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

Project Hanford Management Contractor for the
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Identification of Solid Phases in Saltcake from Hanford Site Waste Tanks

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TERMS

EDS	energy dispersive spectrometer
ICDD	International Centre for Diffraction Data
keV	kilo-electron volts
Kv	kilovolts
Ma	milliamperes
n	refractive index
n_{xtal}	refractive index of a crystal
n_{liq}	refractive index of liquid surrounding a crystal
PLM	polarized light microscopy
SEM	scanning electron microscope
XRD	x-ray diffraction

1.0 INTRODUCTION AND SUMMARY

One of the primary tasks in the clean up of the Hanford Reservation is the retrieval of waste from single-shell and double-shell storage tanks. The waste will be used as feedstock for the vitrification plant under construction in the Hanford 200 East area. To accomplish this tank waste retrieval, the River Protection Project requires physical and chemical data on the nature of the wastes.

For several years, laboratory tests have been conducted at the 222-S Laboratory on the dissolution characteristics of Hanford saltcake waste from single shell tanks (Herting 1998, 1999, 2000, 2001). The bulk physical and chemical data determined during these studies has provided valuable input for evaluating the Environmental Simulation Program produced by OLI Systems, Inc. of Morris Plains, New Jersey. This computer program is used by the River Protection Project to predict chemical solubility during dilution and retrieval of tank wastes.

The characterization of solid phases found in saltcake undergoing dissolution and evaporative concentration is an important adjunct to the bulk physical and chemical properties of the saltcake waste. The phase analysis of Tank BY-109[1] saltcake, described in this report, was conducted using polarized light microscopy (PLM), X-Ray diffraction (XRD) and scanning electron microscopy (SEM).

The phases $Na_7F(PO_4)_2 \cdot 19H_2O$, Na_3FSO_4, $Na_2C_2O_4$ and NaF were identified in BY-109 saltcake by all three instruments, while Na_3AlF_6 was observed in XRD and SEM analyses. In addition, the SEM found significant amounts of a fine-grained aluminum-rich phase, frequently associated with lesser amounts of chromium and sodium. PLM noted the presence of a fine-grained agglomerated phase that was probably this aluminum-rich phase, and XRD confirmed the presence of an aluminum hydroxide, bayerite, in one sample. Finally, the SEM located a number of discrete particles of an aluminosilicate phase and a uranium-rich phase, as well as occasional particles of exotic composition, such as chromium-rich, calcium/strontium-rich and bismuth/palladium-rich. These phases were too rare, or too poorly crystalline, to be identified by PLM or XRD analysis.

While the emphasis of this report is on phase analysis of Tank BY-109 saltcake, examples of phases identified in other tanks are included where appropriate. The hope of the authors is that this document will serve as a visual reference for future phase analysis efforts, both at the 222-S Laboratory and at other Department of Energy sites where similar tank waste samples are characterized. At the same time, the reader is cautioned that this is a work in progress, that is, the methods employed here are still being developed and refined.

---

[1] Hanford Site waste tanks are identified with 8 character tank names that begin with "241-" (e.g., 241-BY-109). For convenience, the tank names in this document have been abbreviated to 5 characters (e.g., BY-109), which identify the tank farm and specific tank.

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2.0 EXPERIMENTAL METHODS

Solid phase analysis was conducted on a composite saltcake sample from Tank BY-109. The untreated composite sample (designated "BY9Orig") contained many mixed phases, making identification of individual phases difficult. An attempt was made to isolate individual phases by dissolving the saltcake through a stepwise process of adding water, mixing to dissolve solids, centrifuging to separate solid and liquid phases, decanting the liquid, and adding fresh water to the undissolved solids. This process has become known as "cascade dissolution."

After each water addition, a sample of the undissolved solids was taken for phase analysis (samples BY9Dil1 – BY9Dil6 and BY9Resi). All of the liquid fractions were combined into a single sample, which was then evaporated slowly while collecting individual crops of crystals as they formed (samples BY9Evap2 – BY9Evap6).

Critics will be quick to point out that the crystalline solids that form during evaporation of the solution are not necessarily the solid phases that were present in the original saltcake. However, in most cases, once the individual phases have been isolated and characterized, it is possible to recognize their presence in the original mixed-phase sample (BY9Orig).

2.1 SAMPLE COMPOSITE AND BREAKDOWN

The composite sample for Tank BY-109 was prepared in a hotcell and contained 225.9 g of saltcake from 9 different core segments (Herting 2001). The chemical composition of this composite is presented in Table 2-1. A portion of this composite was prepared for solids analysis by following the procedure whose steps are summarized below.

Approximately 2 g of the Tank BY-109 composite was placed in a sample vial labeled "BY9Orig." This sample was reserved for PLM, SEM, and XRD analysis. Another aliquot of sample of the Tank BY-109 composite (43.1 g) was transferred to a 50 mL centrifuge cone for dissolution testing.

The 43.1 g of the Tank BY-109 composite saltcake was diluted with 20.9 g of deionized water, representing a dilution of 48 percent by weight. The contents of the centrifuge cone were mixed for at least one hour at 21 °C. About 10 percent of the total volume (approximately 4.5 mL) of suspended slurry was transferred to a sample vial labeled "BY9Dil1." This sample was reserved for PLM, SEM, and XRD analysis (hereafter referred to as phase analysis) and the rest of the sample was centrifuged to separate the solids. The centrifuged liquid was decanted into a collection jar and the remaining solids were weighed. This process was repeated five more times with the centrifuged liquid added to the same collection jar. This resulted in the generation of six slurry samples (BY9Dil1 – BY9Dil6) for phase analysis. The residual solids after these six washings were mixed with 10 mL water to allow the solids to be transferred to a vial labeled "BY9Resi" for phase analysis.

Table 2-1. Composition of Tank BY-109 Saltcake Composite Sample.

Chemicals (wt%)		**Radionuclides (μCi/g)**	
Al	1.97	^{60}Co	< 0.042
Cr	0.27	$^{89/90}Sr$	19.1
Fe	< 0.10	^{99}Tc	0.080
K	0.23	^{125}Sb	< 0.44
Na	24.9	^{137}Cs	74.5
P	0.87	^{154}Eu	< 0.13
S	4.25	^{155}Eu	< 0.26
Si	< 0.10	^{241}Am	< 0.10
F^-	5.32	Total Alpha	0.081
Cl^-	0.19		
NO_2^-	2.80		
NO_3^-	6.94		
PO_4^{3-}	2.57		
SO_4^{2-}	11.8		
$C_2O_4^{2-}$	1.96		
CO_3^{2-}	2.34		
"acetate"[1]	0.69		
OH^-	1.54		
H_2O	33.2		

[1] "Acetate" is the non-oxalate total organic carbon, and is being used as a stand-in for all of the small organic acids, chelating agents, and organic degradation products likely to be present.

The combined centrifuged liquid from the collection jar was examined in order to determine if the mixing of early centrifuged liquids (high ionic strength) with later centrifuged liquids (low ionic strength) had resulted in the precipitation of crystals. No precipitate was observed in the collection jar. Therefore, the sample identification "BY9Evap1" reserved for this sample was not used. The entire contents of the collection jar (approximately 195 mL) were transferred to a 250 mL beaker and a Teflon coated stir bar was added. The combined, centrifuged liquid was evaporated slowly while crystals precipitated from the concentrating solution. When the volume of precipitate reached approximately 20 percent of the total volume, the solids were collected by allowing them to settle or by filtration, then evaporation of the liquid was allowed to continue. Samples of precipitated crystals were transferred to vials and identified as BY9Evap2 through BY9Evap6. In summary, the following Tank BY-109 sample fractions were prepared:

- BY9Orig – original saltcake solids
- BY9Dil1 – slurry with solids undissolved after 48 percent by weight dilution with water
- BY9Dil2 – slurry with solids undissolved after 106 percent by weight dilution with water
- BY9Dil3 – slurry with solids undissolved after 173 percent by weight dilution with water
- BY9Dil4 – slurry with solids undissolved after 254 percent by weight dilution with water
- BY9Dil5 – slurry with solids undissolved after 345 percent by weight dilution with water
- BY9Dil6 – slurry with solids undissolved after 440 percent by weight dilution with water
- BY9Resi – residual undissolved solids suspended in water
- BY9Evap1 – solids precipitated in receiver jar before evaporation [none formed]
- BY9Evap2 – first crop of crystals formed on evaporation of collected dissolved liquid
- BY9Evap3 – second crop of crystals formed on evaporation
- BY9Evap4 – third crop of crystals formed on evaporation
- BY9Evap5 – fourth crop of crystals formed on evaporation
- BY9Evap6 – fifth crop of crystals formed on evaporation.

