PNNL-13394

Status Report on Phase Identification in Hanford Tank Sludges

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Prepared for the U.S. Department of Energy
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Summary

The U.S. Department of Energy plans to vitrify Hanford's tank wastes. The vitrified wastes will be divided into low-activity and high-level fractions. There is an effort to reduce the quantity of high-activity wastes by removing nonradioactive components because of the high costs involved in treating high-level waste. Pretreatment options, such as caustic leaching, to selectively remove nonradioactive components are being investigated. The effectiveness of these proposed processes for removing nonradioactive components depends on the chemical phases in the tank sludges. This review summarizes the chemical phases identified to date in Hanford tank sludges.

Glossary

CC	complex concentrate
CW	cladding waste
DOE	U.S. Department of Energy
DIA	diatomaceous earth
DSSF	double-shell slurry feed
EB	evaporator bottoms
ED	electron diffraction
EDX	electron dispersive X-ray
ESPIP	Efficient Separations and Processing Integrated Program
ESW	enhanced sludge washing
F	ferrocyanide-scavenged waste
HLW	high-level waste
ICDD	International Centre for Diffraction Data
IHLW	immobilized high-level waste
ITS	in-tank solidification
LANL	Los Alamos National Laboratory
LAW	low-activity waste
NCAW	neutralized current acid waste
NCRW	neutralized cladding removal waste
OWW	organic solvent wash from the PUREX process
PNNL	Pacific Northwest National Laboratory
PUREX	plutonium-uranium extraction process
R	high-level REDOX waste
REDOX	reduction oxidation
SEM	scanning electron microscopy
SRS	strontium leached sludge

SR-WASH	particulates from Sr-wash of PUREX waste in the AR vault
TBP	tributyl phosphate waste
TEM	transmission electron microscopy
TFA	Tanks Focus Area
TWRS	Tank Waste Remediation System
USTID	Underground Storage Tank Integrated Demonstration
XRD	X-ray diffraction

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1.0 Introduction

Large volumes of high-level radioactive wastes (HLWs), generated during past Pu production and other operations, are stored in underground tanks at the U.S. Department of Energy's (DOE's) Hanford Site in Washington State. Because of the expected high cost of HLW immobilization and geologic disposal, pretreatment processes will be implemented to reduce the volume of immobilized high-level waste (IHLW). After partitioning of the tank wastes into high-level and low-activity fractions, the currently envisioned pretreatment strategy proposes to 1) remove radionuclides from the aqueous waste fractions to produce streams suitable for disposal as low-activity waste (LAW) and 2) use washing and selective leaching strategies to remove elements from sludges expected to drive HLW production, i.e., aluminum, phosphorus, and chromium. The LAW will be immobilized in a glass matrix and disposed of by shallow burial onsite. The HLW will be immobilized in a borosilicate glass matrix; the resulting glass canisters will then be disposed of in a geologic repository (Orme et al. 1996).

Dilute hydroxide washing is the minimum pretreatment for Hanford tank sludges. This method simply involves mixing the sludge with dilute (0.1 M or less) NaOH and then performing a solid/liquid separation. This is meant to remove water-soluble sludge components (mainly sodium salts) from the HLW stream. Dilute hydroxide is used rather than water to maintain the ionic strength high enough that colloidal suspensions are avoided.

The current baseline pretreatment for Hanford tank sludges involves an enhanced sludge washing (ESW) process. In this process, sludges will be leached with a more concentrated (typically 3 M) aqueous NaOH solution. The leached sludge will be subsequently washed with dilute NaOH to remove excess Na and any dissolved waste components. Leaching with NaOH is expected to remove a large fraction of the Al, which is present in large quantities in Hanford tank sludges. The Al will be removed by converting aluminum oxides/hydroxides to sodium aluminate (Equations 1 and 2). The chemistry involved in this process is well known, as it forms the basis for the Bayer process in the aluminum industry (Weifers and Misra 1987).

