Hanford Waste Mineralogy Reference Report

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U.S. Department of Energy Contract DE-AC27-08RV14800

EDT/ECN: DRF

UC: None

Cost Center: 2GB00

Charge Code:

B&R Code: None

Total Pages: 85

Key Words: Mineralogy, crystal phase, Hanford waste, tank summary, data review

Abstract: This report lists the observed mineral phases present in the Hanford tanks. This task was accomplished by performing a review of numerous reports that used experimental techniques including, but not limited to: x-ray diffraction, polarized light microscopy, scanning electron microscopy, transmission electron microscopy, energy dispersive spectroscopy, electron energy loss spectroscopy, and particle size distribution analyses. This report contains tables that can be used as a quick reference to identify the crystal phases observed in Hanford waste.

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Nancy A Fouad
Release Approval

6-29-10
Date

Release Stamp

Approved For Public Release

Tank Operations Contractor (TOC) RECORD OF REVISION		(1) Document Number: RPP-RPT-46618		Page 1
(2) Title: Hanford Waste Mineralogy Reference Report				
Change Control Record				
(3) Revision	(4) Description of Change – Replace, Add, and Delete Pages	Authorized for Release		
		(5) Resp. Engr. (print/sign/date)	(6) Resp. Mgr. (print/sign/date)	
1 RS	Additional reports added in report and spelling errors corrected in DRF and Title Page forms DRF form – replace Title page – replace Table 1-1. (pg.25) table entry added Table 1-1. (pgs.36,37,38) table entries added REFERENCES (pg.79) – references added	R.S. Disselkamp 6/28/10	J.G. Reynolds 06/28/10	

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Date Published

June 2010

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

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Richland, Washington

Contractor for the U.S. Department of Energy
Office of River Protection under Contract DE-AC27-08RV14800

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LIST OF TERMS

Abbreviations and Acronyms

BBI	Best-Basis Inventory
EDS	energy dispersive spectroscopy
EELS	electron energy loss spectroscopy
PSD	particle size distribution
PLM	polarized light microscopy
PNNL	Pacific Northwest National Laboratory
PUREX	plutonium-uranium extraction
REDOX	reduction-oxidation
SEM	scanning electron microscopy
TEM	transmission electron microscopy
TWINS	tank waste inventory system
WTP	Waste Treatment Plant
XRD	x-ray diffraction

Units

Vol.%	volume percent
Wt.%	weight percent (solid)

1.0 Identification of Hanford Crystalline Phases

Successful operation of the Hanford cleanup operation involves, in part, an identification of the chemical speciation of solids in the Hanford waste. This is because both retrieval activities that depend on mixing and pumping processes, and waste treatment plant (WTP) leaching processes depend upon chemical speciation. Successful WTP leaching depends upon aluminum oxyhydroxide leaching by caustic at elevated temperature, and chromium oxyhydroxide leaching by the oxidizing agent permanganate at near ambient temperature, and requires solid phase identification for successful modeling of the processes. Leaching behavior is directly related to mineralogy type. Successful completion of these tasks includes compiling a readily searchable resource for solid waste. Key information includes mineralogy content for the Hanford tank farms, including detailed crystal and amorphous phases of all solid compounds present in the waste. This report summarizes all currently known mineralogical phases for solids contained within the Hanford tanks. This summary is presented as a “quick reference” source and additional information can be obtained from the referenced published reports. Mineralogical data herein is sorted by Hanford tank type. As new information becomes available, or as existing information is brought to our attention, this document will be revised.

Readers of this document are requested to contact the authors with suggestions of reports/data to be included in the future.

This report is organized into four sections. Each section presents different aspects of mineralogical phase identification in the Hanford tanks. The four sections include the following information:

- Section 1.0 Table 1-1. Crystal Phase Identification in Hanford Tanks.
- Section 2.0 Table 2-1. WTP-PNNL Group 1-8 Waste Characterization Results.
- Section 3.0 Table 3-1. Characterization of Mineral Identification in Boildown Laboratory Studies.
- Section 4.0 Table 4-1. Elemental Identification of Tank Solid Samples using SEM-EDS.

This report is a compilation of mineralogical data for the Hanford tank farm. Each table is sorted by tank and lists the following:

- mineral name;
- chemical formula or elements in solid sample;
- tank from which the analyzed sample was taken;
- experimental techniques used to analyze sample;
- waste type with unique pre-analysis processing; and

- comments that include, at a minimum, the published report from which the data was taken.

This comprehensive list of mineralogical data can be used in efforts to:

- a) examine chromium and aluminum leaching behavior;
- b) correlate particle size distribution and density with mineralogy type;
- c) examine solid-liquid equilibrium using reported solid characterization with supernate composition; and
- d) correlate waste slurry data (e.g., rheology) to supernate and particle type.

Table 1-1 below presents the information above.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium carbonate hydrate (thermonatrite)	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AN-102	A2-saltcake waste type or caustic treated supernate.	From FH-0303343, and 7S110-WSC-05-011. Core 307, Segments 18, 19, 20, 21A, 21B. Identified using PLM and SEM-EDS. A major component (>10 wt.%). Particulates in interstitial liquid. Origin of carbonate likely from breakdown of organics and sequestration of carbon dioxide from atmosphere. See also: 7S110-DLH-05-028. Identified crystalline solids were characterized by XRD and SEM-EDS. This was a major component. Obtained from caustic addition to supernate. This observation suggests that the added caustic (i.e., sodium) may initiate precipitation of Na_2CO_3.
Sodium fluorophosphate hydrate (natrophosphate)	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AN-102	Supernate liquid used; liquid taken from vicinity of A2 saltcake waste.	From 7S110-DLH-05-028. Identified crystalline solids were characterized by XRD and SEM-EDS. This was a minor component. Obtained from caustic addition to supernate. This observation suggests that the added caustic (i.e., sodium) may initiate precipitation of sodium-containing $\text{Na}_7\text{F}(\text{PO}_4)_2$.
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AN-102	A2-saltcake waste type.	From FH-0303343, and 7S110-WSC-05-011. Core 307, Segments 18, 19, 20, 21A, 21B. Study undertaken as part of corrosion mitigation work by caustic demand test. Tank contains PUREX waste, 52 kgal 50 wt.% caustic added in 2001. Identified using PLM and SEM-EDS. A major component (>10 wt.%). This observation suggests that the added caustic (i.e., sodium) may initiate precipitation of sodium-containing sodium oxalate particulates within interstitial liquid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium aluminum carbonate hydroxide (dawsonite)	$\text{Na}_2\text{Al}(\text{CO}_3)(\text{OH})_2$	AN-102	A2-saltcake waste type.	From 7S110-WSC-05-011. Core 307, Segments 18, 19, 20, 21A, 21B. Identified using PLM and SEM-EDS. A major component (>10 wt.%). 52 kgal 50 wt.% caustic added in 2001. The high caustic addition is significant as it can cause aid precipitation of hydroxide solids. This observation suggests that the added caustic (i.e., sodium) may initiate dissolution of sodium-containing sodium-aluminum-carbonate.
Sodium phosphate hydrate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	AN-102	A2-saltcake waste type.	From 7S110-WSC-05-011. Core 307, Segments 18, 19, 20, 21A, 21B. Study undertaken as part of corrosion mitigation work by caustic demand test. Tank contains PUREX waste, 52 kgal 50 wt.% caustic added in 2001. Identified using PLM and SEM-EDS. A minor component (1-10 wt.%). This observation suggests that the added caustic (i.e., sodium) initiates precipitation of sodium-containing tri-phosphate.
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	AN-102	A2-saltcake waste type.	From FH-0303343, and 7S110-WSC-05-011. Core 307, Segments 18, 19, 20, 21A, 21B. Study undertaken as part of corrosion mitigation work by caustic demand test. Tank contains PUREX waste, 52 kgal 50 wt.% caustic added in 2001. Identified using PLM and SEM-EDS. A minor component (1-10 wt.%). This observation suggests that the added caustic (i.e., sodium) initiates precipitation of sodium-containing diuranate.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminum oxide hydroxide (boehmite)	AlOOH	AN-102	A2-saltcake and/or supernatant liquid.	From FH-0303343. Identified using XRD and SEM-EDS. A minor component. Caustic demand test on composite sludge and supernatant samples run from equal volume prepared material. Study undertaken as part of corrosion mitigation work by caustic demand test. Tank contains PUREX waste, 52 kgal 50 wt.% caustic added in 2001. BBI identification of solids were as saltcake, however report gave solids used as sludge. After caustic demand, followed by water-washing, boehmite was observed. See also: PNNL report, PNWD-3300. Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. XRD analysis identified this crystalline phase as existing, but not necessarily abundant. Here water-washed A2-waste type saltcake solids studied and boehmite identified.
Uranium oxide	UO ₃	AN-102	Water-washed sludge [likely complex concentrate waste type from B-Plant].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake. XRD analysis identified uranium oxide crystalline phase. Not necessarily in high abundance. Common component in PUREX waste.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Iron oxide (hematite)	Fe ₂ O ₃	AN-102	Water-washed sludge [likely complex concentrate waste type from PUREX].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. Although it seems odd to have iron in a saltcake, the complexants in the waste could have had an impact on presence of iron in waste. XRD analysis identified hematite crystalline phase. Not necessarily in high abundance.
Calcium carbonate (calcite)	CaCO ₃	AN-102	Water-washed sludge [likely complex concentrate waste type from PUREX].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. A combination of SEM, EDS, XRD, and light scattering analysis techniques used to identify calcite crystalline phase. Not in high abundance.
Cerianite	CeO ₂	AN-102	Water-washed sludge [likely complex concentrate waste type from PUREX].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. A combination of SEM, EDS, XRD, and light scattering techniques used to identify cerianite crystalline phase. Not in high abundance.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate (nitratine)	NaNO_3	AN-102	Water-washed sludge [likely complex concentrate waste type from PUREX].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. A combination of SEM, EDS, XRD, and light scattering analysis techniques used to identify nitratine crystalline phase. In high abundance.
Sodium nitrite	NaNO_2	AN-102	Water-washed sludge [likely complex concentrate waste type from PUREX].	From PNNL report: PNWD-3300 Commissioning and operation of WTP served as motivation for study to characterize composition, morphology, and PSD of Hanford tank solids. BBI inventory identified only A2 saltcake solids present, no sludge. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. A combination of SEM, EDS, XRD, and light scattering analysis techniques used to identify sodium nitrite crystalline phase. In high abundance.
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AN-106	NA sludge waste type.	From Herting, 2004 (7S110-DLH-04-013). Identified by Optical Microscopy. Sludge produced from a neutralizing oxalic acid solution used to dissolve a Heel in Tank C-106. The resulting added caustic (i.e., sodium) likely precipitated (additional) sodium oxalate used in corrosion mitigation. From Warrant, 2010 (WRPS-1000562). C-solids included as part of waste type. Identified by PLM, SEM, PSD, and EDS analyses. Study conducted as part of caustic demand testing. Solids precipitated during caustic addition.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminum hydroxide (gibbsite)	$\text{Al}(\text{OH})_3$	AN-106	NA sludge waste type.	From Herting, 2004 (7S110-DLH-04-013). Identified by Optical Microscopy. Sludge produced from a neutralizing oxalic acid solution used to dissolve a Heel in Tank C-106. Aluminum originated from cladding in PUREX process waste. It is likely that this process reduced gibbsite already present.
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AN-106	C sludge waste type.	From Warrant, 2010 (WRPS-1000739). C-solids included as part of waste type. Identified by PLM, SEM, PSD, and EDS analyses. Study conducted as part of caustic demand testing. Solids precipitated during caustic addition.
Sodium fluorophosphate hydrate (natrophosphate)	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19 \text{H}_2\text{O}$	AN-106	C sludge waste type.	From Warrant, 2010 (WRPS-1000739, WRPS-1000562). C-solids included as part of waste type. Identified by PLM, SEM, PSD, and EDS analyses. Study conducted as part of caustic demand testing. Solids precipitated during caustic addition.
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AN-107	A2-Saltcake solids analysis.	From RPP-20018. Core 304 using segments 18R, 19R, 20, 21A and 21B employed. Work performed to support corrosion mitigation by examining caustic demand. Identification techniques include PLM, SEM-EDS, and XRD. Sodium oxalate was the most abundant crystalline phase identified. An initial examination of tank composition by examining waste template [RPP-8847; LA-UR-96-3860] seems to indicate only trace amounts of oxalate. However, the waste transfer records showed [TWINS; May 2010] indicated 465 kgal waste received from AN-102, a tank with high oxalate concentration. Performed by caustic and supernate addition to saltcake sample for corrosion mitigation.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Dawsonite	$\text{NaAlCO}_3(\text{OH})_2$	AN-107	A2-Saltcake solids analysis.	From RPP-20018. Core 304 using segments 18R, 19R, 20, 21A and 21B employed. Work performed to support corrosion mitigation by examining caustic demand. Identification techniques include PLM, SEM-EDS, and XRD. Dawsonite was the second most abundant crystalline phase identified. PUREX cladding source of aluminum. Very high carbonate concentration of 0.066 g- CO_3 /g, observed from waste inventory [BBI database]. Solids formed from caustic and supernate addition to saltcake sample.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AN-107	A2-Saltcake solids analysis.	From RPP-20018. Core 304 using segments 18R, 19R, 20, 21A and 21B employed. Work performed to support corrosion mitigation by examining caustic demand. Identification techniques include PLM, SEM-EDS, and XRD. Sodium fluoride phosphate was the fourth most abundant crystalline phase identified. Waste template inventory of phosphate is 0.0047 g- PO_4 /g [BBI database]. Performed by caustic and supernate addition to saltcake sample for corrosion mitigation.
Sodium carbonate hydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AN-107	A2-Saltcake solids analysis.	From RPP-20018. Core 304 using segments 18R, 19R, 20, 21A and 21B employed. Work performed to support corrosion mitigation by examining caustic demand. Identification techniques include PLM, SEM-EDS, and XRD. Sodium carbonate hydrate was the fifth most abundant crystalline phase identified. Very high carbonate concentration of 0.066 g- CO_3 /g, observed from waste inventory [BBI database]. Performed by caustic and supernate addition to saltcake sample.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate (nitratine)	NaNO_3	AN-107	A2-Saltcake solids analysis.	From RPP-20018. Core 304 using segments 18R, 19R, 20, 21A and 21B employed. Work performed to support corrosion mitigation by examining caustic demand. Identification techniques include PLM, SEM-EDS, and XRD. Waste template and TWINS also showed high nitrate concentration (ca. 4.0 M). Sodium nitrate was the sixth most abundant crystalline phase identified. Performed by caustic and supernate addition to saltcake sample.
Sodium carbonate hydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AN-107	Supernate (that in contact with A2-saltcake solids).	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. Very high carbonate concentration of 0.066 g- CO_3 /g, observed from waste inventory [BBI database]. Precipitated supernate performed by a caustic demand test. It is likely that the added caustic (i.e., sodium) precipitated initially soluble Na_2CO_3 .
Trona	$\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$	AN-107	Carbonation test of AN-107 supernatant liquid, resulting in solids precipitation.	From 7S110-DLH-06-049. Purpose of study was to examine atmospheric acidification, via carbon dioxide adsorption, into Hanford waste. Identification techniques include PLM, SEM-EDS, and XRD. XRD conclusively identified trona crystalline phase. Very high carbonate concentration of 0.066 g- CO_3 /g, observed from waste inventory [BBI database]. Experiment performed by bubbling CO_2 gas through supernate and examining solids formed. The added carbon dioxide acidified the supernatant and precipitated the observed carbonate-containing product. The CO_2 was not added to the tank. Study part of corrosion mitigation effort.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Thermonatrite, Sodium Carbonate monohydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AP-108	NA saltcake waste type.	From 74A10-GAC-08-164. Techniques used include PLM, SEM, and XRD. Core 330 segments 22, 23, 24A, and 24B were used. Sampling and solid phase identification supported both corrosion mitigation and criticality safety programs. Both centrifuged solids and settled solids were used. Grab sample analysis. This was the major component found in the waste. A transfer in of 1118 kgal of AP-103 waste yielded an increase of carbonate since this material contained 0.090 g- CO_3 /g carbonate [BBI database]. Grab sample analysis. Sample was not treated prior to analysis (e.g., direct analysis).
Sodium Fluoride Phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AP-108	NA saltcake waste type.	From 74A10-GAC-08-164. Techniques used include PLM, SEM, and XRD. Core 330 segments 22, 23, 24A, and 24B were used. Both centrifuged solids and settled solids were used. Grab sample analysis. Minor components of both phosphate and fluoride (<0.01M) [BBI database] were in the waste. Added caustic likely precipitated the crystalline solid material. This was a minor component found in the waste. Grab sample analysis. Sample was not treated prior to analysis (e.g., direct analysis).
Kogarkoite, sodium fluoride Sulfate	Na_3FSO_4	AP-108	NA saltcake waste type.	From 74A10-GAC-08-164. Techniques used include PLM, SEM, and XRD. Core 330 segments 22, 23, 24A, and 24B were used. Both centrifuged solids and settled solids were used. Grab sample analysis. Somewhat high sulfate, 0.012 g- SO_4 /g observed [BBI database], was contained in the waste per inventory report. This was a minor component found in the waste. Grab sample analysis. Sample was not treated prior to analysis (e.g., direct analysis).