2.2 SOLID PHASE STANDARDS

Solid phase standards were prepared by summer intern Kristi Brault to aid in the interpretation of PLM, SEM, and XRD observations. Each standard contains one solid phase in a stock solution containing 1M NaOH, 2M $NaNO_2$, and 2M $NaNO_3$, where feasible. This liquid matrix was intended to reproduce the high-ionic strength liquid in contact with the actual saltcake samples, thereby duplicating the effects that the solution chemistry might have on crystal habit or hydration state. Brief descriptions of the standard preparation methods are as follows.

Standard $Na_3PO_4 \cdot 12H_2O$: The stock solution was saturated at room temperature with excess reagent grade $Na_3PO_4 \cdot 12H_2O$ (1.52 g in 20 mL stock solution). The slurry was heated to dissolve the solids and then cooled to room temperature while stirring. PLM analysis (see below) confirmed that the correct hydrate had formed.

Standard $Na_7F(PO_4)_2 \cdot 19H_2O$: The stock solution (40 mL) was saturated at room temperature with excess reagent grade NaF (0.34 g) and $Na_3PO_4 \cdot 12H_2O$ (3.04 g) in a 1:2 molar ratio. The slurry was heated to dissolve the solids, then cooled again to ambient temperature while stirring.

Standard Na_3FSO_4: 25 mL of 0.8 M NaF solution and 25 mL of 0.8 M Na_2SO_4 solution were mixed with 40 mL of stock solution in a 150 mL beaker. The solution was then evaporated with gentle heating until the slurry volume reached 40 mL.

Standard NaF: 20 mL of 0.95 M NaF solution was mixed with 20 mL stock solution in a 50 mL beaker. The solution was then evaporated with gentle heating until the slurry volume reached 20 mL.

Standard Na_2SO_4 (anhydrous): 50 mL of 1.6 M Na_2SO_4 solution was mixed with 40 mL of stock solution in a 150 mL beaker. The solution was then evaporated with gentle heating until the slurry volume reached 40 mL.

Standard $Na_2C_2O_4$: 60 mL of 0.37 M $Na_2C_2O_4$ solution was mixed with 40 mL of stock solution in a 150 mL beaker. The solution was then evaporated with gentle heating until the slurry volume reached 40 mL.

Standard $Na_2CO_3 \cdot H_2O$: 100 mL of 1.0 M Na_2CO_3 solution was mixed with 40 mL of stock solution in a 150 mL beaker. The solution was then evaporated with gentle heating until the slurry volume reached 40 mL.

Standard $Al(OH)_3$ gibbsite: Technical grade sodium aluminate (14.2 g) was dissolved in 40 mL hot water and reagent grade $NaNO_3$ (6.8 g) and $NaNO_2$ (5.5 g) were added. The hot solution was filtered, and $Al(OH)_3$ precipitated slowly after the solution had cooled.

Standard AlO(OH) boehmite: Solid boehmite was added to the stock solution.

Standard $NaNO_3$: The stock solution was saturated at room temperature with excess reagent grade $NaNO_3$. The slurry was heated to dissolve the solids and then cooled again to ambient temperature while stirring.

Standard $NaNO_2$: The stock solution was saturated at room temperature with excess reagent grade $NaNO_2$. The slurry was heated to dissolve the solids and then cooled again to ambient temperature while stirring.

Standard $NaAl(OH)_4$: Technical grade sodium aluminate (28.0 g) was dissolved in 80 mL hot water and reagent grade NaOH (9.6 g), $NaNO_3$ (6.8 g) and $NaNO_2$ (5.5 g) were added. The hot solution was filtered and then evaporated with gentle heating to 40 mL.

Standard $Na_3NO_3SO_4 \cdot H_2O$: 36 g of $NaNO_3$ and 15 g of $Na_2SO_4 \cdot 10H_2O$ were dissolved in 100 mL water in a 150 mL beaker. The solution was evaporated under vacuum at a constant 35 °C or lower temperature until solids formed. Weights, volumes, and temperatures were chosen based on available literature data (Linke 1958).

Standard $Na_6CO_3(SO_4)_2$: 16 g of $Na_2CO_3 \cdot H_2O$ and 45 g of $Na_2SO_4 \cdot 10H_2O$ were dissolved in 90 mL of water in a 250 mL beaker. The solution was evaporated under vacuum at a constant 35 °C until solids formed. Weights, volumes, and temperatures were chosen based on available literature data (Linke 1958). This method was only partly successful. The standard was shown to contain a large amount of Na_2SO_4 and a small amount of $Na_6CO_3(SO_4)_2$.

2.3 SAMPLE PREPARATION FOR INSTRUMENTAL ANALYSIS

2.3.1 Polarized Light Microscopy

PLM analysis requires that the sample particulate is dispersed in a medium with a refractive index only moderately different from that of the particulate. This minimizes the contrast between particles and the surrounding medium. Most standards and samples were prepared for PLM analysis by suspending the particulate in the supernatant liquid using a plastic disposable pipette and removing a small drop of the slurry with the pipette. The pipette tip was touched to a

glass slide, allowing a portion of the drop to be transferred to the glass surface, and a cover slip was pressed onto the surface to disperse the particulate.

Where samples did not contain enough liquid for the particulate to be dispersed as a slurry for slide preparation (BY9Orig and BY9Resi), a liquid medium was added. Deionized water was added to BY9Resi, because the solids were the residue of exhaustive washing and should contain only water-insoluble particulate. Sample BY9Orig was suspended in some of the liquid generated during the first dissolution contact, under the assumption that this liquid was saturated with all of the phases present in BY9Orig, and should cause little additional dissolution of the original sample.

2.3.2 X-Ray Diffraction

Standards and samples were prepared for XRD analysis by vacuum filtration onto 25 mm diameter filter substrates. Millipore mixed cellulose ester filters were used for the preparation of the standard samples and Whatman cellulose filters were used for the Tank BY-109 samples. Several cellulose backing pads and one sample filter were placed on a fritted glass base on a filtration flask connected to trap and vacuum. A 10 mm diameter filtration funnel was placed on the filter stack and clamped into place. Up to 1 mL of suspended particulate was transferred with a plastic pipette to a small agate mortar. The sample suspension was wet ground to a paste in the mortar with an agate pestle.

For the standards, the paste was transferred to the funnel with a plastic pipette and additional supernatant liquid from the settled standard vial was added to cover the filter and evenly disperse the particulate. The vacuum pump was immediately turned on and the supernatant liquid was allowed to drain from the funnel. The earlier standards contained substantial contamination of $NaNO_3$ and $NaNO_2$ from the supernatant liquid. A step was added to the preparation to add 1-2 mL of deionized water on top of the supernatant liquid in the filtration funnel just before the last of the fluid was pulled through the filter. This step cleaned up the standard diffractograms substantially.

For the tank sample slurries the paste was transferred to the funnel with a plastic pipette. Up to 2 mL of supernatant liquid was added to cover the filter and disperse the particulate. The vacuum pump was immediately turned on and the fluid was allowed to begin draining from funnel and 1-2 mL of deinonized water was add to the top of the supernatant liquid just before the last of it was pulled through the filter to wash dissolved solids from the particulates. When the sample was evenly dispersed across the filter surface, about 0.5 mL of a 10 percent flexible collodion suspension in amyl acetate was added to the funnel with the vacuum on in order to glue the sample particulate to the filter surface.

The clamp and funnel were then removed from the standard or sample filters, and the filter was allowed to completely dry before stopping the vacuum pump. A particle loading of approximately 30 to 60 mg was targeted. The filter with ground sample particulate was transferred to a glass microscope slide on which double-sided transparent tape had been placed in the area to be exposed to the x-ray beam. The filter paper was then pressed onto the tape with a clean glass slide.

2.3.3 Scanning Electron Microscopy

Samples were prepared for SEM analysis by vacuum filtration onto polycarbonate filters. Several cellulose backing pads and a half section of a 37 mm diameter, 0.45 micron pore size, Nucleopore polycarbonate filter were placed on a fritted glass base on a filtration flask connected to trap and vacuum. A filtration funnel was placed on the filter stack and clamped into place.