$$\mathrm{Al(OH)_3(s) + NaOH(aq) \rightarrow NaAl(OH)_4(aq)} \tag{1}$$

$$\mathrm{Al(O)(OH)(s) + H_2O + NaOH(aq) \rightarrow NaAl(OH)_4(aq)} \tag{2}$$

ESW is also expected to remove a significant portion of the P from the sludge by metathesis of water-insoluble metal phosphates to insoluble hydroxides and soluble Na_3PO_4. An example of this is shown for iron(III) phosphate in Equation 3.

$$\mathrm{FePO_4(s) + 3NaOH(aq) \rightarrow Fe(OH)_3(s) + Na_3PO_4(aq)} \tag{3}$$

Similar metathesis reactions can occur for insoluble sulfate salts, allowing the removal of sulfate from the HLW stream.

Based on its known amphoteric behavior (Rai et al. 1987), Cr(III) was expected to be removed by caustic leaching according to Equation 4:

$$\mathrm{Cr(OH)_3(s) + NaOH(aq) \rightarrow Na[Cr(OH)_4](aq)} \qquad (4)$$

However, we have observed that Cr in the caustic-leaching solutions is generally present as Cr(VI), suggesting that an oxidative pathway is involved (Lumetta et al. 1998).

The effectiveness of the proposed ESW process essentially depends on the chemical phases present in the tank sludges. For example, while aluminum in the form of hydroxide, oxides, or oxyhydroxides may be expected to dissolve to a significant extent, dissolution of aluminum in the form of aluminosilicates is problematic. Similarly, while phosphate in the form of iron or uranium phosphate should metathesize to metal hydroxides and soluble sodium phosphate according to Equation 3, metathesis of phosphate in the form of calcium phosphate is thermodynamically unfavorable. Because the specific chemical phases present can impact the effectiveness of pretreatment processes, efforts to identify the chemical phases present in a variety of Hanford tank sludges have been underway over the past several years. These efforts have combined powder X-ray diffraction (XRD), transmission electron microscopy/electron diffraction (TEM/ED), and scanning electron microscopy/electron dispersive X-ray (SEM/EDX) techniques to identify major phases present in Hanford tank sludges both before and after pretreatment processes. In this report, we summarize the results obtained through FY 1999. Aspects of this summary already have been reported (Lafemina 1995; Lumetta et al. 1998; Rapko et al. 1996).

2.0 Tank Sludges Examined

Numerous sludge-type wastes have been examined over the years for phase information. Table 2.1 summarizes the Hanford tanks examined, the assigned grouping of each tank based on the history of waste types added (the Sort on Radioactive Waste Type model for single-shell tanks [Hill et. al 1995]), the specific type(s) of waste believed to be contained in the tank, and the original document(s) describing the phase analyses. All analyses were conducted on actual tank sludge waste.(a) Samples typically were composites derived from core samples taken through risers in the top of the tanks. Specific experimental details used in preparing the caustic-leached samples are available in the cited references. Three types of analyses were performed to determine chemical phases. Powder XRD was used by Los Alamos National Laboratory (LANL) in all their studies and by Pacific Northwest National Laboratory (PNNL) in their initial studies. The other two analysis methods employed either TEM coupled with ED analysis or SEM coupled with EDX techniques.

At PNNL, the XRD analyses were performed using a Scintag Pad x-ray diffractometer. The 2θ range was 5 to 65 degrees with a counting time of 20 sec. X-ray tube conditions were 45 kilovolts and 40 milliamps. The copper $K\alpha$ line was used for the measurement, and the instrument was operated in the $\theta/2\theta$ mode. At LANL, a Rigaku x-ray diffractometer/monochromator was used. In both instances, a computerized search/match routine of the International Centre for Diffraction Data (ICDD) data set # 41 (PNNL) or # 43 (LANL) was used to identify crystalline phases. A chemical filter was used to select the elements present in potential matches; the selected elements were determined from the SEM/EDX analysis.