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate (nitratine)	NaNO_3	AW-101	A2 saltcake.	From HNF-13805. This report is a presentation to the American Chemical Society, so details are omitted. There have been transfers out of AW-101 (into AW-102 and AY-102), so perhaps this analysis supports waste compatibility issues. Both simulant made sodium nitrate solid particulates, and those obtained from tank AW-101 direct sampling, have been compared. PLM analysis only. No additional corroboration, say from SEM-EDS or XRD was given.
Gibbsite	$\text{Al}(\text{OH})_3$	AW-105	Sludge solids. NCRW PUREX (aluminum cladding) waste type.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the caustic leaching (aka: enhanced sludge washing (ESW)) and leaching of Hanford material, to support waste treatment. Identified by TEM, SEM-EDS and XRD. Material was examined after direct sampling.
Sodium nitrate (nitratine)	NaNO_3	AW-106	A2 waste type saltcake.	From 7S110-GAC-08-150. Core 323, segment 10, solids. The techniques of PLM, SEM-EDS and XRD were used to characterize crystal type and abundance. Composition amount ca. 30 wt.%. This phase could have formed from precipitation of supernatant liquid, but analyses were performed on the settled solids collected.
Sodium carbonate monohydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AW-106	A2 waste type saltcake.	From 7S110-GAC-08-150. Core 323, segment 10, solids. The techniques of PLM, SEM-EDS and XRD were used to characterize crystal type and abundance. Composition amount ca. 10 wt.%. This phase could have formed from precipitation of supernatant liquid, but analyses were performed on the settled solids collected. The high concentration of carbonate of 0.045 g- CO_3 /g [BBI database] is the cause of precipitation of this solid crystal material. Purpose of study unclear from reference above (or referenced documents therein). From TWINS waste transfer documentation it is either in support of waste compatibility (for transfer out) or an evaporator campaign performed.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Lithium aluminum hydroxide hydrate	$\text{LiAl}_2(\text{OH})_7 \cdot 2\text{H}_2\text{O}$	AW-106	A2 waste type saltcake.	From 7S110-GAC-08-150. Core 323, segment 10, solids. This solid is most likely due to contamination with 0.3M LiBr hydrostatic fluid, into the collected sample, used at the beginning of core sampling. The aqueous lithium likely reacted with soluble aluminum and hydroxide in the waste to form the observed solid. For LiBr use use/sampling see LMHC-74B20-99-014 memo. The techniques of PLM, SEM-EDS and XRD were used to characterize crystal type and abundance.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AX-101	A composite sample from the tank was cascade dissolved in water and a liquid plus solids fraction obtained. The liquid fraction was usually evaporated to yield precipitated solids. In this particular experiment, the supernatant liquid was cooled from 50 to 20 °C, yielding solids. No mention of existence of solid phase in tank. A1 saltcake	From HNF-11585. The experimental techniques of polarized light microscopy (PLM) and XRD were used to identify this phase in the dissolved saltcake sample. PLM showed isotropic crystals. Both phosphate and fluoride concentrations of waste [BBI database] were very high at 0.004 g/g and 0.0008 g/g, respectively.
Sodium phosphate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	AX-101	A composite sample from the tank was cascade dissolved in water and a liquid plus solids fraction obtained. The liquid fraction was usually evaporated to yield precipitated solids. In this particular experiment, the supernatant liquid was cooled from 50 to 20°C, yielding solids. No mention of existence of solid phase in tank. A1 saltcake	From HNF-11585. The reference used polarized light microscopy (PLM) and XRD to identify this phase in the dissolved saltcake sample. PLM showed isotropic crystals. The phosphate concentration in waste of 0.004 g/g [BBI database] was a very high concentration for precipitation of solids.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Dawsonite	$\text{NaAlCO}_3(\text{OH})_2$	AY-101	NA-sludge studied, mixed with a 50/50 vol.% mixture of NA-sludge/supernate.	From 7S110-RWW-08-147. This was a caustic demand test. Crystalline phases were identified by PLM and XRD analyses. This phase was most abundant in this report. Caustic demand of sludge samples, in support of corrosion mitigation. The added caustic (i.e., Na) with initially high carbonate present of 0.098 g/g [BBI database] yielded solids precipitation.
Dawsonite	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	AY-101	NA-sludge studied, mixed with a 50/50 vol.% mixture of NA-sludge/supernate.	From 7S110-RWW-08-147. This was a caustic demand test. Crystalline phases were identified by PLM, XRD, and SEM-EDS analyses. This phase was second most abundant in this report. Caustic demand of sludge samples, in support of corrosion mitigation. The added caustic (i.e., Na) with initially high carbonate present of 0.098 M [BBI database] yielded solids precipitation.
Iron oxide (Hematite)	Fe_2O_3	AY-101	NA-sludge studied, mixed with a 50/50 vol.% mixture of NA-sludge/supernate.	From 7S110-RWW-08-147. This was a caustic demand test. Crystalline phases were identified by PLM, XRD and SEM-EDS analyses. This phase was the third most abundant solid phase in this report. Caustic demand of sludge samples, in support of corrosion mitigation. According to BBI and transfer records of TWINS, the total iron concentration is appreciable at 0.066 g/g sludge [BBI database]. Therefore this level of iron present, or tank corrosion release of iron, is responsible for hematite formation. In fact, caustic added multiple times to mitigate corrosion issues.
Thermonatrite	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	AY-102	Although the solids are classified as "sludge" [see Herting, FH-0303406], they could contain both solid waste types of BL-solid and unspecified sludge.	From 7S110-DLH-03-007. Sludge sample contained coring from segments 11, 12, 13, and 14A. PLM, SEM-EDS, and XRD were utilized. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g [BBI database] caused precipitation of crystal phase. This observed crystal was a primary component. Sludge plus supernatant mixture.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
			Waste type is either BL- solid and/or unspecified sludge. Centrifuged solids analysis.	From 7S110-DLH-05-040. Obtained from Core 318, segment 8. In this study only PLM was used to determine crystal habit size, shape, and color. This provided good evidence for solids identification. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g [BBI database] caused precipitation of crystal phase.
			Centrifuged NA composite sludge solids.	From 7S110-RWW-06-080. Core 319 material used. XRD and SEM-EDS was used to identify crystal phase. This was the third most abundant component in this study. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g [BBI database] caused precipitation of crystal phase.
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AY-102	Waste type is either BL- solid and/or unspecified sludge. Centrifuged solids analysis.	From 7S110-DLH-05-040. Obtained from Core 318, segment 15. In this study only PLM was used to determine crystal habit size, shape, and color. This provided good evidence for solids identification. The original waste [RPP-8847] and subsequent transfers into tank AY-102 [RPP-8847] all contained less than 0.0013 g/g of oxalate [BBI database]. The high added sodium during caustic demand testing must have precipitated crystal phase.
Dawsonite	$\text{NaAlCO}_3(\text{OH})_2$	AY-102	Although the solids are classified as "sludge" [see Herting, FH-0303406], they could contain both solid waste types of BL- solid and unspecified sludge.	From 7S110-DLH-03-007. Sludge sample contained coring from segments 11, 12, 13, and 14A. PLM, SEM-EDS, and XRD were utilized. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g and high aluminum of 0.064 g/g [BBI database] caused precipitation of crystal phase. This observed crystal was a primary component. Sludge plus supernatant mixture.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
			Centrifuged NA composite sludge solids.	From 7S110-RWW-06-080. Sample taken from Core 319. XRD and SEM-EDS was used to identify crystal phase. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g and high aluminum of 0.064 g/g [BBI database] caused precipitation of crystal phase. This was the fifth most abundant component in this study.
			Leached (and water washed) NA-sludge solids.	From PNNL-14614. Cores 270 (segment 10) and 281 (segment 11) were used. XRD and SEM-EDS techniques used to identify this crystal phase as most abundant. Caustic demand test adding sodium plus high carbonate in waste of 0.066 g/g and high aluminum of 0.064 g/g [BBI database] caused precipitation of crystal phase. Inability to leach suggests kinetic reason to persist as a solid. Approximately 26 wt.% abundant. Dawsonite remained after sludge water washing.
Gibbsite	$\text{Al}(\text{OH})_3$	AY-102	Centrifuged NA composite sludge solids.	From 7S110-RWW-06-080. Core 319 material used. XRD and SEM-EDS was used to identify crystal phase. Caustic demand testing with high hydroxide plus initially high aluminum in waste of 0.064 g/g [BBI database] suggests that precipitation of gibbsite via super-saturation occurred readily. This was the fourth most abundant component in this study.
			Leached (and water washed) NA-sludge solids.	From PNNL-14614. Cores 270 (segment 10) and 281 (segment 11) were used. XRD and SEM-EDS techniques used to identify this crystal phase as third most abundant. Residual caustic in waste and high aluminum formed gibbsite. The dissolution of gibbsite requires moderate temperature so solids persisted in these tests. Approximately 17 wt.% abundant. Un-leached and water leached NA-sludge solids examined. Gibbsite remained after water leaching.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
			Composite NA-waste type sludge solids. Direct sampling.	From HNF-6047 Rev.0A. Cores 270 and 273; whole core composites. Identified using XRD, and acid digestion/chromatography. Residual caustic in waste and high aluminum formed gibbsite. The dissolution of gibbsite requires moderate temperature so solids persisted in these tests. A major component is solids, approximately 30 wt.%.
Iron oxide (Hematite)	Fe ₂ O ₃	AY-102	Although the solids are classified as “sludge” [see Herting, FH-0303406], they could contain both solid waste types of BL-solid and unspecified sludge.	From 7S110-DLH-03-007. Sludge sample contained coring from segments 11, 12, 13, and 14A. PLM, SEM-EDS, and XRD were utilized. Caustic demand test. Iron is of unknown origin as waste plus transfers into AY-102 contained only small iron concentrations. Perhaps corrosion-induced iron formation, as would be consistent with caustic additions to tank. This observed crystal was a primary component. Sludge plus supernatant mixture.
			Centrifuged NA composite sludge solids.	From 7S110-RWW-06-080. Core 319 material used. XRD and SEM-EDS was used to identify crystal phase. Iron is of unknown origin as waste plus transfers into AY-102 contained only small iron concentrations. Perhaps corrosion-induced iron formation, as would be consistent with caustic additions to tank. This was the most abundant component in this study. Caustic demand testing. Also confirmed in: From PNNL-14614. Cores 270 (segment 10) and 281 (segment 11) were used. XRD and SEM-EDS techniques used to identify this crystal phase as second most abundant. Approximately 19 wt.% abundant. Un-leached and water leached NA-sludge solids examined. Hematite remained after water leaching.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Cancrinite	$\text{Na}_6\text{Ca}_{1.5}\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)_{1.6}$	AY-102	Although the solids are classified as “sludge” [see Herting, FH-0303406], they could contain both solid waste types of BL-solid and unspecified sludge.	From 7S110-DLH-03-007. Sludge sample contained coring from segments 11, 12, 13, and 14A. PLM, SEM-EDS, and XRD were utilized. Caustic demand test precipitated out zeolite-like compound due to added sodium or corrosion of glass. This observed crystal was a primary component. Sludge plus supernatant mixture.
		AY-102	Centrifuged NA-sludge solids.	From 7S110-RWW-06-080. Core 319 material used. XRD and SEM-EDS was used to identify crystal phase. Caustic demand test precipitated out zeolite-like compound due to added sodium or corrosion of glass. This was the second most abundant component in this study.
		AY-102	Composite NA-waste type sludge solids. Direct sampling.	From HNF-6047 Rev.0A. Cores 270 and 273; whole core composites. Identified using XRD, and acid digestion/chromatography. A minor, but nonetheless significant component in solids: approximately 6 wt.% Silicon in crystal compound.
Manganese dioxide	MnO_2	AY-102	Composite NA-waste type sludge solids.	From HNF-6047 Rev.0A. Cores 270 and 273; whole core composites. Identified using XRD, and acid digestion/chromatography. Crystal formation consistent with minor, but sufficient, concentration of $1.7\text{E-}5$ M in waste [RPP-8847; LA-UR-97-3860]. A minor component is solids, approximately 5-6 wt.%.
Sodium nitrate (nitratine)	NaNO_3	AY-102	Although the solids are classified as “sludge” [see Herting, FH-0303406], they could contain both solid waste types of BL-solid and unspecified sludge.	From 7S110-DLH-03-007. Sludge sample contained coring from segments 11, 12, 13, and 14A. PLM, SEM-EDS, and XRD were utilized. Caustic demand test plus originally high nitrate in waste of 0.47 M [RPP-8847; LA-UR-97-3860] led to precipitation of solids. This observed crystal was a primary component. Sludge plus supernatant mixture.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Boehmite	Al(O)OH	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	From PNNL report: PNWD-3300. BBI inventory identified P3AZ1 and/or NA sludge as present. XRD analysis identified boehmite crystalline phase as prevalent, but not necessarily in high abundance. Broader XRD pattern than AN-102, suggesting less crystalline. High aluminum in waste type of 0.12 g/g consistent with boehmite formation [BBI database].
Iron oxide (Hematite)	Fe_2O_3	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	From PNNL report: PNWD-3300. XRD analysis identified hematite crystalline phase as prevalent. The iron content in the waste type of 0.059 g/g [BBI database] consistent with hematite formation.
Gibbsite	Al(OH)_3	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	From PNNL report: PNWD-3300. XRD analysis identified hematite crystalline phase as prevalent. High aluminum in waste type of 0.12 g/g consistent with gibbsite formation [BBI database].
Chromite	FeCr_2O_4	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	PNWD-3300. BBI inventory identified primarily A2 sludge solids present. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. SEM, EDS, XRD, and light scattering analysis identified chromite crystalline phase. Not in high abundance.
Sodium nitrate (nitratine)	NaNO_3	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	PNWD-3300. BBI inventory identified primarily A2 sludge solids present. Hence solids must be A2 saltcake, with the iron originating from PUREX waste stream. SEM, EDS, XRD, and light scattering analysis identified nitratine crystalline phase. In high abundance. Could have formed from inadvertent drying of sample.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrite	NaNO_2	AZ-101	Water-washed P3AZ1 and/or NA sludge solids.	PNWD-3300. BBI inventory identified primarily A2 sludge solids present. The iron originating from PUREX waste stream. SEM, EDS, XRD, and light scattering analysis identified sodium nitrite crystalline phase. In high abundance. Could have formed from inadvertent drying of sample.
Sodium fluorosulfate	Na_3FSO_4	AZ-102	Composite sample from multiple cores (261 & 262) containing either P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From PNNL WTP report: PNWD-3235 (WTP-RPT-054). XRD analysis used to identify solid phases and compositions. From BBI database both the fluoride and sulfate concentrations are very high in AZ-101 at ca. 0.0008 g/g and 0.012 g/g, respectively. Filtered supernatant was evaporated to 50% of initial volume at 50 C, then cooled for solids precipitation, concentrating components even further prior to precipitation. Most abundant crystal phase seen (57 wt.%).
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	AZ-102	Composite sample from multiple cores (261 & 262) containing either P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From PNNL WTP report: PNWD-3235 (WTP-RPT-054). XRD analysis used to identify solid phases and compositions. The oxalate concentration, from TWINS, is ca. 3.8 g/L in the supernate. Filtered supernatant was evaporated to 50% of initial volume at 50 C, then cooled for solids precipitation, concentrating components even further prior to precipitation. Second most abundant crystal phase seen (28 wt.%).
Sodium nitrite	NaNO_2	AZ-102	Composite sample from multiple cores (261 & 262) containing P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From PNNL WTP report: PNWD-3235 (WTP-RPT-054). XRD analysis used to identify solid phases and compositions. The nominal nitrite concentration of 0.20 M is not a sufficient condition for solids formation. Solids must have formed from dried sample. Filtered supernatant was evaporated to 50% of initial volume at 50 C, then cooled for solids precipitation, concentrating components even further prior to precipitation. Third most abundant crystal phase seen (7 wt.%).