Before suspending the sample particulate, about 0.5 mL of supernatant liquid was transferred with a plastic disposable pipette into the funnel. The sample was suspended with the pipette and a small amount (about 0.1 mL) of sample slurry was transferred into the funnel. The vacuum pump was immediately turned on. When the fluid had drained from filter surface, a small amount of distilled water was added (standards only), with the pump still on, and allowed to quickly drain. The clamp and funnel were then removed and the filter was allowed to completely dry.

An aluminum SEM stub was covered with an adhesive carbon disc and the carbon disc was pressed onto the surface of the filter with the vacuum still on. The particulate that was transferred to the sample stub was lightly pressed into the carbon disc with a glass slide, and the resulting sample was coated with a conductive coating of carbon by evaporative deposition, using a Cressington carbon coater.

2.4 INSTRUMENTATION

The instruments utilized in this study are housed in the 222-S Laboratory in the 200-West area of the Hanford Reservation. The laboratory is operated by the Fluor Hanford Company as Project Hanford Management Contractor. The instruments are described below.

2.4.1 Polarized Light Microscopy

PLM analysis was performed on a Leitz Laborlux[2] 12 polarized light microscope equipped with a Colorview 12 digital color camera.

2.4.2 X-Ray Diffraction

XRD data were collected using a Rigaku[3] MiniFlex powder diffractometer equipped with $\theta/2\theta$ scanning goniometer and continuously variable slit. The x-ray tube generates copper x-rays at 30 Kv and 15 ma.

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[2] Leitz Laborlux is a registered trademark of Ernst Leitz Wetzlar GmbH, Wetzlar, Germany.

[3] Rigaku MiniFlex is a registered trademark of Rigaku/MSC, The Woodlands, Texas.

2.4.3 Scanning Electron Microscopy

SEM analysis was conducted using an Aspex[4] Personal Scanning Electron Microscope, Model II, with a Noran light element energy dispersive spectrometer (EDS) for chemical analysis. The instrument was operated at an accelerating voltage of 15 Kv, and the samples were mounted at a working distance of 16 mm (+/-0.5 mm). EDS spectra were acquired for 60 seconds (live time) on relatively smooth surfaces (either flat or sloping toward the detector) near the center of the image.

[4] Aspex is a registered trademark of ASPEX Instruments, Delmont, Pennsylvania.

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3.0 RESULTS BY PHASE

Each analytical instrument used in this study (PLM, SEM, and XRD) provides a different type of information about solid particulate. PLM provides information on the optical characteristics (e.g., birefringence) and particle morphology (e.g., shape, size, crystal habit). The SEM also provides morphology information as well as chemical (elemental) composition with the EDS. The crystallographic phase and crystallite size are determined with XRD analysis. When used in concert, these three tools represent a powerful means of describing the solid phase components of a sample.

The following discussion describes certain distinguishing properties for the phases that were synthesized as standards. A brief description of the occurrence of these phases in the Tank BY-109 samples is included after the discussion for each standard phase. Finally, examples are given of cases in which the phase has been identified in other waste tank samples.

Most of the optical data referenced in this report is taken from *The Microscopical Characters of Artificial Inorganic Solid Substances: Optical Properties of Artificial Minerals* (Winchell and Winchell 1964).

3.1 $Na_7F(PO_4)_2 \cdot 19H_2O$

Name: Sodium fluoride phosphate hydrate	**ICDD PDF#:** 30-1189
Chemical Formula: $Na_7F(PO_4)_2 \cdot 19H_2O$	**Crystal System:** Cubic
Refractive Index: n = 1.45	**Mineral Name:** Natrophosphate

Sodium fluoride phosphate hydrate was first identified as a component in Hanford Site tank waste in approximately 1992. Since that time, it has been observed in many tank waste samples, and has proved to be one of the more common sodium salts present in the waste. It is a congruent double salt, i.e., the Na:F:PO_4 mole ratios in the liquid phase in equilibrium with the pure salt are the same as in the solid phase.

The double salt is significantly less soluble than either single salt (sodium fluoride or sodium phosphate). Therefore, the presence of a small amount of fluoride can have a large impact on the properties of a slurry containing phosphate. In the absence of fluoride, the phosphate solids have a needle habit (see Figure 4.4.1-1), and the slurry that contains them has a very high viscosity. In the presence of fluoride the double salt forms and the viscosity of the slurry is greatly reduced because of the more compact crystal habit (Figure 3.1.1-1). Both salts are shown together in Figure 3.1.3-3.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages. The x-ray diffractogram for the standard (Figure 3.1.1-7) contains a major contribution from the contaminants $NaNO_3$ and $NaNO_2$ precipitated from the supernatant liquid during sample preparation.

3.1.1 $Na_7F(PO_4)_2 \cdot 19H_2O$ Standard

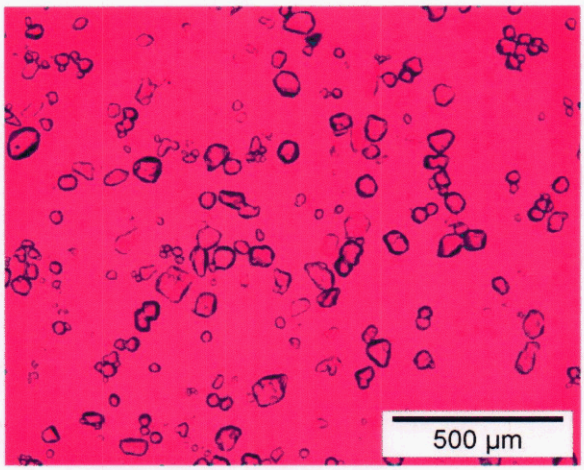

Figure 3.1.1-1. $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

Very recognizable and typical habit for sodium fluoride phosphate double salt in tank waste; isotropic octahedral and cuboctahedral crystals, mostly imperfect.

Crossed polars with Red I compensator, 4x objective.

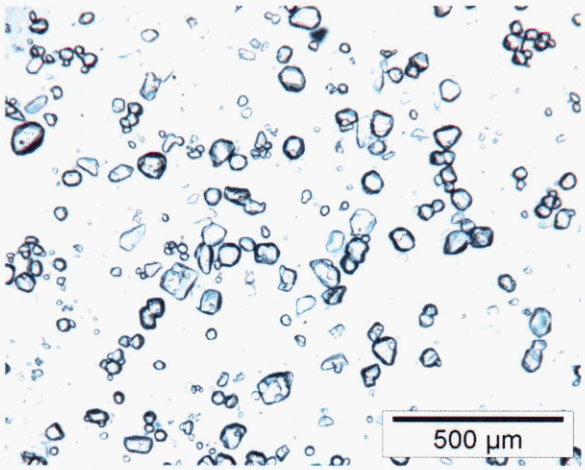

Figure 3.1.1-2. $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

Same view as Figure 3.1.1-1; Becke line goes in as stage is moved away from objective ($n_{xtal} > n_{liq}$), opposite of the other common isotropic crystals in tank waste, NaF (though NaF crystals are far less common than $Na_7F(PO_4)_2 \cdot 19H_2O$ crystals).

Uncrossed polars, 4x objective.

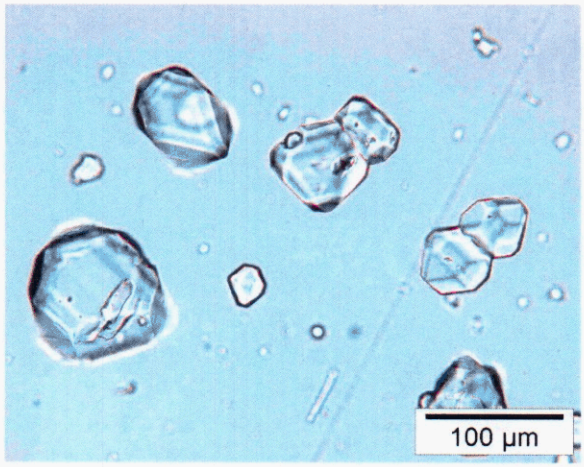

Figure 3.1.1-3. $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

Crystals at higher magnification.

Uncrossed polars, 16x objective.

