk (liquid and solid) and T2-SltCk (liquid and solid). <sup>2</sup> Primary: High-level REDOX waste. Secondary: EB. <sup>5</sup>
241-TX-108	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), MW2 (solid), and TBP (solid). <sup>2</sup> Primary: EB. Secondary: TBP. Tertiary: high-level REDOX waste. <sup>5</sup>
241-TX-109	Non-Complexed Waste. <sup>1</sup> 1C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. Tertiary: TBP. <sup>5</sup>
241-TX-110	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and 1C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. Tertiary: TBP. <sup>5</sup>
241-TX-111	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and 1C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. Tertiary: TBP. <sup>5</sup>
241-TX-112	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid) and T2-SltCk (liquid and solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-TX-113	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and 1C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-TX-114	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid), T2-SltCk (liquid and solid), and 1C (solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-TX-115	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and TBP (solid). <sup>2</sup> Primary: EB. Secondary: high-level REDOX waste. Tertiary: CW. Other: decontamination waste. <sup>5</sup>
241-TX-116	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid), T2-SltCk (solid), and DE (solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-TX-117	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid), T2-SltCk (solid), and DE (solid). <sup>2</sup> Primary: EB. Secondary: 1C. <sup>5</sup>
241-TX-118	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid) and NA (SltCk). <sup>2</sup> Primary: EB. Secondary: TBP. Tertiary: CW. Other: 1C. <sup>5</sup>
241-TY-101	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid) and 1CFeCN (solid). <sup>2</sup> Primary: 1C/FeCN-scavenged waste. Secondary: EB. Tertiary: TBP. Other: high-level REDOX waste. <sup>5</sup>
241-TY-102	Non-Complexed Waste. <sup>1</sup> T1-SltCk (liquid and solid) and T2-SltCk (liquid and solid). <sup>2</sup> Primary: EB. Secondary: 1C. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>



**Table A2-1. SSTs and DSTs with no wash and leach information available for Aluminum.  
(8 sheets)**

Tank	Waste Sources
241-TY-103	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), 1CFeCN (solid), and TBP (solid). <sup>2</sup> Primary: TBP. Secondary: 1C/FeCN-scavenged waste. Tertiary: CW. Other: high-level REDOX waste/ mixture of several miscellaneous wastes. <sup>5</sup>
241-TY-105	Non-Complexed Waste. <sup>1</sup> TBP (solid). <sup>2</sup> Primarily TBP. <sup>5</sup>
241-TY-106	Non-Complexed Waste. <sup>1</sup> DE (solid) and TBP (solid). <sup>2</sup> Primarily TBP. <sup>5</sup>
241-U-101	Non-Complexed Waste. <sup>1</sup> R1 (solid). <sup>2</sup> Primarily high-level REDOX waste. <sup>5</sup>
241-U-102	Non-Complexed Waste. <sup>1</sup> T2-SltCk (liquid and solid), S2-SltSlr (solid), and R1 (solid). <sup>2</sup> Primary: EB. Secondary: high-level REDOX waste. <sup>5</sup>
241-U-103	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid), S2-SltSlr (solid), and R1 (solid). <sup>2</sup> Primary: EB. Secondary: high-level REDOX waste. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-U-104	Non-Complexed Waste. <sup>1</sup> DE (solid) and MW2 (solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: diatomaceous earth. <sup>5</sup>
241-U-105	Non-Complexed Waste. <sup>1</sup> S2-SltSlr (liquid and solid), T2-SltCk (liquid and solid), and CWR1 (solid). <sup>2</sup> Primary: EB. Secondary: CW. Tertiary: high-level REDOX waste. <sup>5</sup>
241-U-106	Non-Complexed Waste. <sup>1</sup> S1-SltCk (liquid and solid). <sup>2</sup> Primary: EB. Secondary: high-level REDOX waste. Tertiary: B Plant low-level waste. Other: Low-level waste from PUREX plant. <sup>5</sup>
241-U-107	Double Shell Slurry Feed. <sup>1</sup> S2-SltSlr (liquid and solid), T2-SltCk (liquid and solid), and CWR1 (solid). <sup>2</sup> Primary: EB. Secondary: CW. Tertiary: mixture of several miscellaneous wastes. <sup>5</sup>
241-U-111	Double Shell Slurry Feed. <sup>1</sup> S1-SltCk (liquid and solid), S2-SltSlr (liquid and solid), 1C (solid), and R1 (solid). <sup>2</sup> Primary: EB. Secondary: high-level REDOX waste. Tertiary: 1C. <sup>5</sup>
241-U-112	Non-Complexed Waste. <sup>1</sup> 1C (solid), CWR1 (solid), and R1 (solid). <sup>2</sup> Primary waste type is unknown. <sup>5</sup>
241-U-201	CWR1 (liquid and solid). <sup>2</sup> Primarily CW. <sup>5</sup>
241-U-202	CWR1 (liquid and solid). <sup>2</sup> Primarily CW. <sup>5</sup>
241-U-203	CWR1 (liquid and solid). <sup>2</sup> Primarily CW. <sup>5</sup>
241-U-204	CWR1 (liquid and solid). <sup>2</sup> Primary: high-level REDOX waste. Secondary: 2C. Tertiary: CW. <sup>5</sup>
Superscript References: <sup>1</sup> WHC-EP-0625. <sup>2</sup> WRPS, 2010. <sup>3</sup> PNNL-13394. <sup>4</sup> PNWD-3300 (WTP-RPT-076). <sup>5</sup> PNL-9814.	

### **A3.0 SINGLE- AND DOUBLE-SHELL TANK LABORATORY CONCENTRATION VERSUS TIME INFORMATION.**

Concentration versus time data was compiled from four laboratory studies (see Table A3-1) on seven separate SSTs. This information was then plotted (see Figure A3-1 through Figure A3-14) to observe correlations in how the percent removal of aluminum was affected due to changes in the solution caustic concentration, leaching temperature, and/or time for the various tanks. Much variation occurred between the different tank samples analyzed, with all of the analyses showing a positive amount of aluminum removal.