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Goethite	FeO(OH)	AZ-102	P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From 7S110-WSC-03-012. From Core 310, segments 18 and 19. Identified from refractive index and PSD information. TWINS indicates a solid iron amount of 55 g/kg solids basis, supporting identification of this phase. Only a tentative assignment can be made from data. Estimated solids 32 mole %.
Iron oxide (Hematite)	Fe_2O_3	AZ-102	P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From 7S110-WSC-03-012. From Core 310, segments 18 and 19. Identified from refractive index and PSD information. TWINS indicates a solid iron amount of 65 g/kg solids basis, supporting identification of this phase. Only a tentative assignment can be made from data. Estimated solids 16 mole %.
Gibbsite	Al(OH)_3	AZ-102	P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From 7S110-WSC-03-012. From Core 310, segments 18 and 19. Identified from refractive index and PSD information. From BBI database list aluminum at 0.053 g/g concentration, supportive of boehmite formation in this PUREX refluxing tank. Only a tentative assignment can be made from data. Estimated solids 16 mole %.
Aluminum oxide (Boehmite)	AlO(OH)	AZ-102	P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass.	From 7S110-WSC-03-012. From Core 310, segments 18 and 19. Identified from refractive index and PSD information. From BBI database list aluminum at 0.053 g/g concentration, supportive of boehmite formation in this PUREX refluxing tank. Only a tentative assignment can be made from data. Estimated solids 16 mole %.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Dawsonite	$\text{NaAl}(\text{CO}_3)(\text{OH})_2$	AZ-102	P3AZ2, PL2, SRR, and/or NA sludge solids studied. P3AZ2 and SRR waste types comprise 71% and 25%, respectively, of solids by mass. [However, cited report states washed saltcake solids. From BBI, no saltcake solids exist.] Settled solids suspended in a simulant representative of supernate. PSD distribution information with refractive index data used to infer particle type.	From 7S110-WSC-03-012. From Core 310, segments 18 and 19. Identified from refractive index and PSD information. With substantial aluminum, carbonate, and hydroxide, this crystal phase at a low concentration is consistent with tank composition and history. Only a tentative assignment can be made from data. Estimated solids 3 mole %.
Gibbsite	$\text{Al}(\text{OH})_3$	BX-101	Leached (and water washed) NA-sludge solids.	From PNNL-14614. Cores 270 (segment 10) and 281 (segment 11) were used. XRD and SEM-EDS used to identify this major abundant crystal phase. Purpose of testing was to characterize material and assess water leachability of sludge. PUREX waste following strontium recovery containing significant aluminum (ca. 1.2 M) [RPP-8847, LA-UR-97-3860]. Without caustic and mild temperature gibbsite would not be expected to dissolve. Gibbsite remained after washing of sludge.
Cancrinite	$\text{Na}_6\text{Ca}_{1.5}\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)_{1.6}$	BX-101	Leached (and water washed) NA-sludge solids.	From PNNL-14614. Cores 270 (segment 10) and 281 (segment 11) were used. XRD and SEM-EDS used to identify this major abundant crystal phase. Purpose of testing was to characterize material and assess water leachability of sludge. PUREX waste following strontium recovery containing significant aluminum (ca. 1.2 M), calcium (0.007 M), and silicon (0.026 M) [RPP-8847; LA-UR-97-3860]. Cancrinite remained after water washing.
Gibbsite	$\text{Al}(\text{OH})_3$	BX-103	Sludge solids. Waste type CWP2.	From PNNL-13394. Identified only through XRD, so amount not quantified.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite	$\text{Al}(\text{OH})_3$	BX-105	Sludge solids. Waste types: BY (39 wt.% of solids) and CWP2 (39 wt.% of solids).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through XRD, so amount not quantified. Was observed in both 3M caustic leached and direct sampled material. This CW waste contains sufficient aluminum for gibbsite to exist and since the direct sampled material was not treated with caustic at moderate temperature, it persisted in sample (i.e., did not dissolve).
Gibbsite	$\text{Al}(\text{OH})_3$	BX-107	Sludge solids. Waste type: 1C.	From PNNL-13394. Identified by TEM, SEM-EDS, and XRD. Was observed in direct sampled material. Aluminum content of bismuth-phosphate waste of 1C solids is 0.015 g-Al/g-solids [BBI database].
Aluminum phosphate	AlPO_4	BX-107	Sludge solids. Waste type: 1C.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM, SEM-EDS, and XRD. Was observed in direct sampled material. Aluminum and phosphate content of bismuth-phosphate waste of 1C solids is 0.015 g-Al/g-solids and 0.069 g- PO_4 /g-solids [BBI database].
Aluminosilicate	Al-Si-O solids	BX-107	Sludge solids. Waste type: 1C.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM, SEM-EDS, and XRD. Was observed in both caustic leached (3M) and direct sampled materials. The XRD analysis was not analyzed to yield an exact composition (i.e., Al:Si ratio). Aluminum and silicon content of bismuth-phosphate waste of 1C solids is 0.015 g-Al/g-solids and 0.006 g-Si/g-solids [BBI database].

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Nordstrandite	$\text{Al}(\text{OH})_3$	BX-109	Sludge solids. Waste type: TBP.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through XRD, so amount not quantified. Was observed in both caustic leached (3M) and direct sampled materials. Was observed in both 3M caustic leached and direct sampled material. This CW waste contains sufficient aluminum for nordstrandite to exist and since the direct sampled material was not treated with caustic at moderate temperature, it persisted in sample (i.e., did not dissolve).
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	BY-102	Found in dissolved BY-waste type saltcake material. Composite core sample, see Herting, HNF-8849 Rev.0. The sample from the tank was dissolved (using sequential multi-step cascade dissolution arrangement) in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids. Hence solids dissolution and re-precipitation were studied.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used polarized light microscopy (PLM) and XRD to identify this phase in the dissolved saltcake sample. PLM showed isotropic crystals. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid. Also confirmed in: From HNF-5193. Waste type is BY saltcake. Identified with PLM, XRD, and SEM. Composite sample obtained from cores 1-7. Found in grab saltcake solids sampling. Dissolution tests performed with water. Residual solids, following dissolution, were analyzed and identified listed solid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride sulfate	Na_3FSO_4	BY-102	BY-saltcake.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Direct sampling of solids. The reference used XRD and PLM to identify solids in dissolved saltcake sample. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid.
				From HNF-7031 Rev.0. Major phase identified from PLM and SEM/EDS. Dissolution tests performed to simulate sluicing using inhibited water (0.01M NaOH).
Cancranite	$\text{Na}_6\text{Ca}_{1.5}\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)_{1.6}$	BY-102	BY-saltcake.	From HNF-5193 Rev.0. Major phase identified from PLM and SEM/EDS. Dissolution tests performed using inhibited water to dissolve material using simulated sluicing technique.
Gibbsite	$\text{Al}(\text{OH})_3$	BY-102	BY-saltcake.	From HNF-5193 Rev.0. Major phase identified from PLM and SEM/EDS. Dissolution tests performed using inhibited water to dissolve material using simulated sluicing technique.
Donathite	$\text{Fe}(\text{Fe,Cr})_2\text{O}_4$	BY-104	Sludge solids. PFeCN.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Tentatively identified by TEM. Seen in direct sampled material. The TBP waste was treated with potassium ferrocyanide as a cesium scavenger, explaining the origin of this iron solid material.
Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	BY-106	BY saltcake	From HNF-7031 Rev.0. Major phase identified from PLM and SEM/EDS. Dissolution tests performed to simulate sluicing using inhibited water (0.01M NaOH).