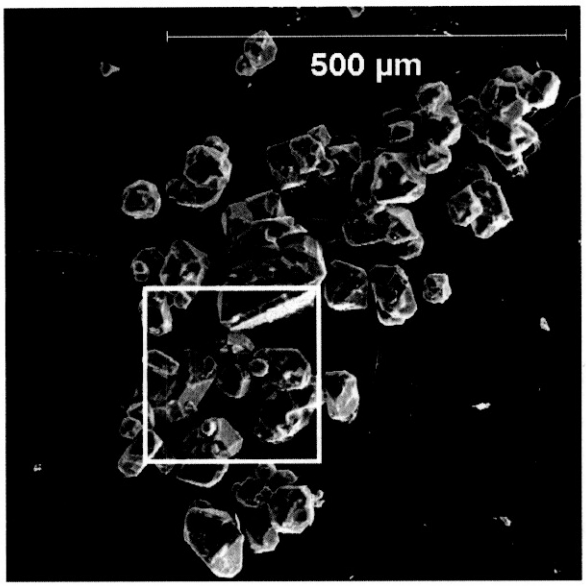

Figure 3.1.1-4. Low magnification secondary electron image of $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

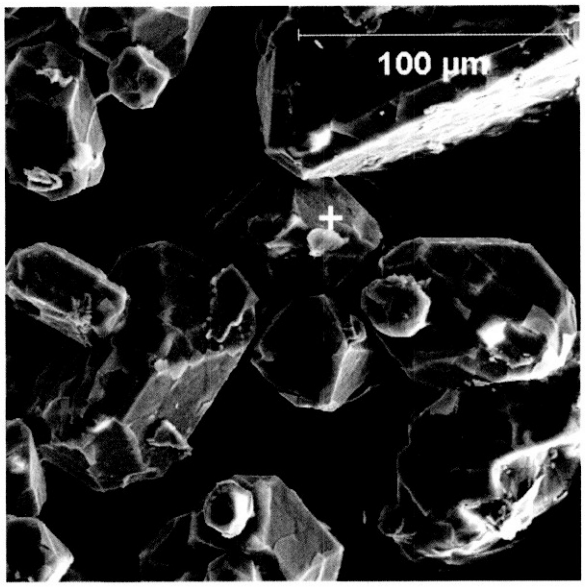

Figure 3.1.1-5. High magnification secondary electron image of $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

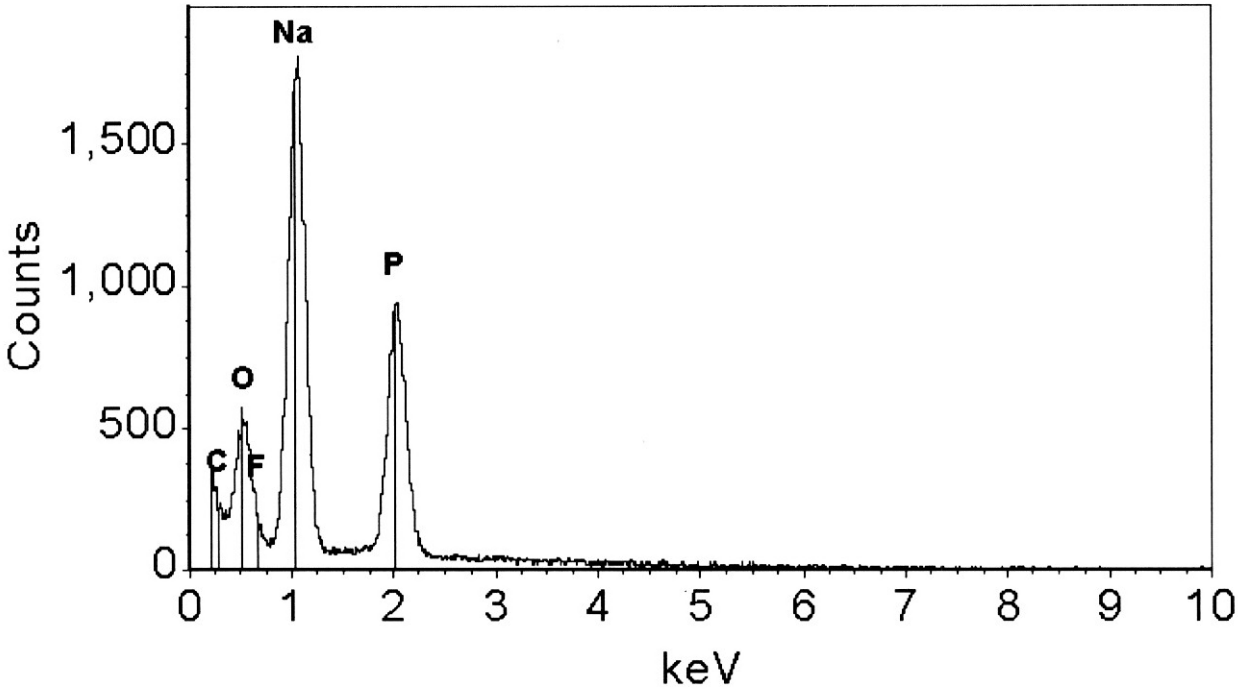

Figure 3.1.1-6. EDS spectrum from spot marked with + in Figure 3.1.1-5, 15Kv, 30% spot.

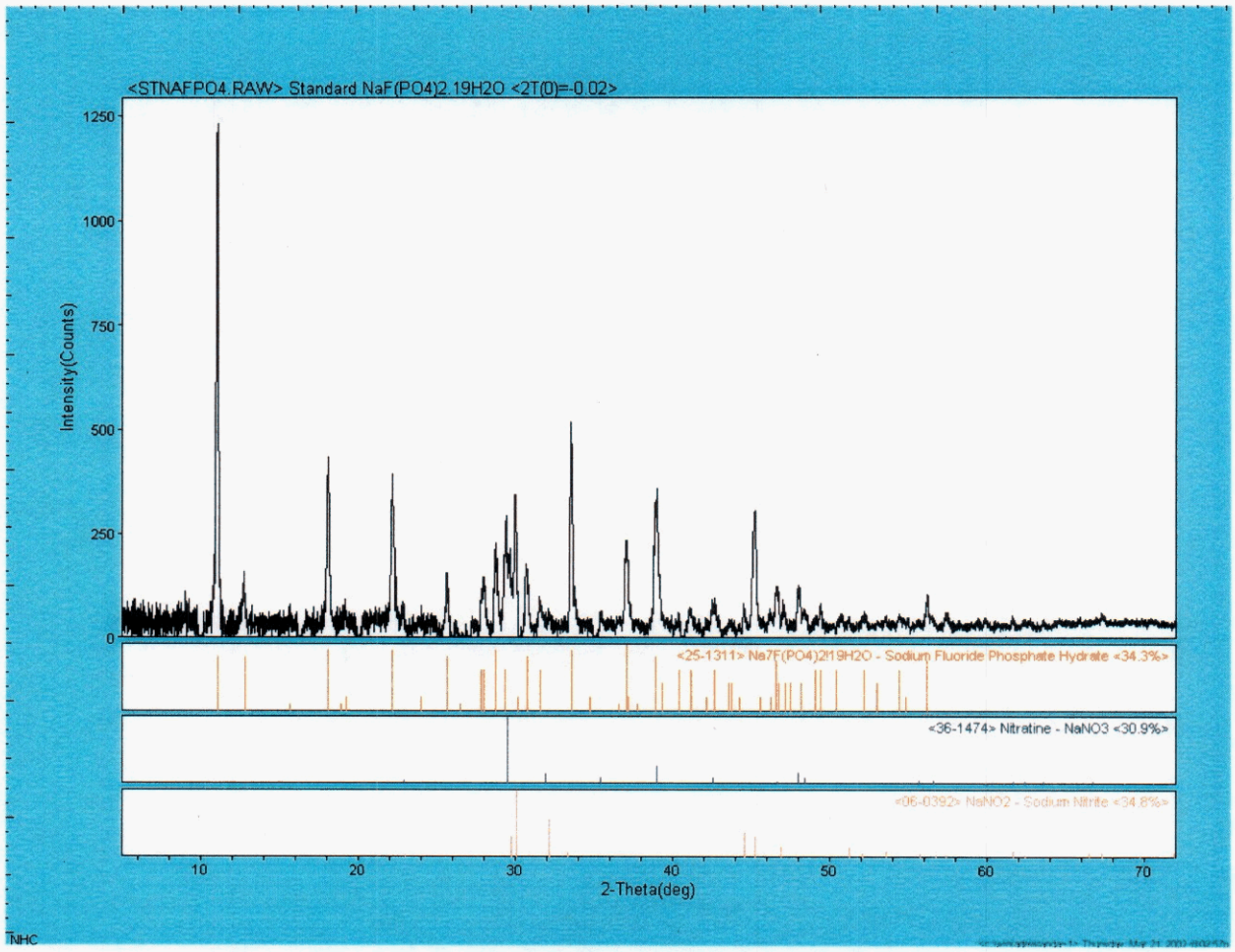

Figure 3.1.1-7. XRD spectrum of $Na_7F(PO_4)_2 \cdot 19H_2O$ standard.