**Table A3-1. Caustic Leaching Aluminum Concentration versus Time Data (2 sheets).**

Reference	Tank, NaOH [M], T (°C)	Liquid to solid ratio	Time (hr)	% Al removal from wash and leach	% Al removal (summation method)	—	5 hr (ug/mL)	24 hr (ug/mL)	72 hr (ug/mL)	168 hr (ug/mL)	—	—	—	—	5 hr (%Al)	24 hr (%Al)	72 hr (%Al)	168 hr (%Al)	—	—	—
PNNL-12026 <sup>(a)</sup>	B-101, 1.1M, 60°C	5 mL leachate per g as-received sludge	168	56	42	—	1600	2000	2000	1900	—	—	—	—	35.4	44.2	44.2	42.0	—	—	—
PNNL-12026 <sup>(a)</sup>	B-101, 3.2M, 60°C	Same as above.	168	63	50	—	2400	2400	2700	2600	—	—	—	—	46.2	46.2	51.9	50.0	—	—	—
PNNL-12026 <sup>(a)</sup>	B-101, 1.0M, 100°C	Same as above.	168	59	46	—	2000	2200	2000	2300	—	—	—	—	40.0	44.0	40.0	46.0	—	—	—
PNNL-12026 <sup>(a)</sup>	B-101, 3.1M, 100°C	Same as above.	168	62	50	—	2400	2600	2500	2600	—	—	—	—	46.2	50.0	48.1	50.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 1.2M, 60°C	10 mL leachate per g as-received sludge	168	95	95	—	1892	2712	3325	3529	—	—	—	—	50.9	73.0	89.5	95.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 3.2M, 60°C	Same as above.	168	99	99	—	3299	3458	3546	3529	—	—	—	—	92.5	97.0	99.5	99.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 1.2M, 80°C	Same as above.	168	98	98	—	2849	3189	3444	3481	—	—	—	—	80.2	89.8	97.0	98.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 3.1M, 80°C	Same as above.	168	97	98	—	3254	3657	3283	3303	—	—	—	—	96.5	108.5	97.4	98.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 1.2M, 95°C	Same as above.	168	99	99	—	3335	3394	3578	3321	—	—	—	—	99.4	101.2	106.7	99.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-110, 3.2M, 95°C	Same as above.	168	99	99	—	3301	3483	3548	3532	—	—	—	—	92.5	97.6	99.4	99.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 1.1M, 60°C	15 mL leachate per g untreated solids	168	64	62	—	1718	1691	1525	1434	—	—	—	—	74.3	73.1	65.9	62.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 2.9M, 60°C	Same as above.	168	69	68	—	2192	2082	1790	1415.8	—	—	—	—	105.3	100.0	86.0	68.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 1.3M, 80°C	Same as above.	168	56	54	—	1727	1412	1443	1394	—	—	—	—	66.9	54.7	55.9	54.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 3.4M, 80°C	Same as above.	168	65	63	—	2039	1683	1492	1470	—	—	—	—	87.4	72.1	63.9	63.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 1.1M, 100°C	Same as above.	168	53	51	—	1431	1293	1323	1268	—	—	—	—	57.6	52.0	53.2	51.0	—	—	—
PNNL-12026 <sup>(a)</sup>	BX-112, 3.4M, 100°C	Same as above.	168	61	59	—	1604	1462	1457	1445	—	—	—	—	65.5	59.7	59.5	59.0	—	—	—
PNNL-12026 <sup>(a)</sup>	C-102, 1.1M, 60°C	5.56 mL leachate per g dry washed solids	72	27	27	—	5185	—	6273	—	—	—	—	—	22.3	—	27.0	—	—	—	—
PNNL-12026 <sup>(a)</sup>	C-102, 2.9M, 60°C	3.67 mL leachate per g dry washed solids	72	95	95	—	12030	—	16260	—	—	—	—	—	70.3	—	95.0	—	—	—	—
PNNL-12026 <sup>(a)</sup>	C-102, 1.0M, 100°C	12.2 mL leachate per g dry washed solids	72	20	20	—	8524	—	9406	—	—	—	—	—	18.1	—	20.0	—	—	—	—
PNNL-12026 <sup>(a)</sup>	C-102, 2.9M, 100°C	6.31 mL leachate per g dry washed solids	72	95	95	—	24160	—	30620	—	—	—	—	—	75.0	—	95.0	—	—	—	—
PNNL-12026 <sup>(a)</sup>	S-101, 1M, 70°C	30 mL leachate per g solids	168	70	66	—	86.3	501	860	1260	—	—	—	—	4.5	26.2	45.0	66.0	—	—	—
PNNL-12026 <sup>(a)</sup>	S-101, 3M, 70°C	14.9 mL leachate per g solids	168	63	59	—	334	1170	1170	3100	—	—	—	—	6.4	22.3	22.3	59.0	—	—	—
PNNL-12026 <sup>(a)</sup>	S-101, 1M, 95°C	8.5 mL leachate per g solid	168	88	87	—	1620	2640	3610	4950	—	—	—	—	28.5	46.4	63.4	87.0	—	—	—
PNNL-12026 <sup>(a)</sup>	S-101, 3M, 95°C	4.5 mL leachate per g solid	168	90	89	—	4670	9950	10900	13400	—	—	—	—	31.0	66.1	72.4	89.0	—	—	—

(a) Amount removed from dilute hydroxide-washed solids after leaching for 168 hours

Reference	Tank, NaOH [M], T (°C)	Liquid to solid ratio	Time (hr)	% Al removal from wash and leach	% Al removal (summation method)	—	5 hr (ug/mL)	24 hr (ug/mL)	72 hr (ug/mL)	168 hr (ug/mL)	Final Wash (ug/mL)	—	—	—	5 hr (%Al)	24 hr (%Al)	72 hr (%Al)	168 hr (%Al)	—	—	—
ORNL/TM-13655	S-101, 1M, 70°C	30 mL leachate per g solid	168	78.4	75.7 <sup>(b)</sup>	—	86.3	501	860	1260	44.9	—	—	—	6.2	35.8	61.4	78.4	—	—	—
ORNL/TM-13655	S-101, 1M, 95°C	14.9 mL per g solid	168	89	87.6 <sup>(b)</sup>	—	1620	2640	3610	4950	75.3	—	—	—	29.1	47.5	64.9	89.0	—	—	—
ORNL/TM-13655	S-101, 3M, 70°C	8.5 mL per g solid	168	64.9	60.6 <sup>(b)</sup>	—	334	1170	3100	5680	469	—	—	—	3.8	13.4	35.4	64.9	—	—	—
ORNL/TM-13655	S-101, 3M, 95°C	4.5 mL per g solid	168	91.5	90.4 <sup>(b)</sup>	—	4670	9950	10900	13400	104	—	—	—	31.9	67.9	74.4	91.5	—	—	—

(b) Amount removed by leach/wash step only.

Reference	Tank, NaOH [M], T (°C)	Liquid to solid ratio	Time (hr)	% Al removal from wash and leach	—	—	4 hr (ug/g)	8 hr (ug/g)	24 hr (ug/g)	72 hr (ug/g)	168 hr (ug/g)	168 hr (ug/g)	Final Wash (ug/g)	—	4 hr (%Al)	8 hr (%Al)	24 hr (%Al)	72 hr (%Al)	168 hr (%Al)	168 hr (%Al)	Final Wash (%Al)
PNNL-13702	S-110, 1M, 60°C	~80 mL solution per g washed solids	168	39	—	—	542	715	919	1262	1811	1739	178	—	12.2	16.0	20.6	28.3	40.6	39.0	4.0
PNNL-13702	S-110, 2.8M, 60°C	Same as above.	168	47	—	—	673	778	932	1267	1892	1870	139	—	16.9	19.6	23.4	31.8	47.6	47.0	3.5
PNNL-13702	S-110, 4.8M, 60°C	Same as above.	168	50	—	—	626	701	830	1201	1926	1896	203	—	16.5	18.5	21.9	31.7	50.8	50.0	5.4