The TEM samples were prepared by dispersing a drop of the sample slurry on TEM copper grids covered with carbon films. This work was performed using a JEOL 1200 analytical TEM operating at 120 kV. The analyses involved 1) evaluating the morphology, distribution, and particle sizes by electron imaging, 2) determining the particle's chemical composition by electron dispersive X-ray analysis, 3) studying the particle's crystallinity by electron diffraction, and 4) identifying the particle's crystalline phase by comparison with the JCPS-EDD Data Base published by the ICDD.

At PNNL, a JEOL JSM840 scanning electron microscope was used to perform the SEM/EDX analyses. Samples were attached to SEM mounts using double-sided sticky tape and were carbon coated before SEM examination. At LANL, these measurements were made with a JEOL 6300F X-Vision scanning electron microscope with a PGT IMIX imaging/x-ray microanalysis system.

The following review is organized by phases that contain major nonradioactive sludge components critical to the ESW process, e.g., Al-, Cr-, and P-containing phases. Other identified phases also are discussed. In general, cases where microscopic examination revealed a particle's elemental composition, but where no phase information could be inferred, are not discussed. Some exceptions are made for

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(a) It should be noted that "as-received" samples typically would have contained interstitial liquid. Water-soluble components (e.g., $NaNO_3$, $NaNO_2$, etc.) observed in the "as-received" samples likely formed during evaporation of the sample.

simple systems where the phases present can be reasonably inferred, such as amorphous metal hydroxides or amorphous aluminosilicates.

Table 2.1. Summary of Hanford Tank Sludges Examined for Chemical Phase Information

		Waste Types(2)			
Tank	**SOWRT group(1)**	**Primary**	**Secondary**	**Testing Laboratory(3)**	**References**
S-101	1	R	EB	PNNL	Lumetta et al. 1997
S-107	1	R	EB	PNNL	Lumetta et al. 1996a
S-111	1	R	EB	PNNL	Lumetta et al. 1997
BY-104	3	TBP-F	EB-ITS	PNNL	Lumetta et al. 1996a
BY-108	3	TBP-F	EB-ITS	PNNL	Lumetta et al. 1997
BY-110	3	TBP-F	EB-ITS	PNNL	Lumetta et al. 1996a
S-104	4	R	-	PNNL(twice), LANL	Lumetta et al. 1997; Rapko et al. 1996; Temer and Villarreal 1995; Lafemina 1995
SX-108	4	R	-	PNNL	Lumetta et al. 1996a
BX-103	5	TBP	CW	LANL	Temer and Villarreal 1997
BX-105	5	TBP	CW	LANL	Temer and Villarreal 1995
BX-109	5	TBP	CW	LANL	Temer and Villarreal 1996
B-202	7	224	-	LANL	Temer and Villarreal 1995
C-107	10	1C	CW	LANL, PNNL	Temer and Villarreal 1996; Lumetta et al. 1996a
T-107	10	1C	CW	LANL	Temer and Villarreal 1995
U-110	10	1C	CW	PNNL	Jones et al. 1992
B-106	12	1C	TBP	LANL	Temer and Villarreal 1997

Table 2.1 (Continued)

		Waste Types(2)			
Tank	**SOWRT group(1)**	**Primary**	**Secondary**	**Testing Laboratory(3)**	**References**
BX-107	12	1C	TBP	PNNL	Rapko et al. 1996; Lafemina 1995
C-108	13	TBP-F	1C	LANL	Temer and Villarreal 1995
C-109	13	TBP-F	1C	PNNL	Colton et al. 1993
C-112	13	TBP-F	1C	PNNL	Colton et al. 1993; Lafemina 1995
B-110	16	2C	5 6	PNNL	Jones et al. 1992
B-111	16	2C	5 6	PNNL	Rapko et al. 1996; Lafemina 1995
C-106	20	SRS	SR-WASH	PNNL	Lumetta et al. 1996b
T-104	Ungrouped	1C	-	PNNL, LANL	Rapko et al. 1996; Lafemina 1995
T-111	15	2C	224	PNNL	Rapko et al. 1996; Lafemina 1995
B-104	Ungrouped	2C	EB	LANL	Temer and Villarreal 1996
TY-104	22	TBP	1C-F	LANL	Temer and Villarreal 1996
SX-113	24	R	DIA	LANL	Temer and Villarreal 1997
C-104	Ungrouped	CW	OWW	LANL	Temer and Villarreal 1997
C-105	Ungrouped	TBP	SR-WASH	LANL	Temer and Villarreal 1997
AN-104	double shell tank	DSSF	-	PNNL	Lumetta et al. 1997