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	BY-109	BY-saltcake waste type. The sample from the tank was cascade-chain dissolution in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The sample was analyzed with polarized light microscopy (PLM) and XRD to identify this phase in the saltcake sample (e.g., BY9Evap2, BY9Evap4, BY9Evap5, BY9Evap6, and BY9Dil1). Identification based on comparison between standard and waste samples. Found in saltcake-evaporated precipitated liquid. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	BY-109	BY-saltcake waste type. The sample from the tank was cascade dissolved in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids.	From HNF-8849. A mixture of cores 201 and 203. PLM used to identify crystal phase. Standard sample analysis of solids collected was employed. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride sulfate	Na ₃ FSO ₄	BY-109	BY-saltcake waste type [see HNF-8849].	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used polarized light microscopy (PLM) and SEM-EDS to identify this phase in the precipitated saltcake liquid sample. PLM showed isotropic crystals. Identification based on comparison between standard and a 50-50 (v/v) waste mix of BY-109 and U-107 samples. Also seen in precipitated BY-109 supernatant. Unclear if forming 50-50 mixture unambiguously confirms phase presence, as well as precipitated BY-109 supernatant. The sample from the tank was cascade-chain dissolution in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids. Found in dissolved saltcake liquid, as studied here. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid. Also confirmed in: HNF-11744-VA. Techniques of PLM, SEM-EDS, and XRD used to identify crystal phase. Identified in water-washed saltcake solids. BY-waste type. From HNF-11744-VA. Techniques of PLM, SEM-EDS, and XRD used to identify crystal phase. Identified in water-washed saltcake solids. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and phosphate of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride	NaF	BY-109	BY-saltcake waste type [see HNF-8849 Rev.0].	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Usually forms double salts instead of NaF. However, was confirmed to exist in BY-109 in one sample collected, as determined by SEM and XRD. The sample from the tank was cascade-chain dissolution in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids. Precipitated supernatant from dissolved saltcake studied here. This was an In-Tank Solidification (ITS) tank that contained significant amount of fluoride of 0.022 g/g [BBI database], consistent with the presence of this solid formed from evaporated liquid. Also in HNF-11744-VA. Techniques of PLM, SEM-EDS, and XRD used to identify crystal phase. Identified in water-washed saltcake solids. This was an In-Tank Solidification (ITS) tank that contained significant amount of fluoride of 0.022 g/g [BBI database], consistent with the presence of this solid formed from evaporated liquid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	BY-109	BY-saltcake waste type.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. This crystal is difficult to conclusively distinguish from sodium carbonate using PLM and SEM-EDS. However, it has a definitive XRD pattern that can be used to confirm its presence. The sample from the tank was cascade dissolved in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids. Re-evaporated saltcake. Confirmed by PLM, SEM-EDS, and XRD. According to BBI Inventory, the oxalate solids concentration is 0.0042 mg/g solids basis. Also confirmed in: From HNF-11744-VA. Techniques of PLM, SEM-EDS, and XRD used to identify crystal phase. Identified in water-washed saltcake solids. Also confirmed in: From 7S110-DLH-06-073. Both PLM (for morphology) and SEM-EDS were used to identify crystal type. Sodium is found primarily in sodium oxalate and sodium aluminosilicate. Solids remaining after BY-waste type saltcake dissolution in water.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminum hydroxide (gibbsite and/or bayerite)	$\text{Al}(\text{OH})_3$	BY-109	BY-saltcake waste type.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The sample from the tank was cascade dissolved in water and a liquid plus solids fraction obtained. The liquid fraction was evaporated to yield precipitated solids. Evaporated supernatant from dissolved liquid studied here. The compound $\text{Al}(\text{OH})_3$ occurs in several forms. Gibbsite, bayerite, and amorphous aluminum hydroxide have been observed in Hanford tank wastes. In addition, Norstrandite and doyleite are the names of two other possible crystallographic forms of $\text{Al}(\text{OH})_3$. Its crystals are generally too small to characterize by PLM, however XRD provides a good analysis technique. Gibbsite and bayerite identified by XRD.
Gibbsite (only)	$\text{Al}(\text{OH})_3$	BY-109	Solids remaining after BY-waste type saltcake dissolution in water.	From 7S110-DLH-06-073. Both PLM (for morphology) and SEM-EDS were used to identify crystal type. This was an In-Tank Solidification (ITS) tank that contained significant amount of aluminum of 0.029 g/g [BBI database], consistent with the presence of this solid formed from evaporated liquid.
Cryolite	Na_3AlF_6	BY-109	Formed from precipitation of evaporating BY-109 dissolved saltcake waste type.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Evaporated supernatant from dissolved liquid studied here. Composite sample. Observed for first time in this report. An unusual crystal to form, it was identified by XRD and SEM-EDS. This was an In-Tank Solidification (ITS) tank that contained significant amount of both fluoride and aluminum of 0.022 g/g and 0.025 g/g, respectively [BBI database], consistent with the presence of this solid.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	BY-109	Evaporated supernatant from dissolved liquid studied here. Composite sample. Formed from precipitation of evaporating BY-109 dissolved saltcake waste type. Composite core sample, see Herting, HNF-8849 Rev.0.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Not an unexpected phase. Identification confirmed from SEM-EDS and slow-scan XRD of washed S-112 residues. The SEM images were taken of the "brightest" images. Particulates in supernate residue. The BBI database U-total concentration of 0.49 mg/g solids basis, considering little expected in aqueous phase, is consistent with solid being present.
Sodium aluminum silicate	$\text{Na}_6\text{Al}_2(\text{SiO}_4)_3$	BY-109	Evaporated supernatant from dissolved liquid studied here. Composite sample. Formed from precipitation of evaporating BY-109 dissolved saltcake waste type. Composite core sample, see Herting, HNF-8849 Rev.0.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The first of two a "novel" phases present is a sodium aluminosilicate phase. Particles with this chemical Compositions were identified by SEM/EDS in residue samples. PLM also recognized this type of particle in BY9Orig. This compound has not been identified by XRD. Phases such as zeolites or cancrinite and sodalite are known to occur in Hanford tank wastes, and would have chemistries and morphologies consistent with this particulate. Particles suspended in supernatant (e.g., residue particulates). This was an In-Tank Solidification (ITS) tank that contained significant amount of both silicon and aluminum of 0.0035 g/g and 0.029 g/g, respectively [BBI database], consistent with the presence of this solid.
Sodium phosphate hydrate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	C-101	CWP1 (62 wt.% by solids mass) and TBP (38 wt.% by solids mass) sludge composite waste type.	From 7S110-JMF-05-015. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. A major constituent (>10 wt.%). TBP waste type has significant phosphate and sodium of 0.025 g/g and 0.27 g/g-solids, respectively [BBI database].

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite	$\text{Al}(\text{OH})_3$	C-101	CWP1, TBP sludge composite waste type.	From 7S110-JMF-05-015. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. A major constituent (>10 wt.%). CWP1 waste type has significant aluminum of 0.031 g/g-solids, however TBP waste does not (0.0018 g/g-solids) [BBI database].
Gibbsite	$\text{Al}(\text{OH})_3$	C-103	CWP1 sludge waste type (only heel left over after retrieval).	From 7S110-DLH-04-015. Identified by XRD. This identification is consistent with the identification of gibbsite in the C-103 heel following retrieval. This tank has been sluiced into tank AN-106, so there is likely still gibbsite in this sludge where it resides in AN-106. CWP1 waste type has significant aluminum of 0.031 g/g-solids [BBI database].
				From PNNL-16738. Purpose of report was to develop release models for key contaminants in C-103 tank, including solids characterization. Gibbsite was identified in the heel left over after the sludge in C-103 was sluiced into AN-106. Thus, there is likely gibbsite in the sludge in AN-106 as well. This mineral was identified by XRD. Gibbsite accounted for more than 90% of the minerals seen by SEM. This identification is consistent with the gibbsite identification observed in bulk C-103 sludge observed prior to sluicing (See 7S110-DLH-04-015). CWP1 waste type has significant aluminum of 0.031 g/g-solids [BBI database].
				From 7S110-DLH-04-015. The techniques of XRD and SEM-EDS were used in solid crystalline phase identification. Both sample types identified gibbsite with 26.0 wt.% Al for untreated sludge and 18.9 wt.% Al for centrifuged solids. Both untreated sludge and centrifuged solids from sample H studied. CWP1 waste type has significant aluminum of 0.031 g/g-solids [BBI database].

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Iron oxide (Hematite)	Fe_2O_3	C-103	CWP1	From 7S110-DLH-04-015. The techniques of XRD and SEM-EDS were used in solid crystalline phase identification. Both sample types identified hematite with 58.6 wt.% Fe for untreated sludge and 63.7 wt.% Fe for centrifuged solids. Both untreated sludge and centrifuged solids from sample H studied. CWP1 waste type has significant Fe of 0.0072 g/g-solids [BBI database].
Gibbsite	$\text{Al}(\text{OH})_3$	C-105	Waste types: TBP (primary) and SR-WASH (secondary). Sludge solids.	From PNNL-13394. Identified by XRD. Seen in direct sampled material. According to the BBI database, the aluminum in the sludge possesses a concentration of 0.0018 g-Al/g-solids.
Iron oxide (Hematite)	Fe_2O_3	C-106	Particulate residue from oxalic acid dissolution of NA sludge waste type solids.	From RPP-17158. Purpose of study was to assess the dissolution of C-106 sludge solids in oxalic/nitric acids. Laboratory experiments. This information could be used in heel dissolution predictions. XRD and SEM-EDS analyses were used as diagnostic tools. The iron concentration in tank possessed a concentration of 0.00032 M in aqueous phase [RPP-8847; LA-UR-96-3860]. Identified as major component.
Aluminum oxide (Boehmite)	AlOOH	C-106	Particulate residue from oxalic acid dissolution of NA sludge waste type solids.	From RPP-17158. Purpose of study was to assess the dissolution of C-106 sludge solids in oxalic/nitric acids. Laboratory experiments. This information could be used in heel dissolution predictions. XRD and SEM-EDS analyses were used as diagnostic tools. The aluminum concentration in tank possessed a concentration of 0.041 M in aqueous phase [RPP-8847; LA-UR-96-3860]. Spectra not given. Identified as minor component.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Silver (zero valent)	Ag	C-106	Organic wash waste (OWW) PUREX waste type.	From PNNL-15372. Purpose of study was to study elemental and speciation (oxidation state) of components in waste. XANES and EXAFS techniques identified elemental zero-valent silver and not AgI as predominant form of silver. This suggests that waste is in a fairly reduced state.
Gibbsite	Al(OH) ₃	C-107	1C, CWP2, SRR sludge composite waste type.	From 7S110-JMF-05-015. Caustic demand test as part of corrosion mitigation project. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g). A major constituent (>10 wt.%).
Sodium fluoride phosphate	Na ₇ F(PO ₄) ₂ -12H ₂ O	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated (laboratory) sluicing using supernate of core samples. Information used to assess dissolution, during sluicing, of tank contents. The techniques of SEM-EDS and PLM were used. Approximately 35 wt.% observed in residual. Cores 68 and 69, and auger sample. The 1C waste type, according to the BBI database, contains very high concentrations of F (0.010 g-F/g) and PO ₄ (0.069 g-PO ₄ /g). Phosphate compounds are generally soluble and will sluice well.
Gibbsite	Al(OH) ₃	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated (laboratory) sluicing using supernate of core samples. Information used to assess dissolution, during sluicing, of tank contents. Both gibbsite and hematite solids were dissolved in C-107 under these conditions. The techniques of SEM-EDS and PLM were used. Between 10-35 wt.% observed in original sample. Cores 68 and 69, and auger sample. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g). The simulated sluicing will reduce hydroxide, enhancing gibbsite dissolution.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Iron oxide (Hematite)	Fe_2O_3	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. Both gibbsite and hematite solids were dissolved in C-107. The techniques of SEM-EDS and PLM were used. Between 10-25 wt.% observed in original sample. Cores 68 and 69, and auger sample, examined. The 1C waste type, according to the BBI database, contains a very high concentration of Fe (0.010 g-Fe/g).
Sodium uranium phosphate	$\text{Na}(\text{UO}_2)(\text{PO}_4)_2$	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. The techniques of SEM-EDS and PLM were used. Between 10-40 wt.% observed in original sample. Cores 68 and 69, and auger sample, examined. The 1C waste type, according to the BBI database, contains a very high concentration of U-total (0.0019 g-U/g) and PO4 (0.069 g-PO4/g).
Sodium nitrate (nitratine)	NaNO_3	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. The techniques of SEM-EDS and PLM were used. Approximately 20 wt.% observed in original sample. Cores 68 and 69, and auger sample. The 1C waste type, according to the BBI database, contains a very high concentration of nitrate (0.13 g-NO3/g).

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrite	NaNO_2	C-107	1C, CWP2, SRR sludge composite waste type.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. The techniques of SEM-EDS and PLM were used. Approximately 25 wt.% observed in original sample. Cores 68 and 69, and auger sample. The 1C waste type, according to the BBI database, contains a very high concentration of nitrite (0.0097 g-NO ₂ /g).
Gibbsite	$\text{Al}(\text{OH})_3$	C-108	Waste types: TBP-F (primary) and 1C (secondary). Sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by XRD. Seen in both as-received and caustic leached (3 M) solids. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g).
Sodium fluoride phosphate (natrophosphate)	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 12\text{H}_2\text{O}$	C-108	Sludge composite auger sample, 1C sludge material.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. For C-108, the simulated sluicing chemically removed gibbsite and phosphates. The techniques of SEM-EDS and PLM were used. Approximately 75 wt.% observed in original (auger) sample. The 1C waste type, according to the BBI database, contains very high concentrations of F (0.010 g-F/g) and PO ₄ (0.069 g-PO ₄ /g).
				From LAB-RPT-10-00001 Rev.0. Direct-sampled composite core examined using PLM and SEM/EDS. This was a major phase existing in tank waste.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite	$\text{Al}(\text{OH})_3$	C-108	Sludge composite auger sample, C1 sludge material.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. For C-108, the simulated sluicing removed gibbsite and phosphates. The techniques of SEM-EDS and PLM were used. Approximately 15 wt.% observed in original (auger) sample. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g). The simulated sluicing will reduce hydroxide, enhancing dissolution.
				From LAB-RPT-10-00001 Rev.0. Caustic-washed composite core examined using SEM/EDS. This was a minor phase identified in the tank waste. Amount not quantified.
Iron oxide (Hematite)	Fe_2O_3	C-108	Sludge composite auger sample, C1 sludge material.	From WRPS-0901014. The technique of SEM-EDS was used. Approximately 5 wt.% observed in original (auger) sample. The 1C waste type, according to the BBI database, contains a very high concentration of Fe (0.010 g-Fe/g).
Aluminosilicates	Al-Si-O zeolite	C-108	1C Sludge solids.	From LAB-RPT-10-00001 Rev.0. Caustic-washed composite core examined using SEM/EDS. This was a minor phase identified in the tank waste. Amount not quantified.
Calcium phosphate	$\text{Ca}_3(\text{PO}_4)_2 \cdot x\text{H}_2\text{O}$	C-108	1C Sludge solids.	From LAB-RPT-10-00001 Rev.0. Caustic-washed composite core examined using SEM/EDS. This was a minor phase identified in the tank waste. Amount not quantified.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	C-108	Sludge composite auger sample, C1.	From WRPS-0901014. Behavior seen during simulated sluicing of core samples. Information used to assess dissolution, during sluicing, of tank contents. The techniques of SEM-EDS and PLM were used. Approximately 5 wt.% observed in original (auger) sample. The 1C waste type, according to the BBI database, contains a very high concentration of U-total (0.0019 g-U/g).
Nastrophite	$\text{NaSr}(\text{PO}_4)_x \cdot 9\text{H}_2\text{O}$	C-108	Sludge solids composite core, 1C waste type.	From LAB-RPT-10-00001 Rev.0. Caustic-washed composite core examined using XRD. This was the only major phase readily identified in the tank waste.
Gibbsite	$\text{Al}(\text{OH})_3$	C-109	Waste sludge types: TBP-F (primary) and 1C (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by SEM-EDS and XRD. Seen in both direct-sampled and caustic leached (3M) materials. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g). The TBP waste template contains approximately an order of magnitude less of Al.
Gibbsite	$\text{Al}(\text{OH})_3$	C-112	Waste sludge types: TBP-F (primary) and 1C (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by SEM-EDS and XRD. Seen in direct sampled material undergoing leaching. The 1C waste type, according to the BBI database, contains a very high concentration of Al (0.015 g-Al/g). The TBP waste template contains approximately an order of magnitude less of Al.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium diuranate and/or clarkeite	$\text{Na}_2\text{U}_2\text{O}_7$ and/or $\text{Na}[(\text{UO}_2)\text{O}(\text{O H})]$ hydrate	C-203	HS-waste type.	From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Images are of hexagonal acicular crystals. Water washed sludge solids. The HS waste type contains significant U-total at 0.041 g-U/g [BBI database]. From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Yellow nugget crystals. Water washed sludge solids. The HS waste type contains significant U-total at 0.041 g-U/g and TOC of 0.018 g-TOC/g [BBI database].
Sodium nitrate (amorphous)	NaNO_3	C-203	HS-waste type.	From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Images are of amorphous solids. Water washed sludge solids. The HS waste type contains only a moderate amount of nitrate at 0.027 g- NO_3 /g [BBI database].
Goethite	$\text{FeO}(\text{OH})$	C-203	Organic wash waste (OWW) PUREX waste type.	From PNNL-15372. Direct sampled solids analyzed. It was observed from micro-XRD that this mineral type existed in solids examined. Solid consistent with inventory of waste.
Clarkeite	$\text{Na}[(\text{UO}_2)\text{O}(\text{O H})]$	C-203	Organic wash waste (OWW) PUREX waste type.	From PNNL-15372. Direct sampled solids analyzed. It was observed from micro-XRD that this mineral type existed in solids examined. Solid consistent with inventory of waste.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	C-203	Organic wash waste (OWW) PUREX waste type.	From PNNL-15372. Direct sampled solids analyzed. It was observed from micro-XRD that this mineral type existed in solids examined. Solid consistent with inventory of waste.
Cejkaite	Na-Al-P-O containing crystals	C-204	HS-waste type.	From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Yellow nugget crystals. Water washed sludge solids. The HS waste type, according to the BBI database, did not give neither phosphate nor aluminum concentrations of solids. No justification for the presence of this solid can be given.
Sodium uranate and/or clarkeite	$\text{Na}_2\text{U}_2\text{O}_7$ and/or $\text{Na}[(\text{UO}_2)\text{O}(\text{O H})]$ hydrate	C-204	HS-waste type.	From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Images are of hexagonal acicular crystals. Water washed sludge solids. The HS waste type contains significant U-total at 0.041 g-U/g [BBI database].
Iron-chromium-nickel crystals	Fe-Cr-Ni-O-C crystals	C-204	HS-waste type.	From PNNL-14903. Purpose of study was to develop release models for key contaminants remaining in sludge after closure of tank C-203. This, in part, required identification of solid phases present in heel material. Identified by XRD and SEM-EDS. Images are of hexagonal angular acicular crystals. Water washed sludge solids. The HS waste type contains significant: Fe at 0.080 g-Fe/g; Cr at 0.0015 g-Cr/g; and Ni at 0.00017 g-Ni/g [BBI database].