**Table A3-1. Caustic Leaching Aluminum Concentration versus Time Data (2 sheets).**

PNNL-13702	S-110, 0.9M, 80°C	Same as above.	168	69	—	—	758	1042	1699	2502	3003	3056	137	—	17.1	23.5	38.4	56.5	67.8	69.0	3.1
PNNL-13702	S-110, 2.7M, 80°C	Same as above.	168	91	—	—	870	1029	1794	2907	3757	3786	186	—	20.9	24.7	43.1	69.9	90.3	91.0	4.5
PNNL-13702	S-110, 4.6M, 80°C	Same as above.	168	96	—	—	855	1032	1880	2911	3788	3670	178	—	22.4	27.0	49.2	76.1	99.1	96.0	4.7
PNNL-13702	S-110, 0.8M, 100°C	Same as above.	168	91	—	—	1253	1823	2781	3550	4087	4031	139	—	28.3	41.2	62.8	80.1	92.3	91.0	3.1
PNNL-13702	S-110, 2.7M, 100°C	Same as above.	168	100	—	—	1428	2059	3186	4038	4167	4150	138	—	34.4	49.6	76.8	97.3	100.4	100.0	3.3
PNNL-13702	S-110, 4.6M, 100°C	Same as above.	168	100	—	—	1462	2239	3499	3951	3891	4043	134	—	36.2	55.4	86.5	97.7	96.2	100.0	3.3
Reference	Tank, NaOH [M], T (°C)	Liquid to solid ratio	Time (hr)	% Al removal from wash and leach	—	—	4 hr (ug/g)	8 hr (ug/g)	24 hr (ug/g)	72 hr (ug/g)	168 hr (ug/g)	168 hr (ug/g)	Final Wash (ug/g)	—	4 hr (%Al)	8 hr (%Al)	24 hr (%Al)	72 hr (%Al)	168 hr (%Al)	168 hr (%Al)	Final Wash (%Al)
PNNL-13956	T-110, 0.6M, 60°C	~20 mL solution per g washed solids	168	27	—	—	5.4	5.6	5.4	5	5.1	3.6	2.3	—	28.6	29.6	28.6	26.5	27.0	19.1	12.2
PNNL-13956	T-110, 2.5M, 60°C	Same as above.	168	42	—	—	11.3	12.1	11.9	9	8.5	8	0	—	55.8	59.8	58.8	44.5	42.0	39.5	0.0
PNNL-13956	T-110, 3.8M, 60°C	Same as above.	168	59	—	—	10.7	12.6	13.5	13.4	15.2	11.3	5.8	—	41.5	48.9	52.4	52.0	59.0	43.9	22.5
PNNL-13956	T-110, 0.6M, 80°C	Same as above.	168	53	—	—	4.1	4.3	3.9	3.7	8.3	4.2	6.3	—	26.2	27.5	24.9	23.6	53.0	26.8	40.2
PNNL-13956	T-110, 2.7M, 80°C	Same as above.	168	67	—	—	11.6	12.5	11.5	12.4	14.3	12.1	7.3	—	54.3	58.6	53.9	58.1	67.0	56.7	34.2
PNNL-13956	T-110, 4.2M, 80°C	Same as above.	168	83	—	—	11.7	12.1	12.8	13.1	15	13.5	9	—	64.7	67.0	70.8	72.5	83.0	74.7	49.8
PNNL-13956	T-110, 0.6M, 100°C	Same as above.	168	50	—	—	4.6	5.2	5	4.5	8.8	2.7	6.4	—	26.1	29.5	28.4	25.6	50.0	15.3	36.4
PNNL-13956	T-110, 2.5M, 100°C	Same as above.	168	75	—	—	19.1	12.6	13.1	13.9	13.1	11.6	7.2	—	109.4	72.1	75.0	79.6	75.0	66.4	41.2
PNNL-13956	T-110, 4.3M, 100°C	Same as above.	168	77	—	—	10.4	14.4	14.1	14.8	17.9	14.1	3.7	—	44.7	61.9	60.7	63.7	77.0	60.7	15.9



Figure A3-1. Compilation of Laboratory Data for Percent Aluminum Removal versus Leaching Time

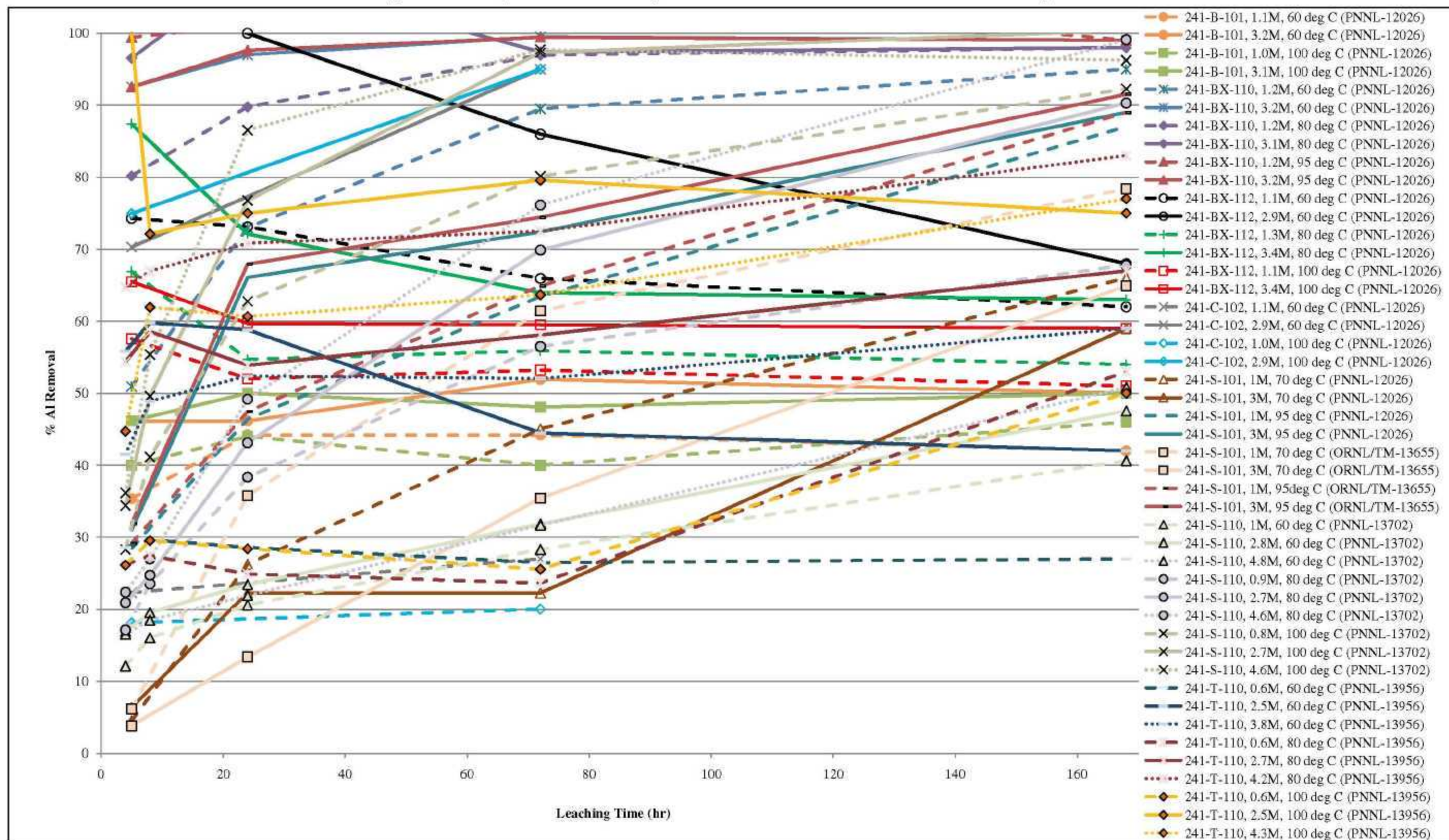




Figure A3-2. All caustic leaching analyses for Tank 241-B-101.