3.1.2 Occurrence of $Na_7F(PO_4)_2 \cdot 19H_2O$ in Tank BY-109 Saltcake

Sodium fluoride phosphate hydrate was identified by all three methods in the original saltcake sample and in BY9Evap2, BY9Evap4, and BY9Evap6. PLM and XRD found this phase in BY9Dil1 and BY9Evap5.

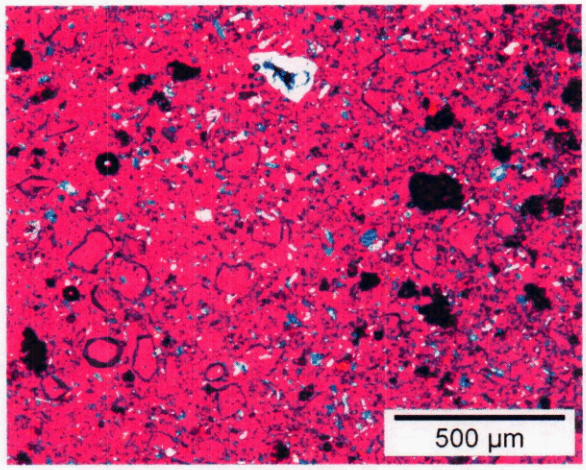

Figure 3.1.2-1. Sample BY9Orig.

Isotropic crystals of $Na_7F(PO_4)_2 \cdot 19H_2O$ dominate this sample of original (untreated) Tank BY-109 saltcake

Crossed polars with Red I compensator, 4x objective.

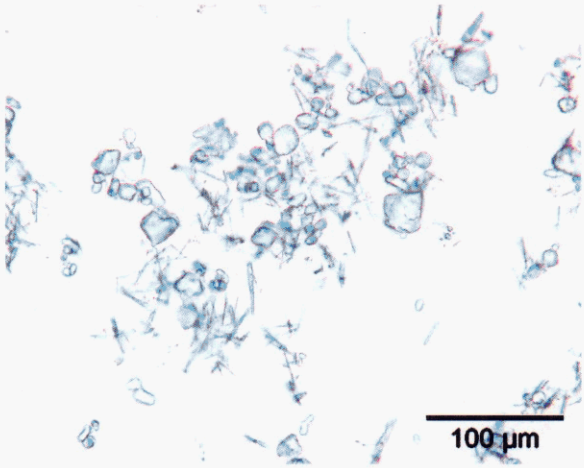

Figure 3.1.2-2. Sample BY9Evap2.

Isotropic $Na_7F(PO_4)_2 \cdot 19H_2O$ octahedra and anisotropic $Na_2C_2O_4$ needles are the first crystals to form when the dissolved Tank BY-109 saltcake is re-evaporated.

Uncrossed polars, 16x objective; same field of view as Figure 3.1.2-3.

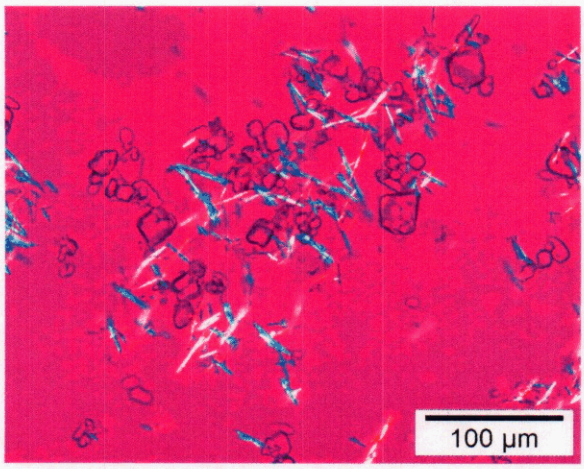

Figure 3.1.2-3. Sample BY9Evap2.

Same field of view as Figure 3.1.2-2.

Crossed polars with Red I compensator, 16x objective.

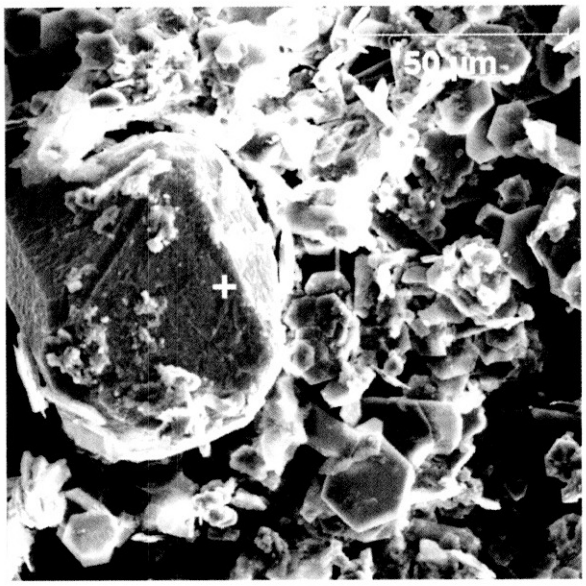

Figure 3.1.2-4. High magnification secondary electron image of BY9Orig sample.

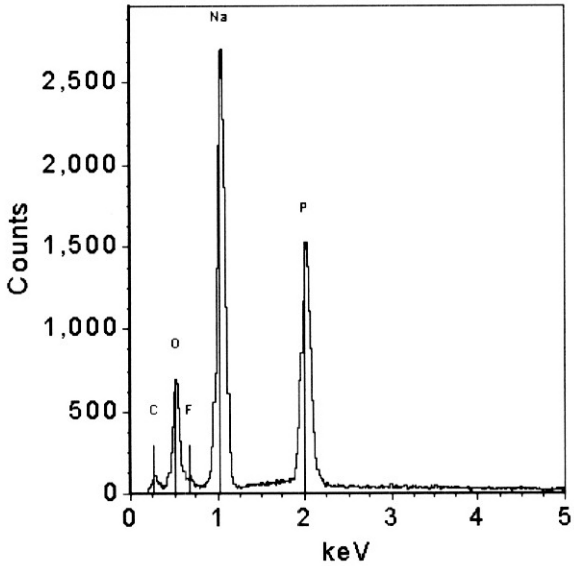

Figure 3.1.2-5. EDS spectrum from spot marked with + in Figure 3.1.2-4, 15Kv, 30% spot.

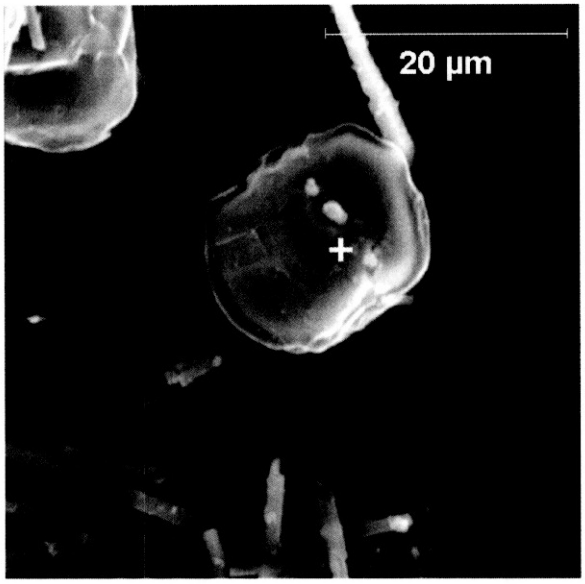

Figure 3.1.2-6. High magnification secondary electron image of BY9Evap2 sample.

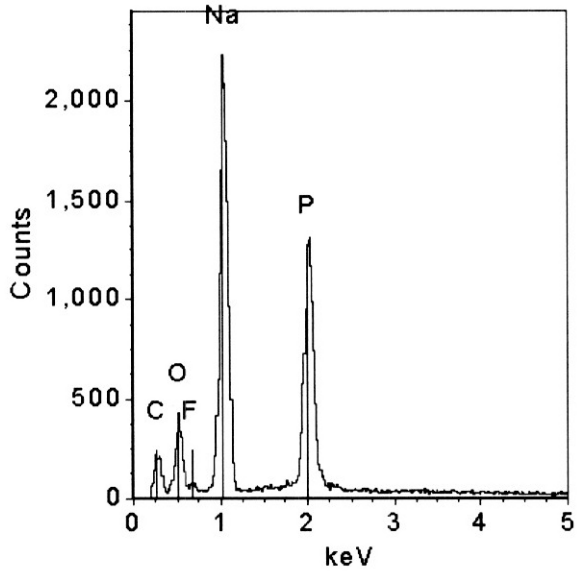

Figure 3.1.2-7. EDS spectrum from spot marked with + in Figure 3.1.2-6, 15Kv, 30% spot.