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminum oxide (Boehmite)	Al(O)OH	S-101	Waste types: R (primary) and EB (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through TEM. Sludge solids. Seen in both direct-sampled and caustic leached (at 3 M concentration) materials. According to the BBI database, the aluminum concentration is 0.047 M, suggesting sufficient aluminum for a solid, such as boehmite, to be present.
Aluminum oxide (Boehmite)	Al(O)OH	S-104	Sludge solids. Waste type is R.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through TEM. Sludge solids. Seen in both direct-sampled and caustic leached (3M) materials. From BBI database, the g-Al/g-solids value is 0.045, thus sufficient quantity for boehmite formation exist in this tank.
Aluminum oxide (Boehmite)	Al(O)OH	S-107	Waste types: R (primary) and EB (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through TEM. Sludge solids. Seen in both direct-sampled and caustic leached (3M) materials. From BBI database, the g-Al/g-solids value is 0.045 for aluminum, thus sufficient quantity for boehmite formation exists.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite	$\text{Al}(\text{OH})_3$	S-110	R1 plus Some CWR1 sludge waste types, cores 240 and 241. The sample was an amalgam of these waste types.	From PNNL-13702. The purpose of the study was to measure caustic leach factors to serve as a baseline for the process flowsheet. The reference provided very little supporting information for this identification, simply stated that it was identified by XRD. However, caustic leach data presented for the data did indicate there was a source of rapidly leached aluminum in the sample, consistent with gibbsite. The TWINS inventory indicated a concentration of aluminum of 0.04 g-Al/g-solids. Also confirmed in: From PNNL-14018. Caustic leaching performed to support process flowsheet development. Crystal phase identified from XRD. Washed thrice with 0.01M NaOH. Composite waste type of A1, A2, B, S1, S2, and R saltcakes. Residual solids analyzed.
Aluminum oxide (Boehmite)	AlOOH	S-110	R1 plus Some CWR1 sludge waste types, cores 240 and 241. The sample was an amalgam of these waste types.	From PNNL-13702. The purpose of the study was to measure caustic leach factors to serve as a baseline for the process flowsheet. The reference provided very little supporting information for this identification, simply stated that it was identified by XRD. However, caustic leach data presented for the data did indicate there was a very slowly leached aluminum phase in the sample, consistent with boehmite. The TWINS inventory indicated a concentration of aluminum of 0.04 g-Al/g-solids. Also confirmed in: From PNNL-14018. Caustic leaching performed to support process flowsheet development. Crystal phase identified from XRD. Washed thrice with 0.01M NaOH. Composite waste type of A1, A2, B, S1, S2, and R saltcakes. Residual solids analyzed.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	S-110	Evaporated precipitated mixture of S-110 and U-107 supernate materials obtained originally from cascade dissolution.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. This crystal is difficult to conclusively distinguish from sodium carbonate using PLM and SEM-EDS. However, it has a definitive XRD pattern that can be used to confirm its presence. Confirmed by PLM, SEM-EDS, and XRD. The TWINS inventory indicated a concentration of oxalate of 0.002 g/g-solids. Although this value is somewhat low, precipitation of solids will increase the abundance of this material.
Sodium nitrate (nitratine)	NaNO_3	S-112	R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. Sample was resistant to water sluicing dissolution, and contained yellow solid residue. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-112 tank. The sodium nitrate (major) phase was at 47 wt.% solids basis. According to the BBI database, for R1 the nitrate concentration is 140 g/L.
Sodium carbonate (thermonatrite)	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. Sample was resistant to water sluicing dissolution, and contained yellow solid residue. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. The sodium carbonate was the third most abundant phase at 8.9 wt.%. According to the BBI database, for R1 the carbonate concentration is only 0.23 g/L. This is too low to account for the presence of this solid. Perhaps atmospheric carbon dioxide could have absorbed into the caustic sample precipitating thermonatrite.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrite	NaNO_2	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. Sample was resistant to water sluicing dissolution, and contained yellow solid residue. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. The sodium nitrite was the fifth most abundant phase at 1.9 wt.%. According to the BBI database, for R1 the nitrite concentration is 35 g/L, sufficient for solids to form.
Burkeite	$\text{Na}_6\text{CO}_3(\text{SO}_4)_2$	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. The sodium carbonate-sulfate phase was the second most abundant phase at 9.8 wt.%. According to TWINS inventory, carbonate has not been measured in the supernate, however sulfate possesses a high concentration of 6.4 g/L and in the solid fraction 0.11 g-SO ₄ /g-solids. According to the BBI database, for R1 the carbonate concentration is only 0.23 g/L. This makes the high abundance of this material, for this tank, somewhat surprising.
Sodium chromate	Na_2CrO_4	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. Only a trace amount of Na_2CrO_4 observed. The yellow color was due to a chromate solid phase.
Sodium phosphate	Na_3PO_4	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. Only a trace amount of Na_3PO_4 observed.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	S-112	Sample was resistant to water sluicing dissolution, and primarily contained yellow solid residue, as part of R1 sludge solids waste type.	From CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-102 tank. Only a trace amount of $\text{Na}_2\text{C}_2\text{O}_4$ observed.
Al-Cr particulates	Al-Cr rich amorphous phase	S-112	R1 sludge solids exist in S-112 according to BBI at heel level of 8.6 kL.	From FH-0202771. Caustic dissolution experiment. Techniques of PLM, SEM-EDS, and XRD used to investigate solid composition/crystal type. Report states solid saltcake waste type. From TWINS inventory, the Al and Cr concentrations are listed as 0.22 g-Al/g-solids and 0.005 g-Cr/g-solids, respectively. These high concentrations are consistent with presence of this phase.
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	S-112	R1 sludge solids exist in S-112 according to BBI at heel level of 8.6 kL.	From FH-0202771. Techniques of PLM, SEM-EDS, and XRD used to investigate solid composition/crystal type. Less than 5 microns in diameter for these crystals. Solid saltcake caustic dissolution in water and 2M NaOH, cores 291 and 292. Report states solid saltcake waste type. From TWINS inventory, the concentration of U is 0.0048 g-U/g-solids. This high concentration is consistent with the common form of uranium.
Ca-Cr crystallites	$\text{Ca}_3\text{Cr}_2(\text{OH})_{12}$	S-112	R1 sludge solids exist in S-112 according to BBI at heel level of 8.6 kL.	From FH-0202771. Techniques of PLM, SEM-EDS, and XRD used to investigate solid composition/crystal type. Particulates generally less than 10 microns in size. Solid saltcake caustic dissolution in water and 2M NaOH, cores 291 and 292. Report states solid saltcake waste type. From the TWINS inventory, the concentrations of Ca and Cr are 0.0021 g-Ca/g-solids and 0.005 g-Cr/g-solids, respectively. These are sufficient for the presence of this hydroxide phase.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Lithium aluminum hydroxide	$\text{LiAl}_2(\text{OH})_7 \cdot n\text{H}_2\text{O}$	S-112	R1 sludge solids exist in S-112 according to BBI at heel level of 8.6 kL.	From FH-0202771. Techniques of PLM, SEM-EDS, and XRD used to investigate solid composition/crystal type. Identified in only one sample. This seems to be a new/unique crystal type. Solid saltcake caustic dissolution in water and 2M NaOH, cores 291 and 292.
Sodium carbonate hydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	S-112	Report states that identification of waste type not possible [see HNF-11585], although R1 sludge is known to exist in S-112 at 8.6 kL heel level.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. Crystals formed from precipitated supernate following cascade water dissolution of composite saltcake. According to the BBI database, for R1 the carbonate concentration is 0.23 g/L, consistent with solids precipitated from concentrated supernatant but not supernate present in the tank.
Gibbsite	$\text{Al}(\text{OH})_3$	S-112	R1 sludge solids exist in S-112 according to BBI at heel level of 8.6 kL.	From PNNL-17593. Study purpose was to assess leachability of components from tank waste solids. Water washing of samples performed, followed by particulate analysis. Identified by XRD and consistent with SEM observations. Note that there was only a small amount of R1 sludge in this tank, and it is possible that the gibbsite in this sample is aluminum that precipitated from a saltcake in the tank prior to retrieval rather than from the R1 sludge. From TWINS inventory, however, the Al concentration is listed as 0.22 g-Al /g-a solid, which is responsible for the presence of gibbsite.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Hydrogarnet-hydrouvarovite	Likely to be (Ca,Sr) ₃ (Cr,Al) ₂ (OH) ₁₂	S-112	R1 waste type obtained from incomplete (cascade) dissolution of sludge material.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The second of two novel phases from this report is a distinctive chemical combination of calcium, strontium, chromium and aluminum. XRD analysis indicates that this compound has a crystalline structure that is consistent with the garnet group of minerals. If so, then this phase is identified as a previously unknown hydrogarnet- hydrouvarovite compound. Caustic leaching of solid material. BBI Waste Type Template document [RPP-8847] suggests that these minerals are in great enough abundance (Ca,Al) to be identifiable in a washed saltcake sample.
Gibbsite	Al(OH) ₃	S-112	Cladding Waste (CWR) and S Saltcake from report. However, BBI only has R1 sludge solids in tank S-112 at 8.6 kL heel level.	From PNNL-17593. Study purpose was to assess leachability of components from tank waste solids. Identified by XRD and SEM. This phase was also found in the water-washed residues after laboratory simulation of the retrieval process reported in FH-0202771. Gibbsite was the dominant mineral in the sludge. The gibbsite was usually coated with a Na-Al bearing phase, probably dawsonite. Heel after retrieval. From TWINS total inventory, the Al concentration is listed as 0.22 g-Al/g-solids, which could be responsible for the presence of gibbsite.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
			Waste type is R1 sludge according to BBI. Pre-leaching particle types identified here.	From 74A10-WSC-08-152. Purpose of the study was to determine the physical nature of the material in the tank after retrieval operations. This is a necessary step in tank closure. The techniques of PLM, SEM-EDS, and XRD confirmed the analysis of material as Gibbsite, with dimensions as large as 100 μm (larger than typically seen). This was major component. Caustic dissolution identified the material as sludge solids, not saltcake. This is contrary to prior studies. From TWINS inventory, however, the Al concentration is listed as 0.22 g-Al/g-solids, that could be responsible for the presence of gibbsite.
Dawsonite	$\text{NaAl}(\text{OH})\text{CO}_3$	S-112	Heel after retrieval of REDOX Process, R1 sludge solids in tank S-112.	From PNNL-17593. Study purpose was to assess leachability of components from tank waste solids. This is a speculative identification based on SEM. The SEM identified a phase that coated the gibbsite particles made up of Na and Al. Dawsonite is the most reasonable identification for this phase because sodium aluminate would be too soluble to be in the waste. From TWINS inventory, however, the Al concentration is listed as 0.22 g-Al/g-solids, together with high carbonate, supports the presence of Dawsonite.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite	$\text{Al}(\text{OH})_3$	S-112	R1 sludge solids.	74A10-WSC-08-152. Purpose of the study was to determine the physical nature of the material in the tank after retrieval operations. This is a necessary step in tank closure. The techniques of PLM, SEM-EDS, and XRD confirmed the analysis of crystalline material. Three experiments performed: (1) direct composite solids; (2) water solubility tests; and (3) caustic digest (19 M) NaOH. Gibbsite was identified as a major component. From TWINS inventory, however, the Al concentration is listed as 0.22 g-Al/g-solids, which could be responsible for the presence of gibbsite. Caustic dissolution identified the material as sludge solids, not saltcake. This is contrary to prior studies. Pre-leaching particle types identified here.
Sodium diuranate	$\text{Na}_2\text{U}_2\text{O}_7$	S-112	Heel after retrieval of REDOX Process, Cladding Waste (CWR) and S Saltcake. However, BBI only has R1 sludge solids contained in tank S-112.	See report FH-0202771. The experiment took saltcake from a sample taken prior to retrieving the tank and dissolved the saltcake in water. The sodium diuranate was identified in residuals that did not dissolve in water by XRD and SEM. This phase was not identified in the heel samples evaluated in PNNL-17593, probably because there was little uranium found in the heel samples. Nonetheless, what little uranium that is in the heel of S-112 is likely in the form of sodium diuranate, given it was conclusively identified in the report FH-0202771. The sodium diuranate was extremely small, 4 microns by SEM, so most of the uranium likely was suspended in the retrieval fluid, given the small abundance in the heel.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate (nitratine)	NaNO_3	S-112	S Saltcake waste type from report [CH2M-0400924]. However, BBI only has R1 sludge solids in tank S-112 (e.g., no saltcake).	See report CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-112 tank. This was identified by XRD. This was identified in the saltcake, which has since been dissolved. Consequently, there is little chance that such a soluble phase would still be present in the heel in S-112, but is indicative of what might be present in other S saltcakes or what might precipitate if this waste is ever concentrated, say through evaporation, again. Approximately 47 wt.% abundant.
Thermonatrite (sodium carbonate monohydrate)	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	S-112	S Saltcake waste type from report [CH2M-0400924]. However, BBI only has R1 sludge solids in tank S-112 (e.g., no saltcake).	See report CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-112 tank. This was identified by XRD. This was identified in the saltcake, which has since been dissolved. Consequently, there is little chance that such a soluble phase would still be present in the heel in S-112, but is indicative of what might be present in other S saltcakes or what might precipitate if this waste is ever concentrated again. Approximately 9 wt.% abundant. Also confirmed in: 74A10-WSC-08-152. Purpose of the study was to determine the physical nature of the material in the tank after retrieval operations. This is a necessary step in tank closure. The techniques of PLM, SEM-EDS, and XRD confirmed the analysis of crystalline material. Three experiments performed: (1) direct composite solids; (2) water solubility tests; and (3) caustic digest (19 M) NaOH. Trace amount of sodium carbonate detected. Pre-leaching particle types identified here.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium Nitrite	NaNO_2	S-112	S Saltcake waste type from report [CH2M-0400924]. However, BBI only has R1 sludge solids in tank S-112 (e.g., no saltcake).	See report CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-112 tank. This was identified by XRD. This was identified in the saltcake, which has since been dissolved. Consequently, there is little chance that such a soluble phase would still be present in the heel in S-112, but is indicative of what might be present in other S saltcakes or what might precipitate if this waste is ever concentrated again. Approximately 2 wt.% abundant.
Burkeite (sodium carbonate sulfate)	$\text{Na}_6\text{CO}_3(\text{SO}_4)_2$	S-112	S Saltcake waste type from report [CH2M-0400924]. However, BBI only has R1 sludge solids in tank S-112 (e.g., no saltcake).	See report CH2M-0400924. Core 292 segment 5 material. SEM-EDS and XRD were used to analyze and identify solid material contained in a solid residue in the S-112 tank. This was identified by XRD. This was identified in the saltcake, which has since been dissolved. Consequently, there is little chance that such a soluble phase would still be present in the heel in S-112, but is indicative of what might be present in other S saltcakes or what might precipitate if this waste is ever concentrated again. Approximately 10 wt.% abundant.
			NA saltcake from concentrated NA-supernate. In support of retrieval.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used XRD and PLM to identify solids in dissolved saltcake sample. From TWINS inventory, however, the Al concentration is listed as 0.10 g-SO ₄ /mL, together with high carbonate, supports the presence of mineral phase.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminum hydroxide (gibbsite)	$\text{Al}(\text{OH})_3$	SX-101	NA saltcake from concentrated NA-supernate. In support of retrieval.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The compound $\text{Al}(\text{OH})_3$ occurs in several forms. The phases gibbsite, bayerite, and amorphous aluminum hydroxide have been observed in Hanford tank wastes. In addition, Norstrandite and doyleite are the names of two other possible crystallographic forms of $\text{Al}(\text{OH})_3$. Its crystals are generally too small to characterize by PLM, however XRD provides a good analysis technique. Gibbsite identified by XRD. Crystals remaining following cascade water dissolution of composite saltcake. From TWINS database, the aluminum total concentration in the solids is approximately 0.06 g-Al/g-solids.
Sodium nitrite	NaNO_2	SX-101	Waste type not specified. From Best Basis Inventory must be R, R1, S1, and/or S2 waste type(s).	From HNF-13805. Both simulants formed sodium nitrite solid particulates and those obtained from tank SX-101 via direct analyses have been compared. No additional corroboration, say from SEM-EDS or XRD was mentioned. From TWINS database, the nitrite total concentration in the solids is approximately 0.04 g-NO_2/g-solids, so it is a substantial fraction of the solids.
Aluminum oxide (Boehmite)	$\text{Al}(\text{O})\text{OH}$	SX-108	R sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through TEM. Sludge solids. Seen in both direct-sampled and caustic leached (3M) materials. From TWINS database, the aluminum total concentration in the solids is approximately 0.30 g-Al/g-solids. This high solids fraction explains it being a major constituent.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium oxalate (natroxalate)	$\text{Na}_2\text{C}_2\text{O}_4$	SY-101	S1 saltcake. [see Esch, 2000; FH-0003514].	From RPP-6517. Most abundant solid phase. Approximately 40% of solid phase for this crystal type. From BBI database, solid composition has 0.090 g-oxalate/g-solids. Therefore presence in solid phase well documented in native tank waste material.
Gibbsite	$\text{Al}(\text{OH})_3$	SY-101	S1 saltcake. [see Esch, 2000; FH-0003514].	From RPP-6517. Second most abundant solid phase. Approximately 30% of solid phase for this crystal type. From BBI database, solid composition has 0.036 g-aluminum/g-solids. Therefore presence in solid phase is reasonable.
Sodium phosphate hydrate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	SY-101	S1 saltcake. [see Esch, 2000; FH-0003514].	From RPP-6517. Third most abundant solid phase. Approximately 20% of solid phase for this crystal type. From BBI Waste Template compositions [RPP-8847], the phosphate solids composition is 0.0044 g- PO_4 /g-solids.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 12\text{H}_2\text{O}$	SY-101	S1 saltcake. [see Esch, 2000; FH-0003514].	From RPP-6517. Fourth most abundant solid phase. Approximately 10% of solid phase for this crystal type. From BBI Waste Template compositions [RPP-8847], the phosphate solids composition is 0.0044 g- PO_4 /g-solids and the fluoride is 0.000027 g-F/g-solids. This supports presence of solid phase, albeit in low abundance.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Calcium carbonate or Calcium oxalate	CaCO_3 or CaC_2O_4	SY-101	S1 saltcake. [see Esch, 2000; FH-0003514].	From RPP-6517. Fifth most abundant solid phase. Approximately 1% (trace amount) of solid phase for this crystal type. Identified by EDS only (e.g., no imaging), so analysis technique cannot distinguish between carbonate from oxalate. From BBI database, solid composition has 0.00040 g-calcium/g-solids. Therefore presence in solid phase well documented in native tank waste material. Understandably less than oxalate and gibbsite materials.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 12\text{H}_2\text{O}$	SY-102	Solids waste type, although not specified, is likely NA sludge plus NA liquid. The other possibility is R1 sludge, but due to the grab sample location and amount of solids (50-50 vol.% solids-liquid), NA waste type is most probable.	From 7S110-DLH-05-041. Grab sample from Riser 3 (2SY-05-09). Major phase. Identified by PLM and SEM-EDS, but not XRD. Settled solids sample obtained from sampling 6 inches below liquid-solid phase interface. Direct sampling of solids from collected slurry sample. From TWINS database, the phosphate and fluoride concentrations are 0.008 g- PO_4 /g-solids and 0.0005 g-F/g-solids. The fluoride, although observed in the solids fraction, is somewhat low for the observed abundance seen.
Sodium oxalate or sodium carbonate	$\text{Na}_2\text{C}_2\text{O}_4$ or $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	SY-102	Solids waste type, although not specified, is likely NA sludge plus NA liquid. The other possibility is R1 sludge, but due to the grab sample location and amount of solids (50-50 vol.% solids-liquid), NA waste type is most probable.	From 7S110-DLH-05-041. Grab sample from Riser 3 (2SY-05-09). Major phase. Identified by PLM, SEM-EDS, but not XRD. Analysis distinguishing two components was inconclusive. Settled solids sample obtained from sampling 6 inches below liquid-solid phase interface. Direct sampling of solids from collected slurry sample. From TWINS database, the oxalate concentration was observed to be 0.002 g-oxalate/g-solids.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Gibbsite or Al/Cr salts	Al(OH) ₃ Or Al/Cr salts	SY-102	Solids waste type, although not specified, is likely NA sludge plus NA liquid. The other possibility is R1 sludge, but due to the grab sample location and amount of solids (50-50 vol.% solids-liquid), NA waste type is most probable.	From 7S110-DLH-05-041. Grab sample from Riser 3 (2SY-05-09). Major phase. Identified by PLM, SEM-EDS and XRD. XRD identified gibbsite with varying amounts of chromium impurity. Settled solids sample obtained from sampling 6 inches below liquid-solid phase interface. Direct sampling of solids from collected slurry sample. From TWINS database, the aluminum concentration was observed to be 0.018 g-aluminum/g-solids.
Sodium diuranate	Na ₂ U ₂ O ₇	SY-102	Solids waste type, although not specified, is likely NA sludge plus NA liquid. The other possibility is R1 sludge, but due to the grab sample location and amount of solids (50-50 vol.% solids-liquid), NA waste type is most probable.	From 7S110-DLH-05-041. Grab sample from Riser 3 (2SY-05-09). Major phase. Identified by SEM-EDS and XRD, but not PLM (too small crystal size). Settled solids sample obtained from sampling 6 inches below liquid-solid phase interface. Direct sampling of solids from collected slurry sample. From TWINS database, the uranium concentration was observed to be 0.00085 g-U/g-solids, sufficient to be a minor phase present.
Sodium phosphate dodecahydrate	Na ₃ PO ₄ -12H ₂ O	SY-102	Solids waste type, although not specified, is likely NA sludge plus NA liquid. The other possibility is R1 sludge, but due to the grab sample location and amount of solids (50-50 vol.% solids-liquid), NA waste type is most probable.	From 7S110-DLH-05-041. Grab sample from Riser 3 (2SY-05-09). Major phase. Identified by SEM-EDS, but not XRD nor PLM. Settled solids sample obtained from sampling 6 inches below liquid-solid phase interface. Direct sampling of solids from collected slurry sample. From TWINS database, the phosphate concentration is 0.008 g-PO ₄ /g-solids. High phosphate concentration consistent with observed solid phase seen. From 7S110-DLH-06-068. Supernate samples were cooled below ambient temperature to see if precipitation of solids occurred. It is stated that the samples contain approximately 0.055 M phosphate, by an unspecified analysis technique (either ICP or IC). PLM was used via morphology to identify crystal type (they were needles). From TWINS database, the phosphate solids concentration is 0.005 g-PO ₄ /g-solids, sufficient to account for observed solids amount.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Plutonium oxide	PuO_2	SY-102	Not specified but likely NA sludge centrifuged REDOX solids waste type.	From CH2M-0400872. Study characterized settling behavior of plutonium oxide particles for criticality issue. PuO_2 comprised 7.4 wt.% of fines and 5 wt.% overall, and therefore are a substantial amount of particulate material present.
Gibbsite	$\text{Al}(\text{OH})_3$	SY-103	CC waste type sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM, SEM-EDS and XRD. Seen in both direct-sampled and caustic leached (3M) materials.
Gibbsite	$\text{Al}(\text{OH})_3$	T-104	Sludge solids. Waste type is 1C.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM/SEM-EDS and XRD. Seen in direct-sampled, leached, material. According to the BBI database, the aluminum solids concentration is 0.015 g-Al/g-solids. This supports the high abundance of this phase.
Aluminum phosphate	AlPO_4	T-104	1C Sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM/SEM-EDS and XRD. Seen in direct-sampled material. According to the BBI database, the aluminum solids concentration is 0.015 g-Al/g-solids and the PO_4 concentration is 0.069 g- PO_4 /g-solids. These high concentrations support the high abundance of this phase.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Aluminosilicates	Al-Si-O zeolite	T-104	1C Sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM/SEM-EDS and XRD. Seen in both direct-sampled and caustic leached (3M) materials. According to the BBI database, the aluminum solids concentration is 0.015 g-Al/g-solids and the silicon concentration is 0.0062 g-Si/g-solids. These high concentrations support the moderate abundance of this phase.
Sodium iron aluminum phosphate	Na_2Fe_2 $\text{Al}(\text{PO}_4)_3$	T-104	1C Sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM/SEM-EDS and XRD. Seen in direct-sampled material. According to the BBI database, the aluminum solids concentration is 0.015 g-Al/g-solids, the PO4 concentration is 0.069 g-PO4/g-solids, and the iron concentration is 0.010 g-Fe/g-solids. These high concentrations only offer weak support of this unusual and non-typical mineral phase.
Gibbsite	$\text{Al}(\text{OH})_3$	T-107	1C (primary) and CW (secondary) sludge solids.	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by XRD. According to the BBI database, the aluminum solids concentration is 0.015 g-Al/g-solids (for 1C waste type). This supports the high abundance of this phase.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate sulfate	$\text{Na}_3\text{NO}_3\text{SO}_4$	TX-113	From Best Basis Inventory must be T2 saltcake and/or 1C sludge particulates.	From HNF-13805. Both simulant formed sodium nitrate sulfate solid particulates and those obtained from tank TX-113 have been compared. No additional corroboration, say from SEM-EDS or XRD was mentioned. This report was an American Chemical Society presentation so little experimental detail was given.
			T2-saltcake. Direct sampling of solids.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used XRD and PLM to identify solids in dissolved saltcake sample. According to the BBI database, the T2 waste type contains both high sulfate at 0.0080 g-SO ₄ /g-solids and high nitrate at 0.15 g-NO ₃ /g-solids, consistent with the presence of this phase.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 12\text{H}_2\text{O}$	TX-113	1C sludge solid. A 1998 sample, core 253.	From HNF-7031 Rev.0. Inhibited water (0.01M NaOH) was used to simulate sluicing dissolution of sludge solid sample. Solid existed after contacts of 2,3,4 sluicing attempts. Identified by PLM and SEM/EDS.