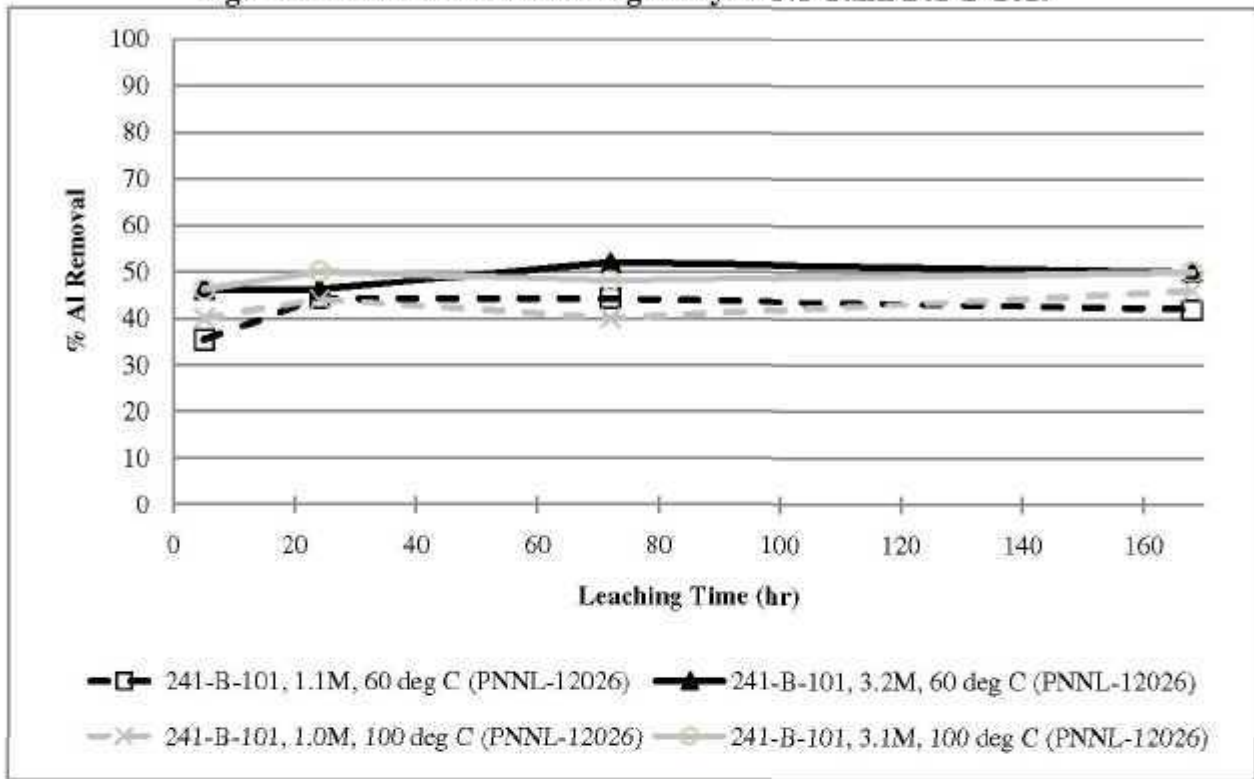


Figure A3-3. All caustic leaching analyses for Tank 241-BX-110.

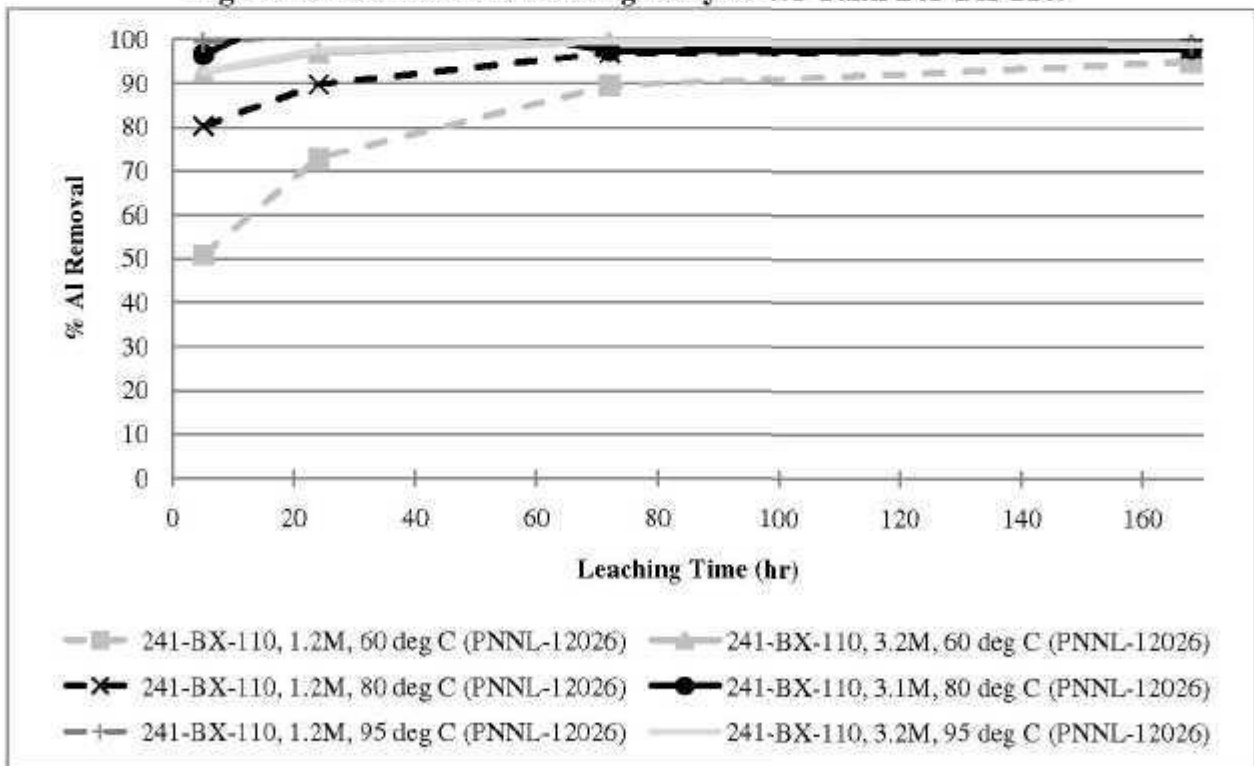




Figure A3-4. All caustic leaching analyses for Tank 241-BX-112.

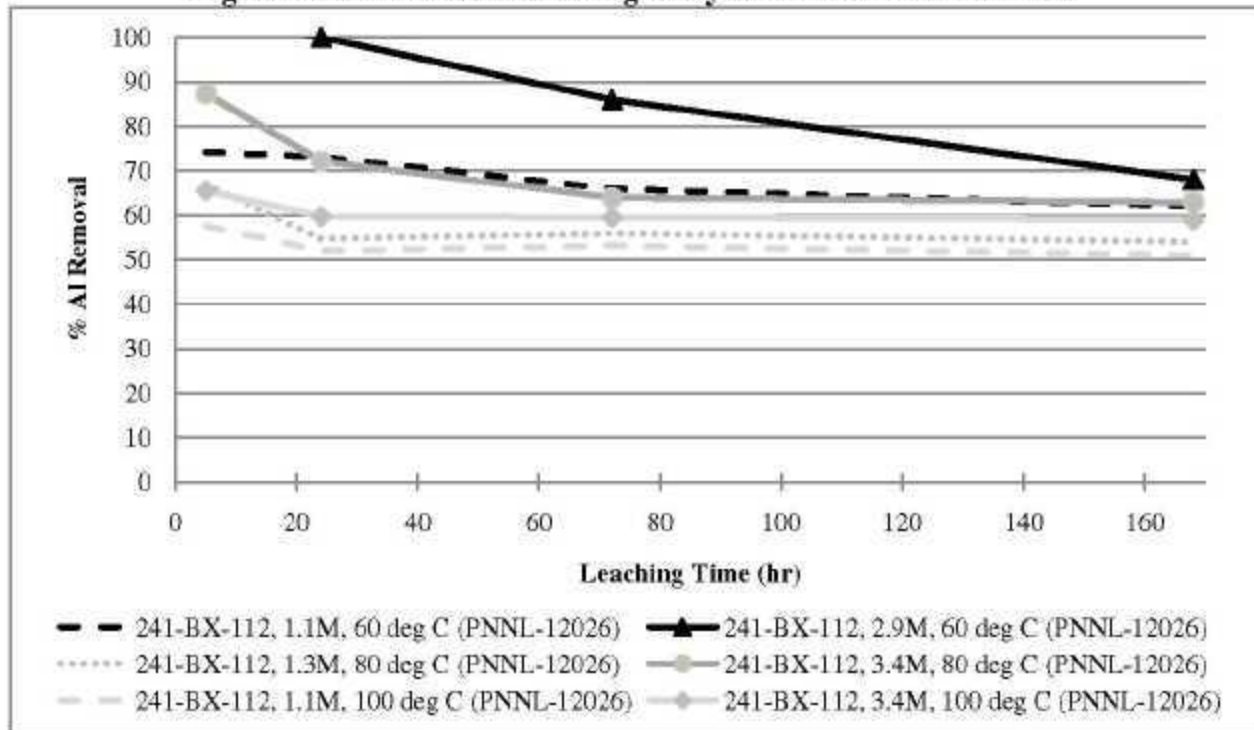


Figure A3-5. All caustic leaching analyses for Tank 241-C-102.