3.1.3 Other Occurrences of $Na_7F(PO_4)_2 \cdot 19H_2O$

Before 1992, this phase was not known to be present in Hanford Site tank waste. Over the ensuing years, it has proved to be almost ubiquitous, as tanks that contain the double salt are more numerous than those that contain sodium phosphate single salts.

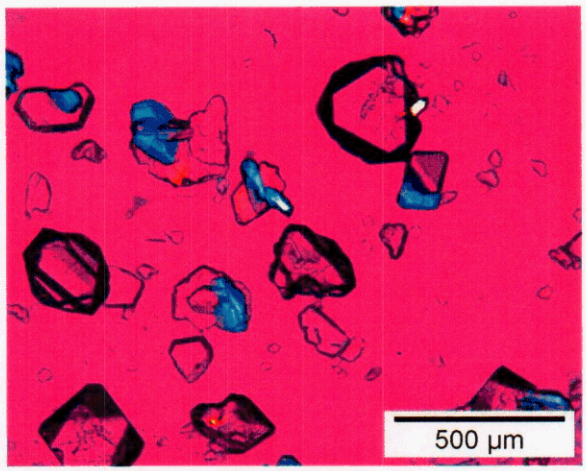

Figure 3.1.3-1. Mixture of $Na_7F(PO_4)_2 \cdot 19H_2O$ (high-contrast isotropic crystals) and Na_3FSO_4 (low-contrast blue/orange anisotropic plates), formed by mixing equal amounts of dissolved Tank BY-109 saltcake and dissolved Tank U-107 saltcake.

Crossed polars with Red I compensator, 4x objective.

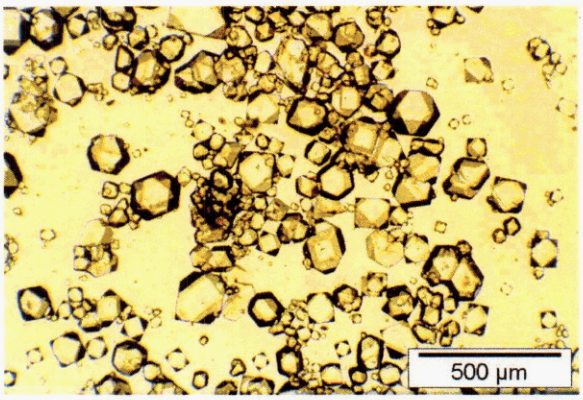

Figure 3.1.3-2. $Na_7F(PO_4)_2 \cdot 19H_2O$ from Tank BY-102.

Several very good examples of near-perfect cuboctahedral crystals, which appear in two dimensions as a light square inscribed within a dark square, with the squares at a 45 degree angle to one another.

Uncrossed polars, 4x objective.

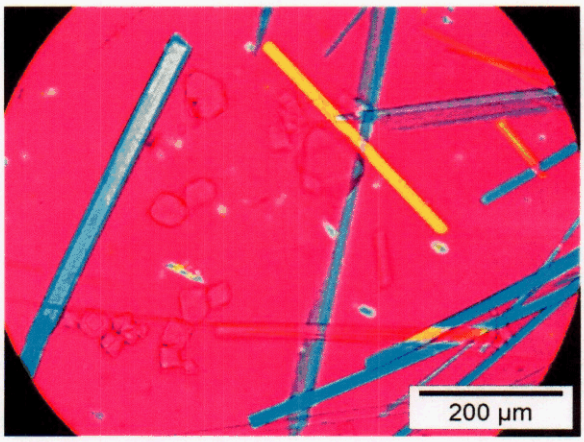

Figure 3.1.3-3. Mixture of $Na_7F(PO_4)_2 \cdot 19H_2O$ (isotropic octahedra) and $Na_3PO_4 \cdot 12H_2O$ (anisotropic needles) from Tank AX-101; solids formed on cooling AX-101 supernatant liquid from 50 °C to 20 °C.

Crossed polars with Red I compensator, 16x objective.

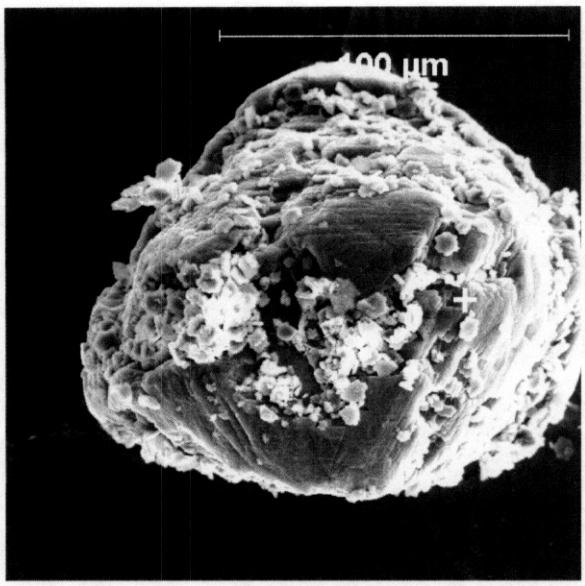

Figure 3.1.3-4. High magnification secondary electron image of Tank SX-101, solids forming from dissolution water on cooling from 50 °C to ambient temperature.

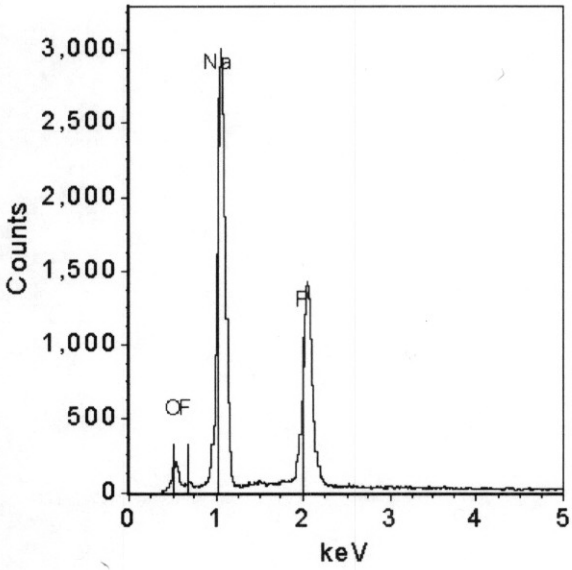

Figure 3.1.3-5. EDS spectrum from spot marked with + in Figure 3.1.3-4, 15Kv, 30% spot.

3.2 Na_3FSO_4

Name: Sodium fluoride sulfate	**ICDD PDF#:** 25-0827
Chemical Formula: Na_3FSO_4	**Crystal System:** Hexagonal
Refractive Index: $n_o = 1.436$, $n_e = 1.439$	**Mineral Name:** Kogarkoite

Sodium fluoride sulfate is a congruent double salt that was first identified as a component of Hanford Site tank waste in approximately 1997. It is significantly less soluble than either of the corresponding single salts, so its presence is a major factor in predicting the important SO_4:Na mole ratio in feed streams to the vitrification plant.

The crystal habit tends to be somewhat irregular, but the most characteristic shape is the stacked hexagonal plates like those shown in Figure 3.2.3-3.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages. The x-ray diffractogram of the standard (Figure 3.2.1-7) contains a major contribution from the contaminant $NaNO_3$ precipitated from the supernatant liquid during sample preparation.

3.2.1 Na_3FSO_4 Standard

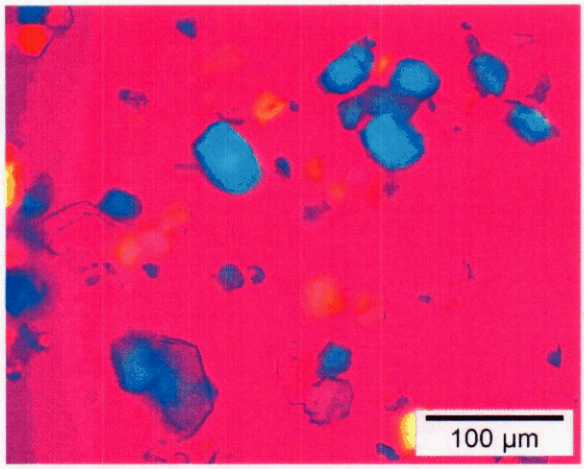

Figure 3.2.1-1. Na_3FSO_4 standard.