Table 1-1. Crystalline phases identified in Hanford tank waste samples. (56 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	U-107	S2 and/or T2 saltcake.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used polarized light microscopy (PLM) and XRD to identify this phase in the dissolved saltcake sample. PLM showed isotropic crystals. Identification based on comparison between standard and a 50-50 (v/v) waste mix of BY-109 and U-107 samples. Obtained from evaporation of water-dissolved saltcake material. According to the BBI database, the T2 waste type possess 0.0027 g-F/g-solids and 0.014 g-PO ₄ /g-solids, sufficient for this mineral to be present in dissolved saltcake material.
Sodium fluoride sulfate	Na_3FSO_4	U-107	S2 and/or T2 saltcake.	From HNF-11585. Purpose of study was to measure physical and chemical data for support of Hanford waste retrieval and remediation. The reference used polarized light microscopy (PLM) and XRD to identify this phase. PLM showed isotropic crystals. Identification based on comparison between standard and a 50-50 (v/v) waste mix of BY-109 and U-107 samples. Obtained from evaporation of water-dissolved saltcake material. According to the BBI database, the T2 waste type possess 0.0027 g-F/g-solids and 0.015 g-SO ₄ /g-solids, sufficient for this mineral to be present in dissolved saltcake material.
Aluminosilicate	Al-Si-O solids	U-110	Sludge solids. Waste types are 1C (primary) and CW (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by SEM-EDS and XRD. Seen in direct-sampled, leached, material. According to the BBI database, the solids concentrations are 0.015 g-Al/g-solids and 0.0062 g-Si/g-solids. These concentrations are sufficient for minor abundances of aluminosilicates to be present.