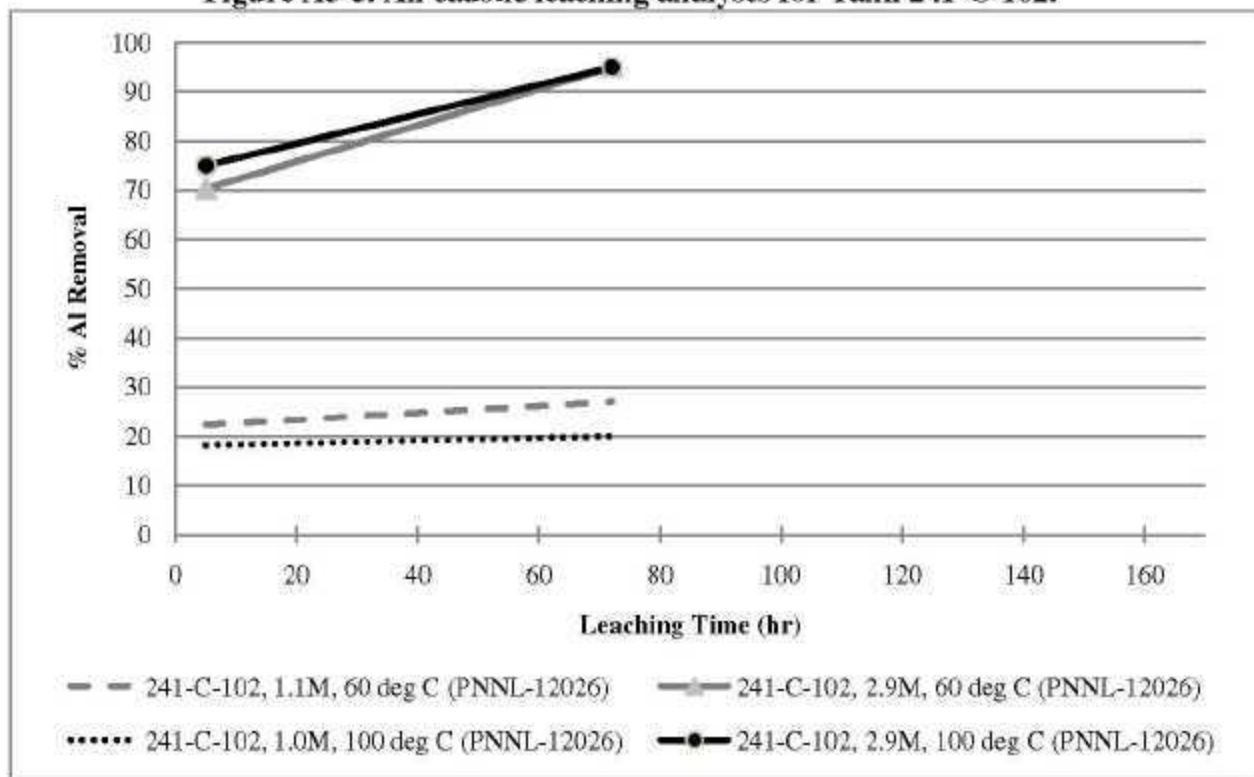


Figure A3-6. All caustic leaching analyses for Tank 241-S-101.

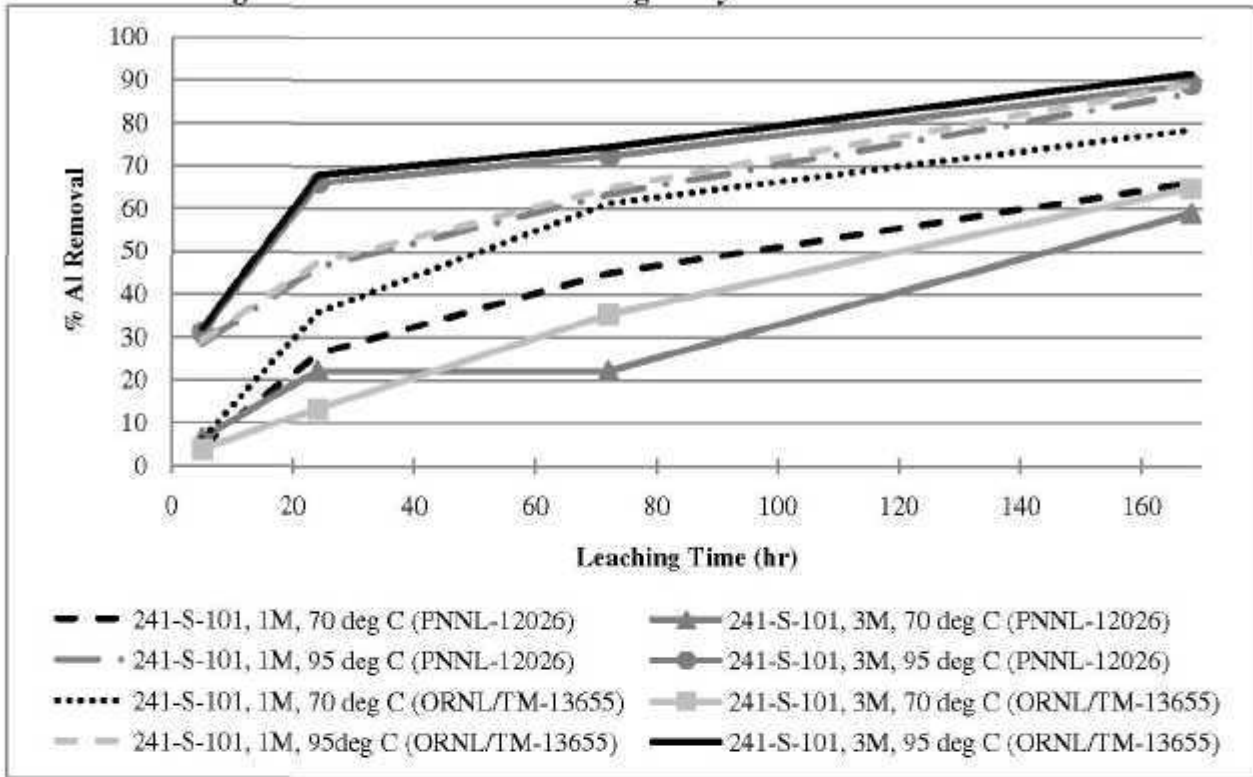


Figure A3-7. All caustic leaching analyses for Tank 241-S-110 .