Table 2.1 (Continued)

		Waste Types(2)			
Tank	**SOWRT group(1)**	**Primary**	**Secondary**	**Testing Laboratory(3)**	**References**
SY-103	double shell tank	CC	-	PNNL	Rapko et al. 1996; Lafemina 1995
AW-105	double shell tank	NCRW	-	PNNL	Lafemina 1995
SY-101	double shell tank	CC	-	PNNL	Lafemina 1995
AZ-101	double shell tank	NCAW	-	PNNL	Uziemblo(a)
AZ-102	double shell tank	NCAW	-	PNNL	Uziemblo(a)

1. Assignments of groups of single-shell tanks based on a statistical method (Hill et. al 1995).
2. For a fuller description of the waste types in single-shell tanks see Hill et. al (1995); for double-shell tanks see Hanlon (2000).
3. PNNL = Pacific Northwest National Laboratory; LANL = Los Alamos National Laboratory

224 = Lanthanum fluoride decontamination waste
1C = First cycle bismuth phosphate decontamination waste
2C = Second cycle bismuth phosphate decontamination waste
5-6 = High-level B Plant waste from the bottom of Section 5
CC = Complex concentrate
CW = Cladding waste
DIA = Diatomaceous earth
DSSF = Double-shell slurry feed
EB = Evaporator bottoms
ITS = In-tank solidification
F = Ferrocyanide-scavenged waste
NCAW = Neutralized Current Acid Waste
NCRW = Neutralized Cladding Removal Waste
OWW = Organic solvent wash from PUREX
R = High-level REDOX waste
SRS = Strontium leached sludge
SR-WASH = Particulates from Sr-wash of PUREX waste in the AR vault
TBP = Tributyl phosphate waste

(a) NH Unziemblo, B Mastel, and RR Adee. Unpublished results (1987).

3.0 Summary of Results

3.1 Aluminum-Containing Phases

Table 3.1 summarizes the Al-containing phases that have been identified in the Hanford tank sludges. The species reported were identified either in the as-received sample or in material that has been subjected to caustic leaching (generally at 80°C–100°C for several hours with a leachate nominally 3 M NaOH followed by dilute hydroxide washing to remove components in the interstitial liquid). Clearly, the sludges contain a variety of chemical species containing Al. For the sludges derived from the reduction oxidation (REDOX) process, boehmite is generally the dominant Al-containing species. Microscopic examinations also have revealed species such as $Al(OH)_3$ (amorphous or crystalline), $Al_2O_3 \bullet xH_2O$, $AlPO_4$, and both amorphous and crystalline aluminosilicates. It is unclear whether the frequency of aluminosilicates in the leached solids is due to their formation during the caustic-leaching process or to the effective removal of the other, more abundant, aluminum phases by the caustic-leaching process. The response of the phases identified in the as-received solids to caustic leaching has been summarized previously (Lumetta et al. 1998).

Not included in Table 3.1 is some early SEM/EDX work conducted on sludge from Hanford Tank SY-102 (Lumetta and Swanson 1993). This work is not included in the table because the solid phases present were not definitively identified. Nevertheless, in light of the work performed in more recent years, it is instructive to re-examine this earlier work. The water-washed SY-102 sludge consisted mostly of agglomerated particles. The EDS taken over a wide range of this material indicated the predominant elements present to be Al, Ca, Cr, Mn, and Fe. The presence of Th was also evident. In addition to the agglomerated particles, distinct cubic-shaped particles were evident. The latter particles remained in the solid residue following acid dissolution. The EDX of these particles showed them to be Al-rich. The morphology of these particles was very similar to the morphology of the boehmite particles identified in the S-101 and S-104 sludges (Lumetta et al. 1998); thus, they were likely boehmite.