2.0 Group 1-8 Solid Sample Waste Type Mineralogy

To date there are nine WTP-PNNL reports issued that summarize mineralogical phase of actual waste used in various investigations. These reports identify eight groups of composite samples collected from multiple tanks for each group. Information in these reports included a solids characterization to support caustic and oxidative leaching, and water washing of waste. These investigations warrant a separate table from Table 1-1 because there are eight groups of solid waste that were studied that did not have its origin a single tank. Each of these groups was a composite of solids from several Hanford tanks of the same waste type. The minerals identified are listed in Table 2-1 below.

Table 2-1. A summary of Waste Treatment and Immobilization Plant Pacific Northwest National Laboratory (WTP-PNNL) studies involving Hanford actual waste solid material. (5 pages)

Mineral	Chemical Formula	Tank / Report	Experimental Techniques and Waste Type Discussion
Boehmite	AlOOH	REDOX (sludge) / PNNL-17368 (Group 5)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. Grouping includes sludge from following tanks: S-101, S-107, S-110, SX-103.
Gibbsite	Al(OH) ₃		
Zeolite	Al-Si-O		
Cancrinite	Na-Al-Si-NO ₃ -H ₂ O		
Sodium uranium oxide	Na ₂ U ₂ O ₇		
Gibbsite	Al(OH) ₃	REDOX (saltcake)/ PNNL-17368 (Group 6)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. Grouping includes saltcake from following tanks: S-101, S-103, S-105, S-106, S-108, S-109, S-110, S-111, S-112, SX-102, SX-105, SX-106, SY-101, SY-103, U-103, U-106, U-108, U-109, U-111.
Boehmite	AlOOH		
Sodium oxalate	Na ₂ C ₂ O ₄		
Cancrinite	Na-Al-Si-NO ₃ -H ₂ O		
Silicon dioxide	SiO ₂		
Sodium uranium oxide	Na ₂ U ₂ O ₇		

Table 2-1. A summary of Waste Treatment and Immobilization Plant Pacific Northwest National Laboratory (WTP-PNNL) studies involving Hanford actual waste solid material. (5 pages)

Mineral	Chemical Formula	Tank / Report	Experimental Techniques and Waste Type Discussion
Chromium oxide	Cr_2O_3		
Nitratine	NaNO_3	Bismuth Phosphate sludge / PNNL-17992 (Group 1)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. 1C and 2C bismuth phosphate waste types. Grouping includes saltcake from following tanks: B-105, B-110, B-112, BX-107, BX-111, BX-112, C-108, C-110, T-104, T-108, T-110, TX-109, TX-110, TX-111, TX-113, TX-114. Only those minerals listed as Excellent, Good, or Probable are given.
Ammonium aluminum hydrogen phosphate	$\text{NH}_4\text{AlHPO}_3\text{O}_{10}$		
Calcium sulfate	$\text{Ca}(\text{SO}_4)(\text{H}_2\text{O})_2$		
Bismuth phosphate	BiPO_4		
Gibbsite	$\text{Al}(\text{OH})_3$	Bismuth Phosphate saltcake / PNNL-17992 (Group 2)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. A, B, BY, R, S, and T bismuth phosphate saltcake waste types. Grouping includes saltcake from following tanks: BX-110, BX-111, BY-104, BY-105, BY-107, BY-108, BY-109, BY-110, BY-112, T-108, T-109, TX-104, TX-113.
Nitrate Cancrinite	$\text{Na}_{7.92}(\text{AlSiO}_4)_6$ $(\text{NO}_3)_{1.7}(\text{H}_2\text{O})_{2.34}$		
Uranocalcarite	$\text{Ca}(\text{UO}_2)_3\text{CO}_3$ $(\text{OH})(\text{H}_2\text{O})_3$		

Table 2-1. A summary of Waste Treatment and Immobilization Plant Pacific Northwest National Laboratory (WTP-PNNL) studies involving Hanford actual waste solid material. (5 pages)

Mineral	Chemical Formula	Tank / Report	Experimental Techniques and Waste Type Discussion
Dorfmanite	$\text{Na}_2\text{HPO}_4(\text{H}_2\text{O})_2$		
Gibbsite	$\text{Al}(\text{OH})_3$	PUREX cladding waste sludge/ PNNL-18054 (Group 3)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. CWP sludge waste type. Grouping includes saltcake from following tanks with percentages given: BY-109(51%), B-108(11%), C-105(14%), B-109(11%), C-103(3%), C-104(10%).
Iron oxide	$\text{Fe}_{1.67}\text{H}_{0.99}\text{O}_3$		
Nitrate Cancrinite and/or Hydroxycancrinite	$\text{Na}_{8.16}(\text{Al}_6\text{Si}_6\text{O}_{24})$ $(\text{NO}_3)_{2.16}(\text{H}_2\text{O})_{1.62}$ $1.06\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot$ $1.60\text{SiO}_2 \cdot 1.60\text{H}_2\text{O}$		
Gibbsite	$\text{Al}(\text{OH})_3$	REDOX cladding waste sludge/ PNNL-18054 (Group 4)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. CWR sludge waste type. Grouping includes saltcake from following tanks with percentages given: U-202(32%), U-204(17%), U-203(4%), U-105(34%), U-201(3%).
Sodium aluminum silicate hydrate	$(\text{Na}_2\text{O})_{1.31}\text{Al}_2\text{O}_3$ $(\text{SiO}_2)_{2.01}(\text{H}_2\text{O})_{1.65}$		
Boehmite	AlOOH		

Table 2-1. A summary of Waste Treatment and Immobilization Plant Pacific Northwest National Laboratory (WTP-PNNL) studies involving Hanford actual waste solid material. (5 pages)

Mineral	Chemical Formula	Tank / Report	Experimental Techniques and Waste Type Discussion
Zeolite	NaAlSiO_4 $(\text{H}_2\text{O})_{1.1}$	TBP waste composite / PNNL-18119 (Group 7)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. TBP sludge waste type. Major components include phosphate, aluminum, sulfate, and iron. Grouping includes saltcake from following tanks BX-109 (primary) and B-106 (minor).
Threadgoldite	$\text{Al}(\text{UO}_2)_2(\text{PO}_4)_2$ $(\text{OH})(\text{H}_2\text{O})_8$		
Sodium Iron Phosphate	$\text{Na}_7(\text{FeP}_2\text{O}_7)_4\text{PO}_4$		
Lepidocrocite	$\text{FeO}(\text{OH})$		
Humboldtine	$\text{C}_2\text{FeO}_4 \cdot 2\text{H}_2\text{O}$		
Iron (III) phosphate oxide	Fe_2PO_5		
Dioxouranium(VI) bis(dihydrogenphosphate(I)) hydrate	$(\text{UO}_2)(\text{H}_2\text{PO}_2)_2$ (H_2O)		
Sodium Uranyl Phosphate	$\text{Na}_6(\text{UO}_2)_2(\text{PO}_4)_4$		
Gibbsite	$\text{Al}(\text{OH})_3$		

Table 2-1. A summary of Waste Treatment and Immobilization Plant Pacific Northwest National Laboratory (WTP-PNNL) studies involving Hanford actual waste solid material. (5 pages)

Mineral	Chemical Formula	Tank / Report	Experimental Techniques and Waste Type Discussion
Gibbsite	$\text{Al}(\text{OH})_3$	Ferrocynide (FeCN) waste / PNNL-18120 (Group 8)	Experimental techniques used include micro-imaging SEM, TEM, XRD, and EELS. 1FeCN, PFeCN, and TFeCN composite waste type. Grouping includes sample include contributions from following tanks BY-110 (major), BY-108, BY-105, BY-104 (minor), BY-106 (minor), plus lesser contributions from other tanks.
Sodium uranium oxide	$\text{Na}_2\text{U}_2\text{O}_7$		
Hematite	Fe_2O_3		
Strontium Hydrogen Phosphite	$\text{Sr}(\text{H}_2\text{PO}_3)_2$		
Hydroxycancrinite	$1.06\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot$ $1.60\text{SiO}_2 \cdot$ $1.60\text{H}_2\text{O}$		
Bassanite	$\text{Ca}(\text{SO}_4)(\text{H}_2\text{O})_{0.5}$ (minor phase)		
Sodium Oxide Cyanide	$\text{Na}_3(\text{CN})\text{O}$		
Sodium Uranyl Carbonate	$\text{Na}_4(\text{UO}_2)(\text{CO}_3)_3$ (minor phase)		

3.0 Boil-down Solid Phase Identification in Hanford Tank Samples

A common Hanford Tank practice uses tank samples, heats them at sub-ambient temperature and pressure, causes evaporation, and initiates solids precipitation. The results are then used to estimate the end point of so-called evaporation campaigns used in tank management by liquid phase reduction. The resulting slurry or solids are cooled to ambient temperature. This practice concentrates the non-volatile material present and reduces the total amount of waste present, via water removal. The rationale for this processing is waste volume minimization. Table 3-1 below lists solids identified from these laboratory (i.e., small scale) studies. The waste type group is not listed here as the solids formed originated from supernate (e.g., the liquid fraction).

An interesting comparison that can be made, on a tank-specific basis, is that for direct saltcake sampling (Table 1) to boil-down solids production in this section (Table 3). Unfortunately, only one comparison can be made, and this corresponds to tank AP-105. In particular, the fluoride-containing solids for this tank reveals that the saltcake contains the phase of Na_3FSO_4 , whereas the $\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$ crystalline phase was identified in the boil-down solid sample. There was no mention of Na_3FSO_4 in the boildown referenced report. From this it can be concluded that other differences must exist between the two precipitated solid materials to account for the discrepancy.

A second comparison that can be made is between observed crystal phases from direct sampling (with caustic treatment in some instances) of section 1, Table 1-1, results with the boildown results of Table 3-1 here. All observed boildown phases were observed as part of the saltcake minerals of Table 1-1, with the exception of sodium nitrite. Examples include cancrinite, sodium oxalate, sodium fluoride phosphate, sodium carbonate, and sodium fluoride sulfate.

Table 3-1. The characteristics of Hanford tank boil-down laboratory studies. (4 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Cancrinite	$\text{Na}_6\text{Ca}_{1.5}\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)_{1.6}$	AP-101	Boil-down from supernatant liquid.	From 7S110-RWW-04-029. Identification techniques in report include PLM, SEM-EDS, and XRD. Here identified by chemical composition and morphology.
Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	AP-101	Boil-down from supernatant liquid.	From 7S110-RWW-04-029. Identification techniques include PLM, SEM-EDS, and XRD.
Sodium fluoride phosphate hydrate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AP-101	Boil-down from supernatant liquid, resulting in solids precipitation.	From 7S110-WSC-08-145. Identification techniques are PLM, SEM-EDS, and XRD. Composition ca.90 wt.%.
Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	AP-101	Boil-down from supernatant liquid, resulting in solids precipitation.	From 7S110-WSC-08-145. Identification techniques are PLM, SEM-EDS, and XRD. Composition ca.9 wt.%.
Sodium nitrate	NaNO_3	AP-101	Boil-down from supernatant liquid, resulting in solids precipitation	From 7S110-WSC-08-145. Identification techniques are PLM, SEM-EDS, and XRD. Composition ca.75 wt.%.
Sodium carbonate hydrate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	AP-101	Boil-down from supernatant liquid, resulting in solids precipitation.	From 7S110-WSC-08-145. Identification techniques are PLM, SEM-EDS, and XRD. Composition ca.10 wt.%.
Sodium nitrite	NaNO_2	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.

Table 3-1. The characteristics of Hanford tank boil-down laboratory studies. (4 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium nitrate	NaNO_3	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.
Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.
Sodium carbonate monohydrate	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.
Sodium fluoride phosphate hydrate	$\text{Na}_7\text{F}(\text{PO}_4)_2\cdot 19\text{H}_2\text{O}$	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.
Sodium phosphate dodecahydrate	$\text{Na}_3\text{PO}_4\cdot 12\text{H}_2\text{O}$	AP-104	Precipitated supernate from evaporator boil-down study.	From 7S110-DBB-03-013. Use of polarized light microscopy (PLM), SEM-EDS, and XRD was used to confirm crystal type. A major constituent.
Sodium nitrate	NaNO_3	AP-105	Boil-down of supernatant liquid, resulting in precipitation of solids.	From 7S110-WSC-07-143. Determined from XRD, SEM-EDS, and PLM. Approximately 49 wt.% abundance.
Sodium fluoride phosphate hydrate	$\text{Na}_7\text{F}(\text{PO}_4)_2\cdot 19\text{H}_2\text{O}$	AP-105	Boil-down of supernatant liquid, resulting in precipitation of solids.	From 7S110-WSC-07-143. Determined from XRD, SEM-EDS, and PLM. Approximately 49 wt.% abundance.