Blue and orange colors characteristic of very low birefringence (n_o=1.436, n_e=1.439).

Crossed polars with Red I compensator, 16x objective.

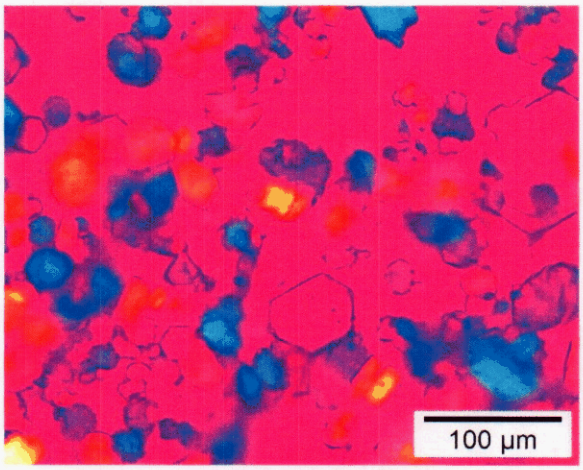

Figure 3.2.1-2. Na_3FSO_4 standard.

Low birefringence, with occasional six-sided plate with optic axis parallel to light path, giving crystal the appearance of being isotropic.

Crossed polars with Red I compensator, 16x objective.

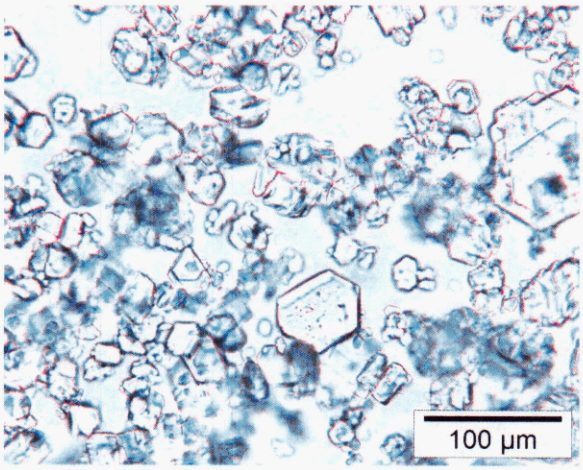

Figure 3.2.1-3. Na_3FSO_4 standard.

Same field of view as Figure 3.2.1-2; Becke line goes in as stage is moved away from objective ($n_{xtal} > n_{liq}$).

Uncrossed polars, 16x objective.

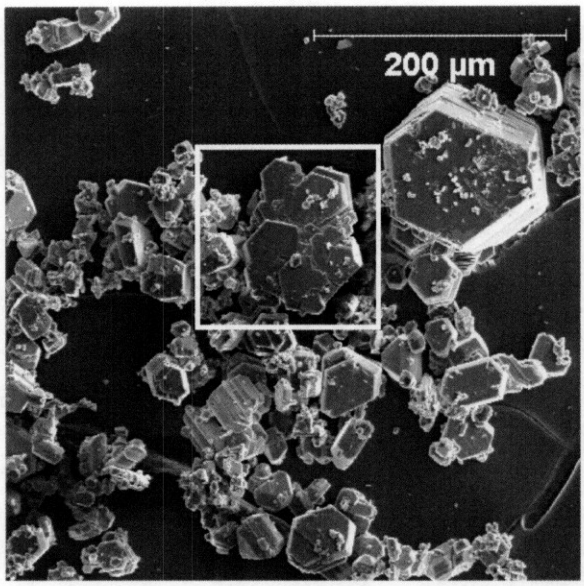

Figure 3.2.1-4. Low magnification secondary electron image of $Na_3F(SO_4)$ standard.

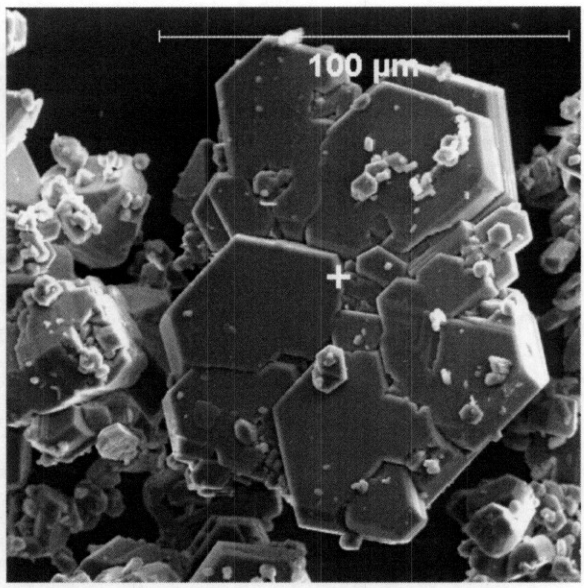

Figure 3.2.1-5. High magnification secondary electron image of $Na_3F(SO_4)$ standard.

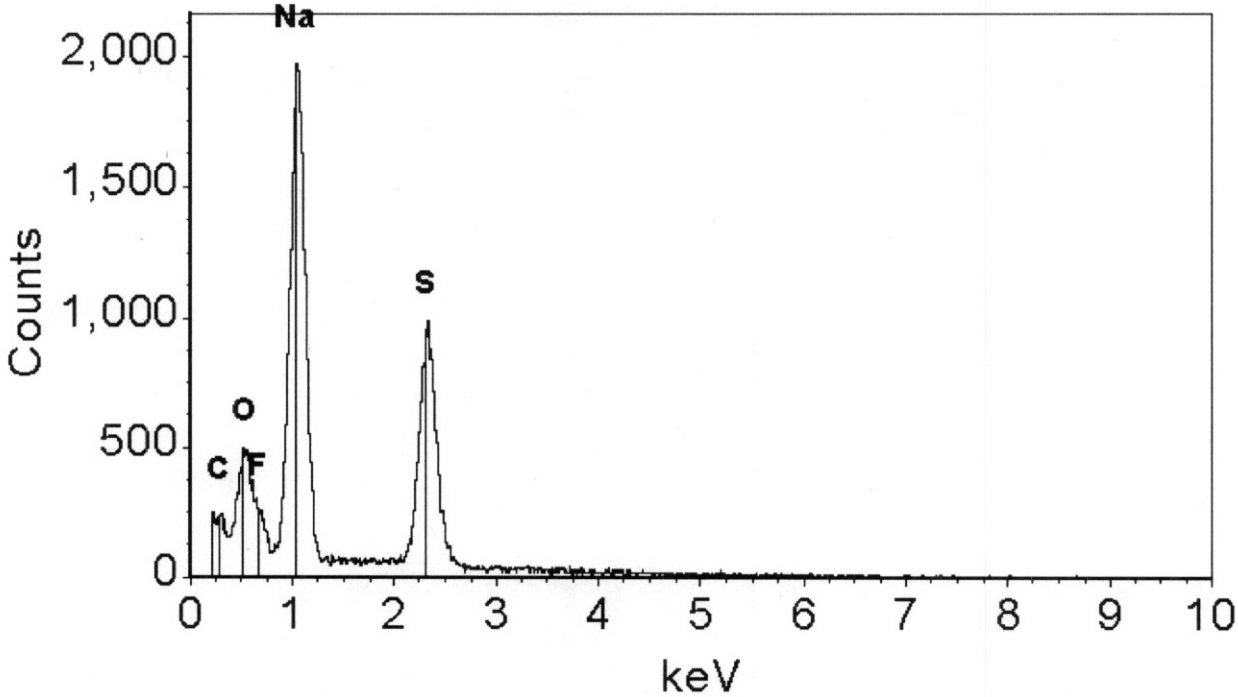

Figure 3.2.1-6. EDS spectrum from spot marked with + in Figure 3.2.1-5, 15Kv, 30% spot.