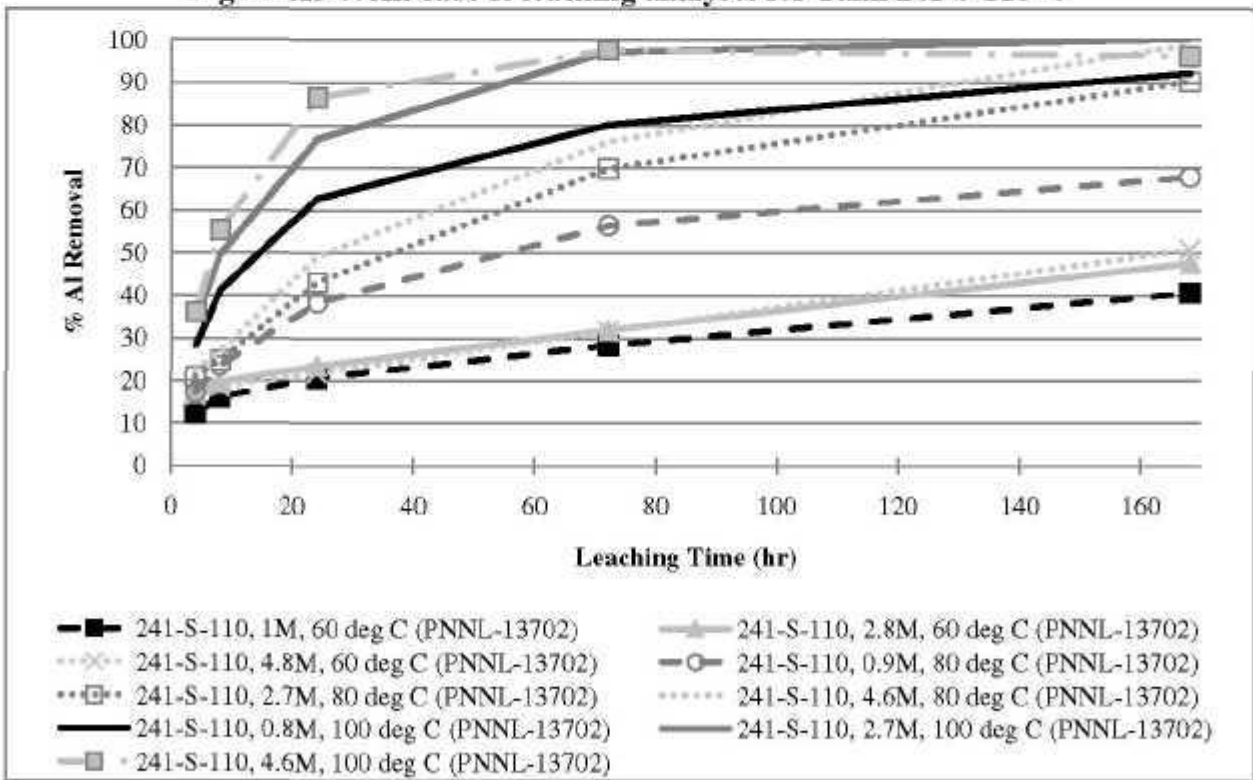


Figure A3-8. All caustic leaching analyses for Tank 241-T-110.

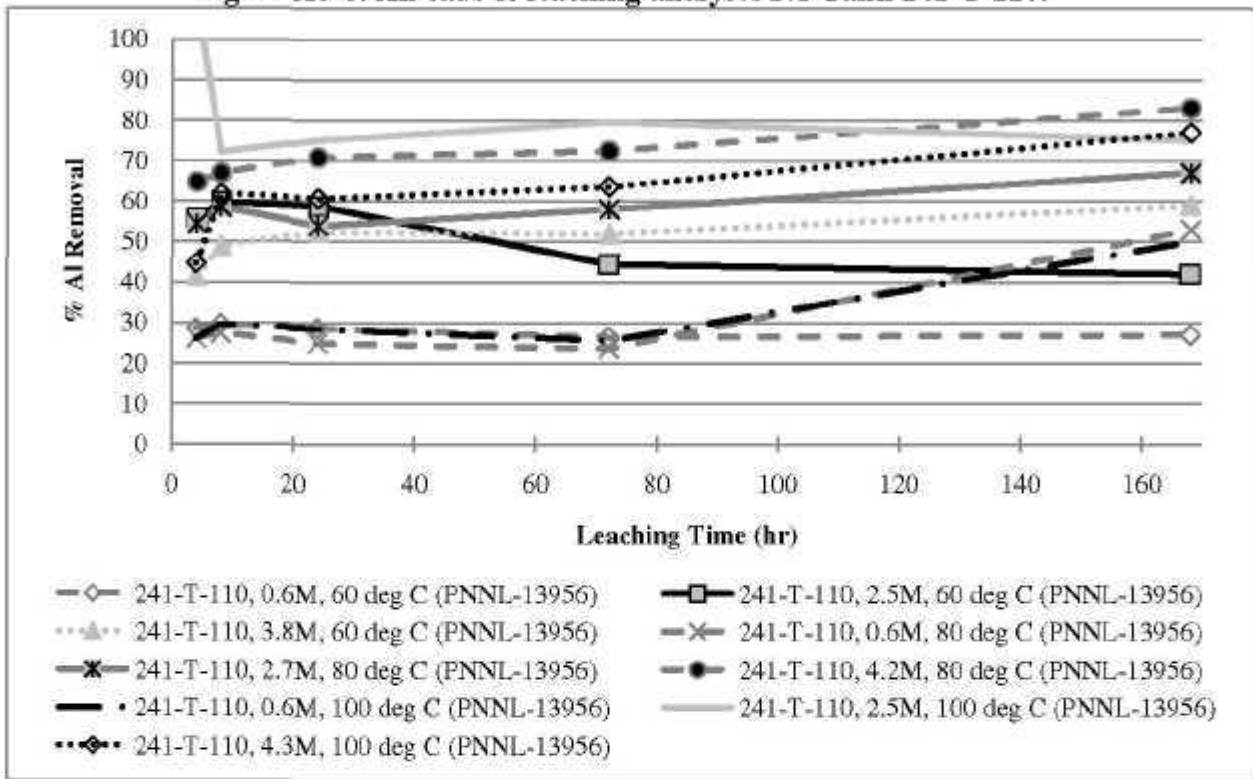


Figure A3-9. Caustic leaching using 1M NaOH at 60°C .

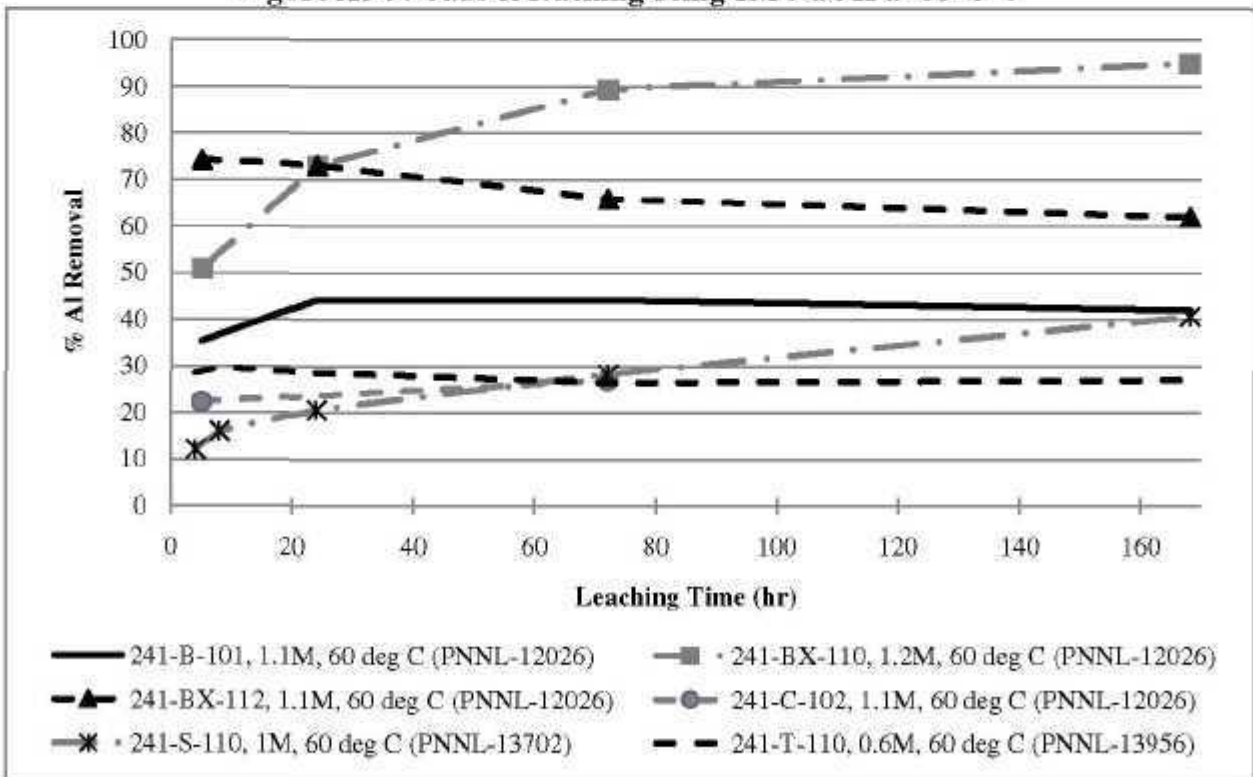




Figure A3-10. Caustic leaching using 3M NaOH at 60°C.