Table 3-1. The characteristics of Hanford tank boil-down laboratory studies. (4 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium fluoride sulfate	Na_3FSO_4	AP-105	Evaporated boil-down of the supernate resulting in solids precipitation.	From 7S110-GAC-06-064. PLM morphology and SEM-EDS used to identify crystal type. Treated liquid sample is the boil-down liquid supernate. This study was to help identify the unexpected solids formation a year after the boil-down of tank AP-108 (63 inches of new solids).
Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	AP-105	Boil-down of supernatant liquid, resulting in precipitation of solids.	From 7S110-WSC-07-143. Determined as a trace constituent from SEM-EDS, and PLM. Approximately 2 wt.% abundance.
Sodium carbonate hydrate	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	AP-105	Boil-down of supernatant liquid, resulting in precipitation of solids.	From 7S110-WSC-07-143. Determined as a trace constituent from SEM-EDS, and PLM. Approximately 8 wt.% abundance.
Sodium fluoride phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2\cdot 19\text{H}_2\text{O}$	AP-107	Evaporated supernatant resulting in solids precipitation (i.e., boil-down study).	From 7S110-GAC-06-064. Work supported evaporator campaign. PLM (for morphology) and SEM-EDS used to identify crystal composition. Waste template inventories of phosphate and fluoride showed only dilute concentrations of both prior to boil-down of waste. Post boil-down crystals were needle like. The needle crystals were cemented together by sodium carbonate solid.
Thenardite, Sodium Carbonate monohydrate	$\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$	AP-108	Supernate boil-down. A salt cake that precipitated when evaporator concentrate was put into tank. Taken by grab sample.	From 7S110-GAC-06-058. Identified by both XRD and Optical Microscopy (PLM). Comprised >90 wt.% of solids.

Table 3-1. The characteristics of Hanford tank boil-down laboratory studies. (4 pages)

Mineral	Chemical Formula	Tank	Waste Type	Comments
Sodium Fluoride Phosphate	$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$	AP-108	Supernate boil-down. A salt cake precipitated when evaporator concentrate was put into tank.	From 7S110-GAC-06-058. Trace amounts seen by Optical Microscopy, not confirmed by XRD because the quantity was too small. Comprised <10 wt.% solids.
Sodium Oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	AP-108	Supernate boil-down. A salt cake precipitated when evaporator concentrate was put into tank.	From 7S110-GAC-06-058. Very trace amounts by Optical Microscopy, too trace to observe by XRD. Comprised much less than 1 wt.% of solids.
Kogarkoite, sodium fluoride Sulfate	Na_3FSO_4	AP-108	Supernate boil-down. A salt cake precipitated when evaporator concentrate was put into tank.	From 7S110-GAC-06-058. Identified By XRD. Some crystals that could be this phase were observed by Optical Microscopy.

4.0 SEM-EDS Identification in Hanford Tank Samples

Elemental associations are typically identified by SEM-EDS observation where stoichiometric identification of specific solid crystal phases is not possible. This was included because there are often trace amounts of a phase that cannot be conclusively identified, but the elements in the mineral can be identified. Note that SEM cannot see hydrogen and because oxygen is ubiquitous (e.g., an impurity), many of the minerals that contain oxygen and/or hydrogen can be hydroxides or oxides because SEM cannot distinguish the difference.

Table 4-1. The elemental composition of various Hanford tank waste samples. (5 pages)

Elements in Solid	Tank	Waste Type	Comments
Fe-O	AN-107	A2 saltcake	From RPP-20018. SEM-EDS, PLM, EDS analysis. Saltcake solids analysis.
Na-Al-Si	AP-105	Evaporated supernatant resulting in solids precipitation.	From 7S110-GAC-06-064. PLM tentatively identified crystal as sodium aluminum sulfate; however SEM-EDS used to identify crystal composition as some Al-Si containing material.
Al-Si-O	AW-105	CWZr2 sludge solids	From PNNL-13394. Identified by TEM, SEM-EDS and XRD.
Zr-Fe-O	AZ-101	P3AZ1 washed saltcake solids.	From PNNL report WTP-RPT-076 Rev.0. TEM and EDS used to identify solid phase.
Na-Al-Si-O	BY-109	Solids remaining after CWP2 saltcake dissolution in water. Likely zeolite.	From 7S110-DLH-06-073. Both PLM (for morphology) and SEM-EDS were used to identify crystal type. Sodium is found primarily in sodium oxalate and sodium aluminosilicate.
Al-Si-O	B-111	2C sludge solids.	From PNNL-13394. Identified by SEM-EDS, TEM, and XRD.
Ag-O	C-103	CWP1 sludge (only heel left over after retrieval)	Ag-O was identified as a phase rich in silver and oxygen with no other elements by SEM in the heel left over after the sludge in C-103 was sluiced into AN-106. Thus, there is likely Ag-O in the sludge in AN-106 as well. This mineral was identified in report PNNL-16738. Because there was too little of this mineral to be observed by SEM, the exact stoichiometry is speculative, and this mineral could easily be Ag(OH) rather than Ag ₂ O, because SEM cannot distinguish between them. This tank has been sluiced into tank AN-106, so this phase likely also exists in An-106.

Table 4-1. The elemental composition of various Hanford tank waste samples. (5 pages)

Elements in Solid	Tank	Waste Type	Comments
(Ag, Hg)-O	C-103	CWP1 sludge (only heel left over after retrieval)	Ag-Hg-O solid solution was identified as a phase rich in silver, mercury, and oxygen with no other elements by SEM in the heel left over after the sludge in C-103 was sluiced into AN-106. Thus, there is likely Hg-Ag-O in the sludge in AN-106 as well. This mineral was identified in report PNNL-16738. Because there was too little of this mineral to be observed by XRD, the exact stoichiometry is speculative, and this mineral could easily be $\text{Ag}(\text{OH})_2\text{-Hg}(\text{OH})_2$ rather than Ag-O, because SEM cannot distinguish between them. The waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106. The Hg-Ag-O phase was also observed in the bulk CWP-1 Sludge in C-103 prior to retrieval in report 7S110-DLH-04-015.
Zr-O	C-103	CWP1 sludge (only heel left over after retrieval)	Rarely observed by SEM in PNNL-16738. The bulk of the waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106.
Th-O	C-103	CWP1 sludge (only heel left over after retrieval)	Rarely observed by SEM in PNNL-16738. The bulk of the waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106.
Fe-Pb-O	C-103	CWP1 sludge (only heel left over after retrieval)	Rarely observed by SEM in PNNL-16738. The bulk of the waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106.
Na-Ca-U-O	C-103	CWP1 sludge (only heel left over after retrieval)	Rarely observed by SEM in PNNL-16738. The bulk of the waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106. The stoichiometry identified by SEM is very similar to the known mineral Andersonite $[\text{Na}_2\text{Ca}(\text{UO}_2)(\text{CO}_3)_3 \cdot 6\text{H}_2\text{O}]$. This phase grew on Gibbsite particles.
Ca-P-O	C-103	CWP1 sludge (only heel left over after retrieval)	Occasionally observed by SEM in PNNL-16738. This phase likely also occurs in the sludge transferred from C-103 to AN-106. This is likely a calcium phosphate because no other elements are incorporated into it. However, there are many calcium phosphates, so we cannot conclusively identify them. An email from Ken Krupka indicated that this phase also has 2-4 Wt% fluoride that was not documented in the report. A reasonable hypothesis for this phase is hydroxyapatite with fluoride substituted for some of the hydroxyls. This is reasonable because hydroxyapatite and fluoroapatite are known to form a solid solution series (Lindsay and Others, 1989). This tank has been sluiced into tank AN-106, so this phase likely also exists in An-106.

Table 4-1. The elemental composition of various Hanford tank waste samples. (5 pages)

Elements in Solid	Tank	Waste Type	Comments
Na-Al-Si-O	C-103	CWP1 sludge	Identified by SEM in 7S110-DLH-04-015. The bulk of the waste from this tank has been sluiced into tank AN-106, so this phase likely also exists in AN-106.
(Ag, Hg)-O	C-103	CWP1 sludge (only heel left over after retrieval)	Ag-Hg-O solid solution was identified as a phase rich in silver, mercury, and oxygen with no other elements by SEM in the heel left over after the sludge in C-103 was sluiced into AN-106. Thus, there is likely Hg-Ag-O in the sludge in AN-106 as well. This mineral was identified in report 7S110-DLH-04-015 on the bulk sludge prior to retrieval, but has also been identified in PNNL-16738 in the heel that remains in C-103. Because there was too little of this mineral to be quantified by Energy Dispersive Spectroscopy (EDS) attached to the SEM, the exact stoichiometry is speculative, and this mineral could easily be $\text{Ag}(\text{OH})_2\text{-Hg}(\text{OH})_2$ rather than Ag-O, because SEM cannot distinguish between them.
Al-Si-O solids	C-107	Waste types: 1C (primary) and CW (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified by TEM and XRD. Sludge solids. Seen in both as-received and caustic leached (3 M) solids.
Na-Al-P-rich solid	C-107	1C, CWP2, SRR sludge composite waste type.	From 7S110-JMF-05-015. Caustic demand test as part of corrosion mitigation project. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. The 1C waste type, according to the BBI Template Composition report [RPP-8847], contains very high concentrations of Al (0.015 g-Al/g) and PO_4 (0.069 g-P/g). A major constituent (>10 wt.%).
Si-Al-Na-rich solid	C-107	1C, CWP2, SRR sludge composite waste type.	From 7S110-JMF-05-015. Caustic demand test as part of corrosion mitigation project. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. The 1C waste type, according to the BBI Template Composition report [RPP-8847], contains very high concentrations of Al (0.015 g-Al/g) and Si (0.0062 g-Si/g). A minor constituent (1-10 wt.%).
Na-Al-F-rich solid	C-107	1C, CWP2, SRR sludge composite waste type.	From 7S110-JMF-05-015. Caustic demand test as part of corrosion mitigation project. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. The 1C waste type, according to the BBI Template Composition report [RPP-8847], contains very high concentrations of Al (0.015 g-Al/g) and F (0.015 g-F/g). A minor constituent (1-10 wt.%).

Table 4-1. The elemental composition of various Hanford tank waste samples. (5 pages)

Elements in Solid	Tank	Waste Type	Comments
Na-Bi-Fe-P-rich sodium bismuth phosphate hydroxide	C-107	1C, CWP2, SRR sludge composite waste type.	From 7S110-JMF-05-015. Caustic demand test as part of corrosion mitigation project. The techniques of PLM and SEM-EDS were used to identify crystalline solid phases. The 1C waste type, according to the BBI database, contains very high concentrations of Bi (0.019 g-Bi/g), Fe (0.010 g-Fe/g) and PO ₄ (0.069 g-P/g). A major constituent (>10 wt.%).
Al-Si-O	S-107	Waste types: R (primary) and EB (secondary).	From PNNL-13394. Study undertaken to characterize solid phases to understand better the enhanced sludge washing (ESW) and leaching of Hanford material, to support waste treatment. Identified only through TEM. Sludge solids. Seen in both direct-sampled and caustic leached (3M) materials. From BBI database, the quantities of aluminum and silicon are 0.045 g-Al/g-solid and 167 ug-Si/g-solid. As a minor identified component, this is consistent with tank composition.
Ca-Cr-O	S-112	Heel after retrieval of REDOX Process Cladding Waste (CWR) and S Saltcake	Identified by SEM in S-112 Heel samples in PNNL-17593. FH-0202771 simulated the dissolution of the S Saltcake in S-112 and identified minerals in the residuals that did not dissolve. They found Ca-Al-Cr-O bearing particles, which they thought might be a solid solution of calcium aluminate and calcium chromite. They (FH-2002771) also identified Ca ₃ Al ₂ (OH) ₁₂ by XRD. It is likely that the particles observed in FH-0202771 are agglomerates of calcium aluminate and calcium chromite. The Ca-Cr-O phase identified in PNNL-17593 is likely calcium chromate that had not agglomerated with calcium aluminate in the real tank.
Pb-Cl-(maybe oxygen as well)	S-112	Heel after retrieval of REDOX Process Cladding Waste (CWR) and S Saltcake	Rare particles identified in PNNL-17593 by SEM. They could not tell if the oxygen atoms came from the same particle as the Pb-Cl particles or were from other nearby particles.
Fe-O (maybe Mn)	S-112	Heel after retrieval of REDOX Process Cladding Waste (CWR) and S Saltcake	Rare particles identified in PNNL-17593 by SEM. There was some Mn associated with the Fe particles, but it was not clear if the Mn was incorporated into the crystal structure or in the form of even smaller particles stuck to the Fe surface.

Table 4-1. The elemental composition of various Hanford tank waste samples. (5 pages)

Elements in Solid	Tank	Waste Type	Comments
Na-Al-O, a silicate	S-112	R1 sludge	From PNNL-17593. Identified by SEM, so cannot see light elements (such as H) that may be present. Thermodynamic calculations reported in PNNL-17593 indicated that the solution was near saturation with dawsonite, so the Na-Al-O phase may be dawsonite. The SEM analysis indicated that there was more Na in this solid than is typical of dawsonite.
Al-Si-O	SX-108	Sludge solids.	From PNNL-13394. Identified only through TEM.
Al-O	SY-102	Solids waste type, although not specified, is likely NA sludge centrifuged REDOX solids.	From HNF-7437. Study performed analysis of solids using ICP technique. Major constituent mineral type is consistent with waste type as aluminum concentration is 0.036 g-Al/g-solids from TWINS inventory database.
Cr-O	SY-102	Solids waste type, although not specified, is likely NA sludge centrifuged REDOX solids.	From HNF-7437. Study performed analysis of solids using ICP technique. Major constituent mineral type is consistent with waste type as chromium concentration is 0.024 g-Cr/g-solids from TWINS inventory database.
Fe-O	SY-102	Solids waste type, although not specified, is likely NA sludge centrifuged REDOX solids.	From HNF-7437. Study performed analysis of solids using ICP technique. Major constituent mineral type is consistent with waste type as iron concentration is 0.004 g-Fe/g-solids from TWINS inventory database.

5.0 REFERENCES

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