Table 3.1. Aluminum-Containing Phases Identified in Hanford Tank Sludges

Tank	As-Received	Leached	Method
S-101	Al(O)(OH) (boehmite)	Al(O)(OH) (boehmite), aluminosilicates (am), Al(O)(OH) (diaspore)	TEM
S-107	Al(O)(OH) (boehmite), aluminosilicates (am)	Al(O)(OH) (boehmite), aluminosilicates (am)	TEM
S-111	NP	$Al(OH)_3$ (bayerite), Al(O)(OH) (boehmite)	TEM
BY-104	$(Al_2O_3)_x$-$(H_2O)_y$, aluminosilicates (am)	$(Al_2O_3)_x$-$(H_2O)_y$	TEM

Table 3.1 (continued)

Tank	As-Received	Leached	Method
BY-108	none	none	TEM
BY-110	NP	none	TEM
S-104	Al(O)(OH) (boehmite)	Al(O)(OH) (boehmite), aluminosilicate (am)	TEM/SEM/ XRD
SX-108	Al(O)(OH) (boehmite), aluminosilicate (a), $(Al_2O_3)_x$-$(H_2O)_y$	Al(O)(OH) (boehmite), aluminosilicate (am), $(Al_2O_3)_x$-$(H_2O)_y$, $Ca_3Al_2O_6$	TEM
BX-103	$Al(OH)_3$ (gibbsite)	none	XRD
BX-105	$Al(OH)_3$ (gibbsite)	$Al(OH)_3$ (gibbsite)	XRD
BX-109	$Al(OH)_3$ (nordstrandite)	none	XRD
B-202	none	NP	XRD
C-107	none, $(Al_2O_3)_x$-$(H_2O)_y$, aluminosilicates (am)	none, $(Al_2O_3)_x$-$(H_2O)_y$, aluminosilicates (am)	XRD, TEM
T-107	$Al(OH)_3$ (gibbsite)	aluminosilicates (c), $Al(PO_4)$	XRD
U-110	$Al(OH)_3$ (gibbsite), Al(O)(OH) (boehmite)	NP	SEM/XRD
B-106	none	none	XRD
BX-107	$AlPO_4$, $Al(OH)_3$ (am), aluminosilicates (c,am)	aluminosilicates (c,am)	TEM/SEM/ XRD
C-108	$Al(OH)_3$ (gibbsite)	none	XRD
C-109	$Al(OH)_3$ (gibbsite)	$Al(OH)_3$ (gibbsite), Al(O)(OH) (boehmite), Nickel Aluminum Oxide	SEM/XRD
C-112	$Al(OH)_3$ (gibbsite)	none	SEM/XRD
B-110	Sodium Aluminum Silicate Hydrate	NP	SEM/XRD
B-111	aluminosilicates (c)	aluminosilicates (c)	TEM/SEM/ XRD
C-106	$Al(OH)_3$ (am), aluminosilicates (am)	aluminosilicates (am)	TEM

Table 3.1 (continued)