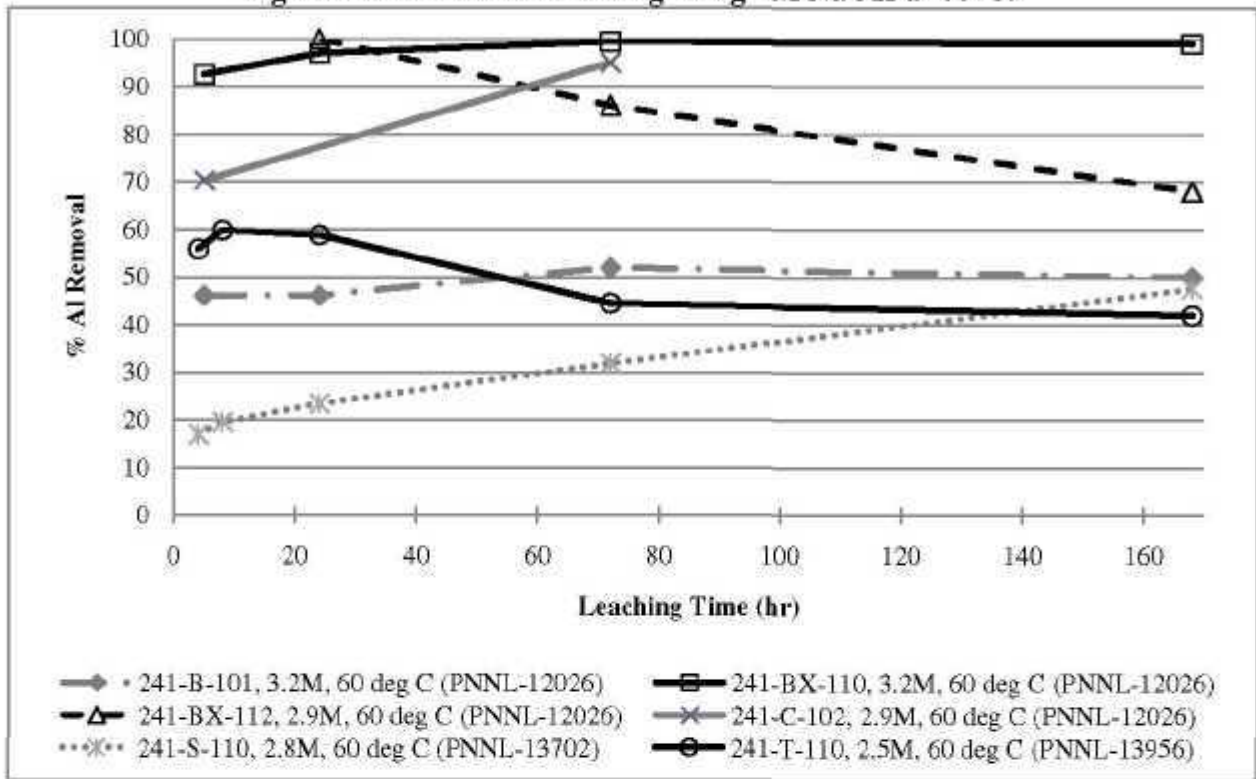


Figure A3-11. Caustic leaching using 1M NaOH at 80°C.

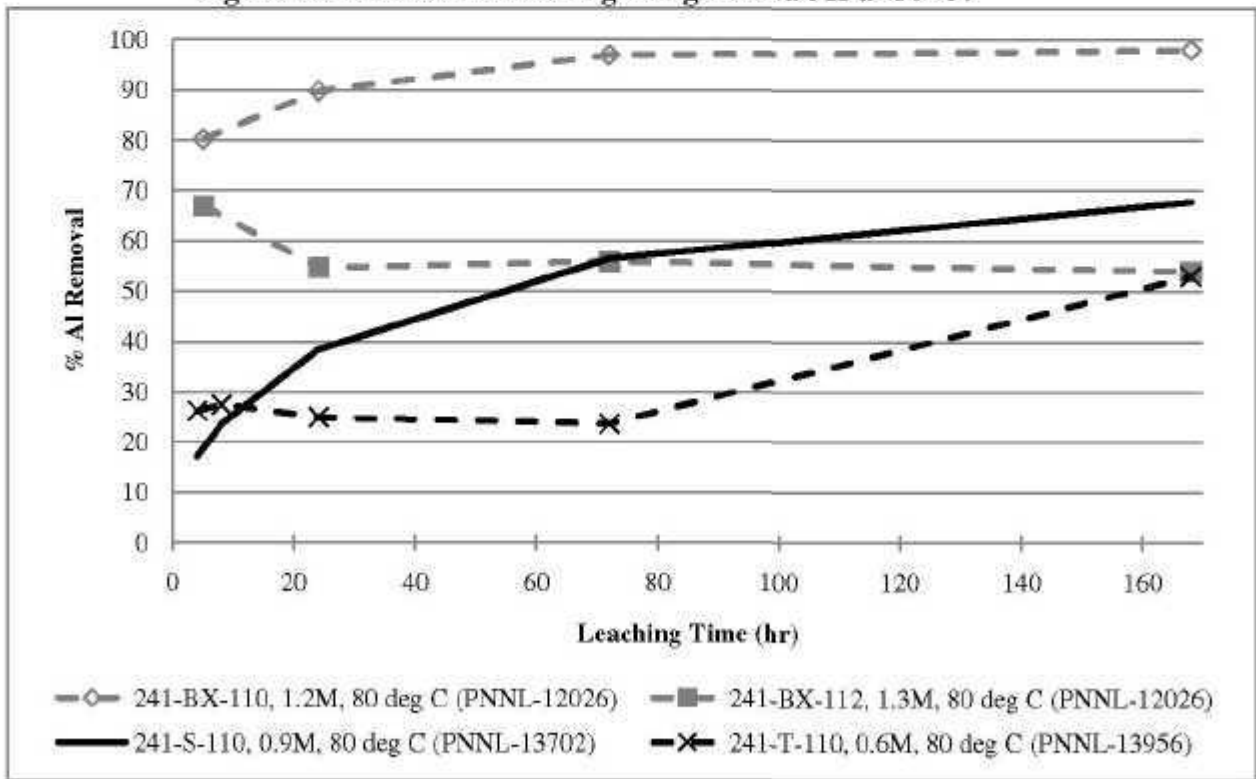


Figure A3-12. Caustic leaching using 3M NaOH at 80°C.