Tank	As-Received	Leached	Method
T-104	$AlPO_4$, $Al(OH)_3$ (am), aluminosilicates (c,am), $Na_2Fe_2Al(PO_4)_3$	aluminosilicates (c,am), $Bi_{24}Al_2O_{39}$	TEM/SEM/ XRD
T-111	none	none	TEM/SEM/ XRD
B-104	none	none	XRD
TY-104	none	none	XRD
SX-113	none	none	XRD
C-104	none	none	XRD
C-105	$Al(OH)_3$ (gibbsite)	none	XRD
AN-104	aluminosilicate (am)	none	TEM
SY-103	$Al(OH)_3$ (am), $Al_2O_3(H_2O)_x$ (c)	$Al(OH)_3$ (am), $Al_2O_3(H_2O)_x$ (c)	TEM/SEM/ XRD
AW-105	$Al(OH)_3$, aluminosilicate (c)	aluminosilicate (c)	TEM/SEM/ XRD
SY-101	$NaAlO_2$, $Al(OH)_3$ (am)	none	TEM/SEM/ XRD
AZ-101	none	NP	SEM
AZ-102	Aluminosilicate (am)	NP	SEM

NP = measurement not performed, am = amorphous, c = crystalline

3.2 Chromium-Containing Phases

Table 3.2 summarizes the Cr-containing phases that have been identified in the Hanford tank sludges. Leaching conditions are the same as those described for Table 3.1. Few phases have been identified, especially in the untreated solids, which is presumably due to their generally low concentrations in the as-received sludges. In the high-Cr sludge SY-103, the chromium seems to be present as an amorphous hydroxide. In another case, the Cr was found to be present as a crystalline oxyhydroxide, gramaldite. But in most of these infrequent cases, the identified Cr phase has the chromium bound up with other transition metals in spinel-type structures or, in the high Bi-containing solids, in the compound $Bi_{38}CrO_{60}$. The response of the phases identified in the as-received solids to caustic leaching has been summarized previously (Lumetta et al. 1998).

3.3 Phosphorus-Containing Phases

Microscopy studies have revealed several P-containing species in the Hanford sludges (Table 3.3). For Table 3.3, the leaching conditions are the same as those described for Table 3.1. In general, these materials are present as simple metal phosphate salts. However, in T-111, phosphorus was determined to be present as the pyrophosphate in lanthanum pyrophosphate. The response of the phases identified in the as-received solids to caustic leaching has been summarized previously (Lumetta et al. 1998).

3.4 Other Phases

Microscopy studies have revealed several other species in the Hanford sludges (Table 3.4). Again, for Table 3.4, the leaching conditions are the same as those described for Table 3.1. Most of the materials identified in the as-received sludges are either simple salts (such as sodium nitrate), the metal oxides or oxyhydroxides of such common elements as iron, silicon, or bismuth (for the high Bi-containing tanks), and, in several cases, uranium oxide, either as the $U_2O_7^{2-}$ salt, or as an oxide compound such as UO_3, UO_2, or U_3O_7. The leached solids differ primarily by the disappearance of the simple sodium salts such as nitrate and nitrite and, often, the identification of other metal oxide phases. It is likely that these new phases were present even in the initial solids, but that the other, more abundant, material obscured their presence.

Table 3.2. Chromium-Containing Phases Identified in Hanford Tank Sludges

Tank	As-Received	Leached	Method
S-101	none	none	TEM
S-107	none	none	TEM
S-111	NP	$FeCr_2O_4$ Mn_2CrO_4 $Mn_{1.5}Cr_{1.5}O_4$	TEM
BY-104	$Fe(Fe,Cr)_2O_4$ (donathite)	none	TEM
BY-108	none	none	TEM
BY-110	NP	Cr(O)(OH) (grimaldite)	TEM
S-104	none	none	TEM/SEM/XRD
SX-108	none	none	TEM
BX-103	none	none	XRD
BX-105	none	none	XRD
BX-109	none	none	XRD
B-202	none	NP	XRD
C-107	none	none	XRD, TEM
T-107	none	none	XRD
U-110	none	NP	SEM/XRD
B-106	none	none	XRD
BX-107	none	none	TEM/SEM/XRD
C-108	none	none	XRD
C-109	none	none	SEM/XRD
C-112	none	none	SEM/XRD
B-110	none	NP	SEM/XRD
B-111	$Bi_{38}CrO_{60}$	$Bi_{38}CrO_{60}$	TEM/SEM/XRD
C-106	none	none	TEM
T-104	none	$Bi_{38}CrO_{60}$ chromium phosphate	TEM/SEM/XRD

Table 3.2 (Continued)

Tank	As-Received	Leached	Method
T-111	none	none	TEM/SEM/XRD
B-104	none	none	XRD
TY-104	none	none	XRD
SX-113	none	none	XRD
C-104	none	none	XRD
C-105	none (XRD)	none	XRD
AN-104	none	none	TEM
SY-103	$Cr(OH)_3$ (am)	$Cr(OH)_3$ (am)	TEM/SEM/XRD
AW-105	none	none	TEM/SEM/XRD
SY-101	none	none	TEM/SEM/XRD
AZ-101	none	NP	SEM
AZ-102	none	NP	SEM

NP = measurement not performed, am = amorphous, c = crystalline

Table 3.3. Phosphorus-Containing Phases Identified in Hanford Tank Sludges

Tank	As-Received	Leached	Method
S-101	none	none	TEM
S-107	none	none	TEM
S-111	NP	none	TEM
BY-104	none	$Ca_5(OH)(PO_4)_3$	TEM
BY-108	$Ca_xSr_{10-x}(PO_4)_6(OH)_2$	$Ca_xSr_{10-x}(PO_4)_6(OH)_2$	TEM
BY-110	NP	$Ca_5(OH)(PO_4)_3$	TEM
S-104	none	none	TEM/SEM/XRD
SX-108	none	none	TEM
BX-103	none	none	XRD
BX-105	none	none	XRD
BX-109	none	none	XRD
B-202	none	NP	XRD
C-107	none (XRD) $Pb_5(OH)(PO_4)_3$	none (XRD) none	XRD, TEM
T-107	none	$Al(PO_4)$	XRD
U-110	none	NP	SEM/XRD
B-106	none	none	XRD
BX-107	$AlPO_4$, $Bi/FePO_4$	$Bi/FePO_4$	TEM/SEM/XRD
C-108	$Ca_3(PO_4)_2$	$Ca_5(OH)(PO_4)_3$ $Ca_5F(PO_4)_3$	XRD
C-109	none	none	SEM/XRD
C-112	none	none	SEM/XRD
B-110	$BiPO_4$	NP	SEM/XRD
B-111	Na_3PO_4	Na_3PO_4	TEM/SEM/XRD
C-106	none	none	TEM
T-104	$AlPO_4$ $Na_2Fe_2Al(PO_4)_3$ (XRD)	chromium phosphate, uranyl phosphate hydrate	TEM/SEM/XRD

Table 3.3 (continued)

Tank	As-Received	Leached	Method
T-111	Na_3PO_4 $La_4(P_2O_7)_3$ $Ca_5(OH)(PO_4)_3$ Bi, Fe phosphate	$Ca_5(OH)(PO_4)_3$ $La_4(P_2O_7)_3$ Bi, Fe phosphate	TEM/SEM/XRD
B-104	none	none	XRD
TY-104	none	none	XRD
SX-113	none	none	XRD
C-104	none	none	XRD
C-105	none	none	XRD
AN-104	none	none	TEM
SY-103	none	none	TEM/SEM/XRD
AW-105	none	none	TEM/SEM/XRD
SY-101	none	none	TEM/SEM/XRD
AZ-101	none	NP	SEM
AZ-102	none	NP	SEM