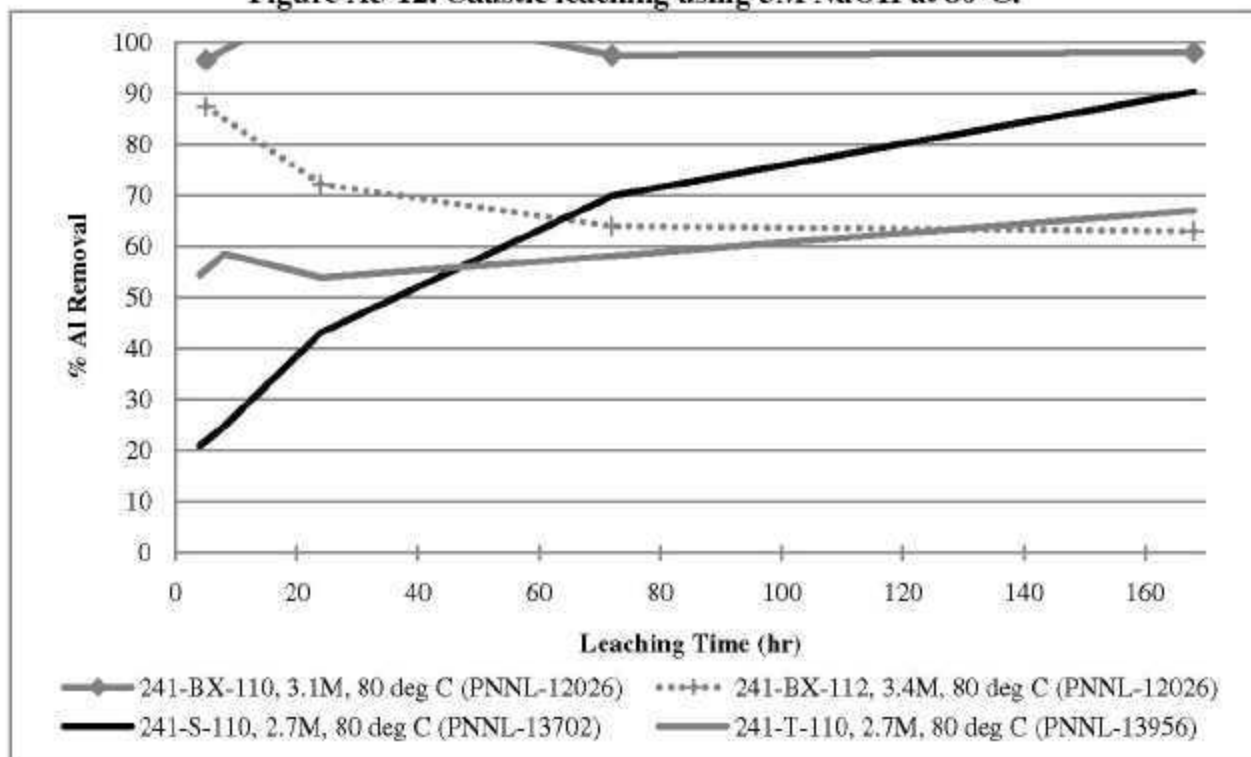


Figure A3-13. Caustic leaching using 1M NaOH at 100°C.

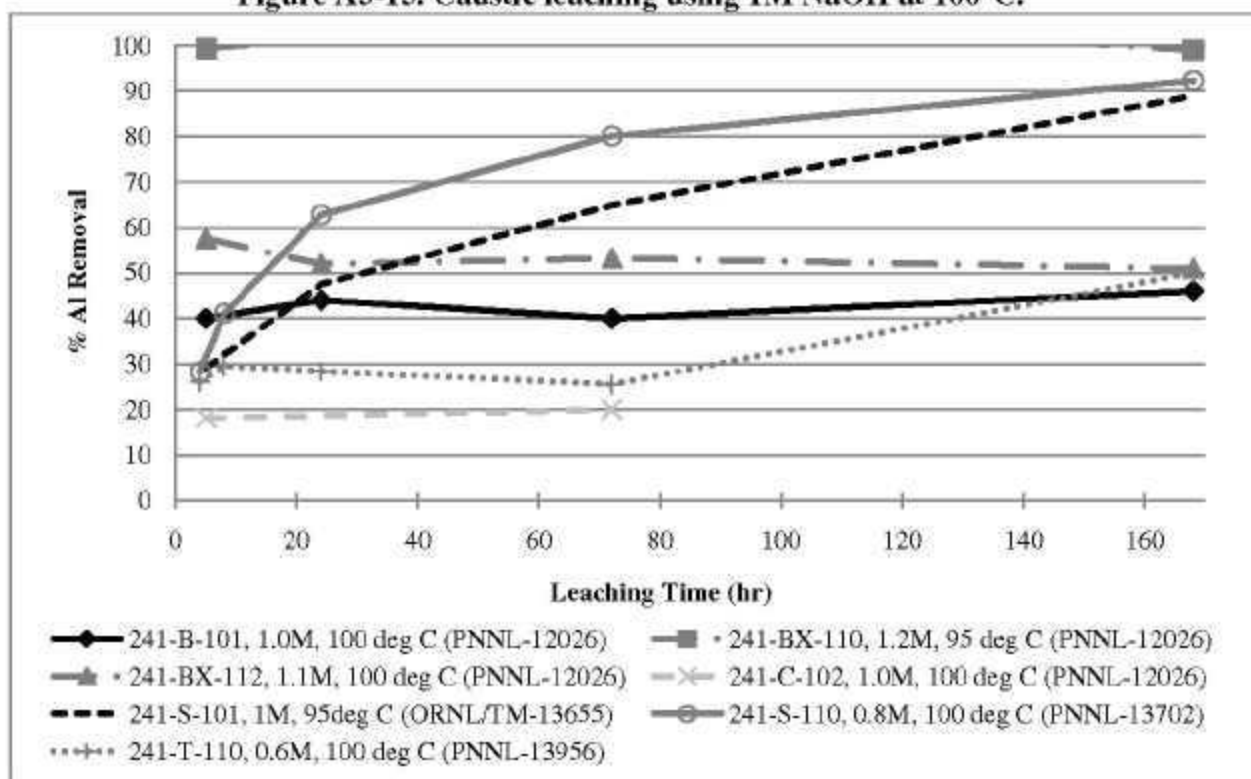
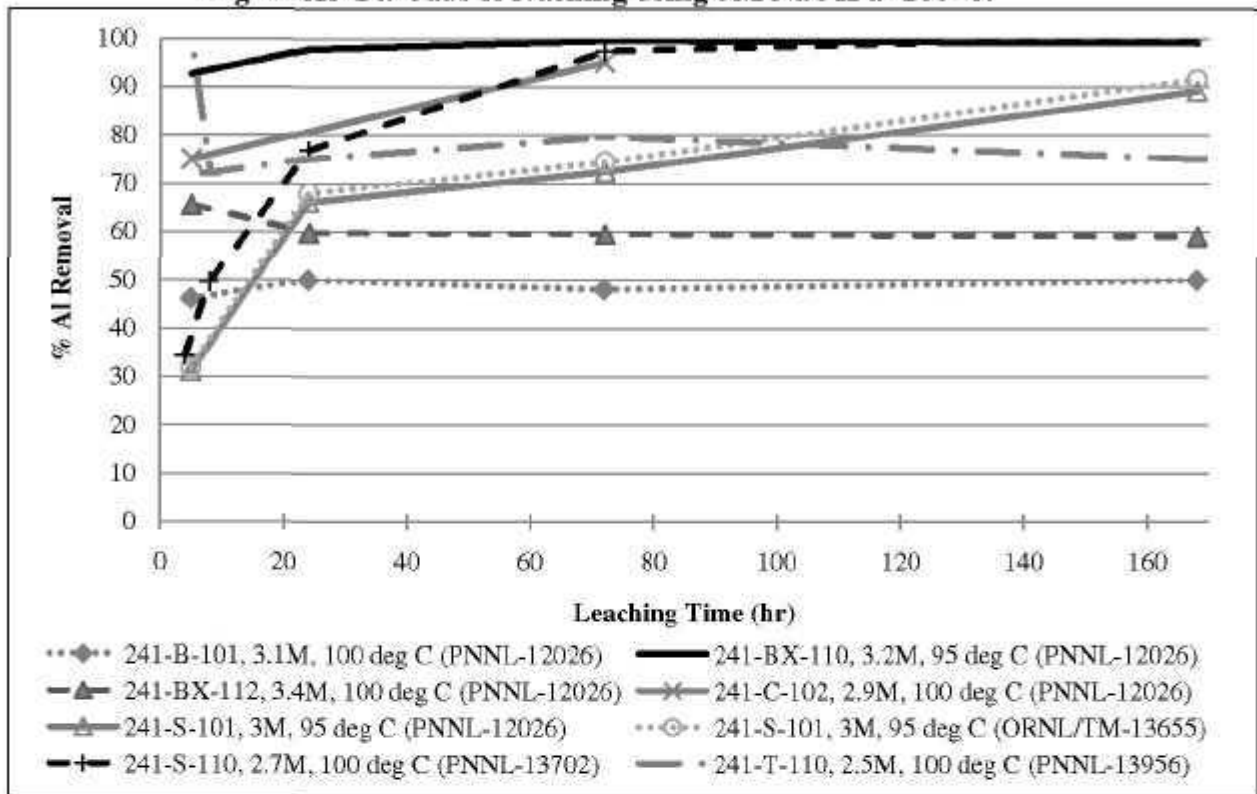


Figure A3-14. Caustic leaching using 3M NaOH at 100°C.





#### A4.0 APPENDIX REFERENCES

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