NP = measurement not performed, am = amorphous, c = crystalline

Table 3.4. Other Phases Identified in Hanford Tank Sludges

Tank	As-Received	Leached	Method
S-101	Mn/Fe(O)(OH)	Mn/Fe(O)(OH)	TEM
S-107	ZrO_2 Fe(O)(OH) UO_3	ZrO_2 Fe(O)(OH) UO_3	TEM
S-111	NP	UO_3	TEM
BY-104	none	β -U_3O_8 $Ni_3O_2(OH)_4$ Fe(O)(OH)	TEM
BY-108	β-U_3O_8 γ-Fe_2O_3 (maghemite) Fe(O)(OH)	β-U_3O_8 γ -Fe_2O_3 (maghemite) Fe(O)(OH)	TEM
BY-110	NP	β-U_3O_8 $Ni_3O_2(OH)_4$ γ -Fe_2O_3 (maghemite)	TEM
S-104	β-U_3O_8 $NaNO_3$ (natratine)	UO_3-$2H_2O$ $FeMnO_4$	TEM/SEM/XRD
SX-108	β-U_3O_8, Fe(O)(OH)	β-U_3O_8 Fe(O)(OH) $(Mn,Fe)_3O_4$	TEM
BX-103	none	$Na_2U_2O_7$	XRD
BX-105	none	none	XRD
BX-109	$NaNO_3$ (natratine)	$Na_2U_2O_7$	XRD
B-202	$NaNO_3$ (natratine)	NP	XRD
C-107	Fe_3O_4 (hematite) (XRD) Fe(O)(OH) ZrO_2	Fe_3O_4 (hematite) (XRD) Fe(O)(OH)	XRD, TEM
T-107	$NaNO_3$ (natratine)	none	XRD
U-110	$NaNO_3$	NP	SEM/XRD
B-106	none	none	XRD
BX-107	$Fe_2Bi(SiO_4)_2(OH)$ Bi_2O_3	$Fe_2Bi(SiO_4)_2(OH)$ Bi_2O_3	TEM/SEM/XRD

Table 3.4 (continued)

Tank	As-Received	Leached	Method
C-108	$NaNO_3$ (natratine)	none	XRD
C-109	$NaNO_3$ $NaNO_2$ SiO_2	$Ni(OH)_2$ UO_3	SEM/XRD
C-112	$NaNO_3$ $NaNO_2$ SiO_2 CaU_2O_7	$Na_2U_2O_7$	SEM/XRD
B-110	$NaNO_3$	NP	SEM/XRD
B-111	$Fe(OH)_3$ (am) Bi_2O_3 $Fe_2Bi(SiO_4)_2(OH)$	$Fe(OH)_3$ (am) Bi_2O_3 $Fe_2Bi(SiO_4)_2(OH)$	TEM/SEM/XRD
C-106	none	$Fe(O)(OH)$ Ag_2O ZrO_2	TEM
T-104	$Fe_2Bi(SiO_4)_2(OH)$ Bi_2O_3	$Fe_2Bi(SiO_4)_2(OH)$ Bi_2O_3 Bismuth Iron Oxide	TEM/SEM/XRD
T-111	$Fe(OH)_3$ (am) Mn_2MnO_4 Fe_2MnO_4 (jacobsite) $Fe(O)(OH)$, (goethite)	$Fe(OH)_3$ (am) Mn_2MnO_4 Fe_2MnO_4 (jacobsite) $Fe(O)(OH)$, (goethite)	TEM/SEM/XRD
B-104	$Na_3(NO_3)(SO_4)(H_2O)$ (darapskite)	$KMg_3(Si_3AlO)$-$10H_2O$ (phlogopite)	XRD
TY-104	$NaNO_3$ (natratine)	none	XRD
SX-113	none	none	XRD
C-104	none	none	XRD
C-105	$UO_3(H_2O)$	$Na_2U_2O_7$	XRD
AN-104	UO_2 or U_3O_7	UO_2 or U_3O_7	TEM
SY-103	Fe, Mn oxide	Fe, Mn oxide	TEM/SEM/XRD

Table 3.4 (continued)

Tank	As-Received	Leached	Method
AW-105	none	none	TEM/SEM/XRD
SY-101	none	none	TEM/SEM/XRD
AZ-101	$NaNO_3$, $NaNO_2$, $Na_2CO_3(H_2O)$	NP	SEM
AZ-102	$NaNO_3$, $NaNO_2$, $Na_2U_2O_7$	NP	SEM

NP = measurement not performed, am = amorphous, c = crystalline

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