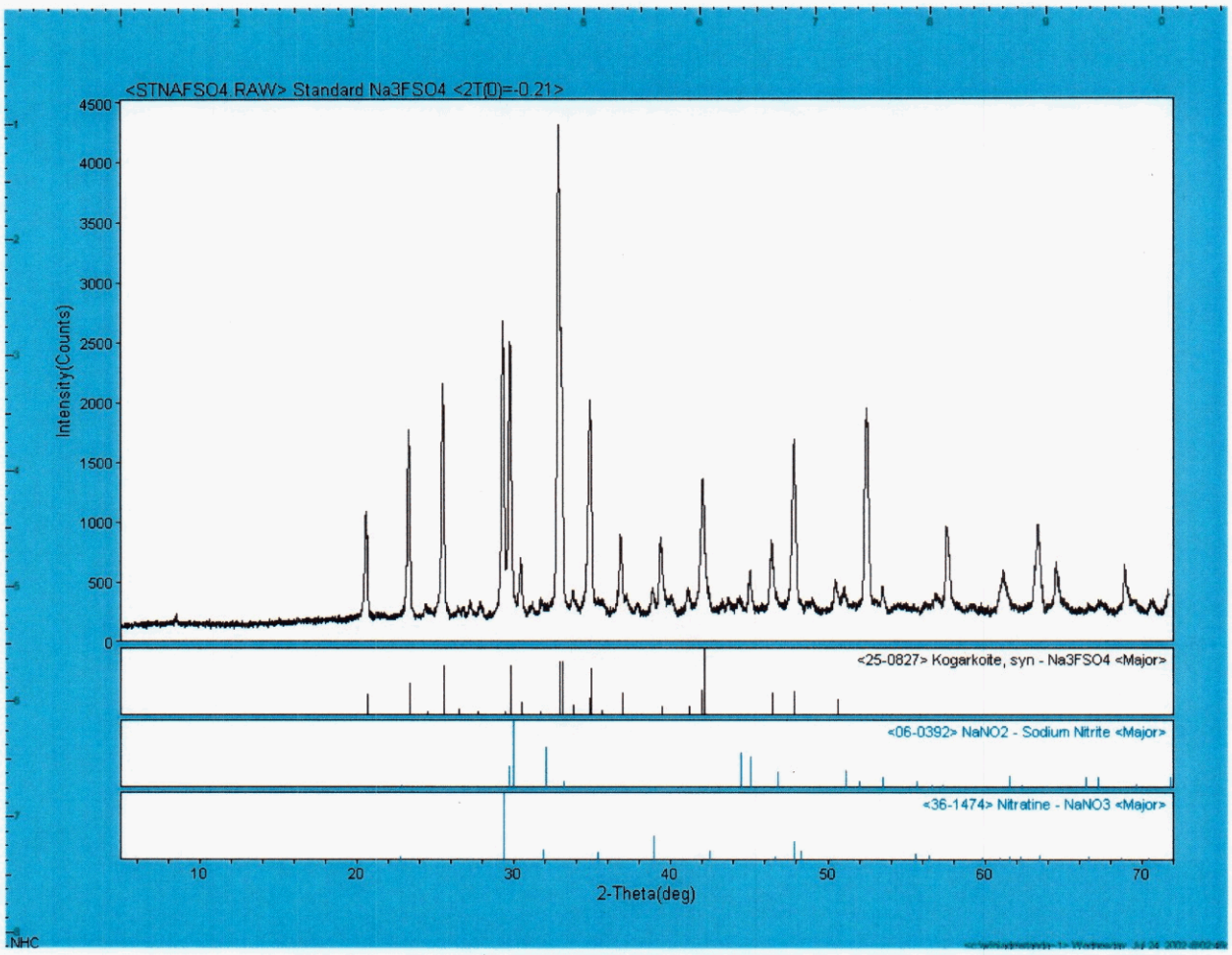

Figure 3.2.1-7. XRD spectrum of $Na_3F(SO_4)$ standard.

3.2.2 Occurrence of Na_3FSO_4 in Tank BY-109 Saltcake

Na_3FSO_4 was identified by all three methods in BY9Orig, BY9Dil1, BY9Dil2, BY9Evap4 and BY9Evap5. This phase was identified by two methods in samples BY9Dil3 and BY9Evap6. Only PLM detected it in sample BY9Evap3.

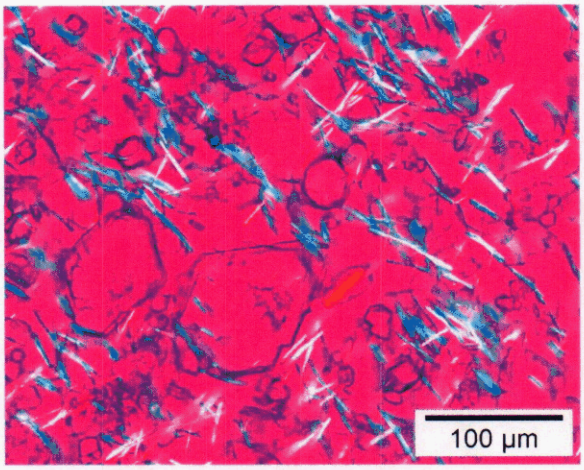

Figure 3.2.2-1. Sample BY9Evap4.

Three phases:

1. $Na_2C_2O_4$ anisotropic needles, blue perpendicular to slow direction of compensator.
2. Isotropic $Na_7F(PO_4)_2 \cdot 19H_2O$ distorted octahedral crystals.
3. Large six-sided plates of Na_3FSO_4 with optic axis perpendicular to stage.

Crossed polars with Red I compensator, 16x objective.

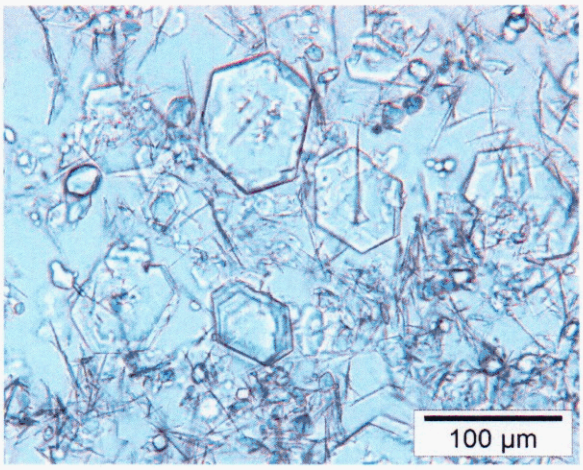

Figure 3.2.2-2. Sample BY9Evap4.

Large six-sided Na_3FSO_4 crystals more obvious with uncrossed polars.

Uncrossed polars, 16x objective.

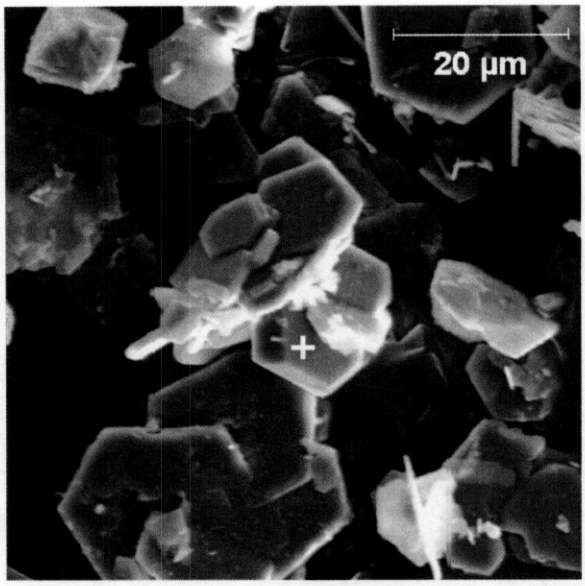

Figure 3.2.2-3. High magnification secondary electron image of BY9Dil1 sample.

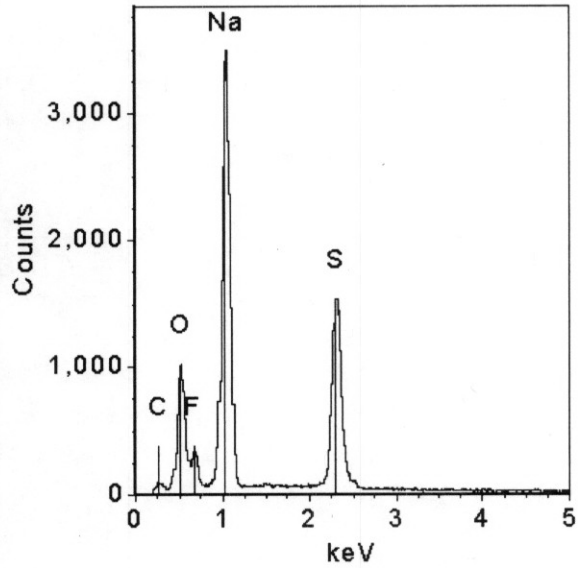

Figure 3.2.2-4. EDS spectrum from spot marked with + in Figure 3.2.2-3, 15Kv, 30% spot.

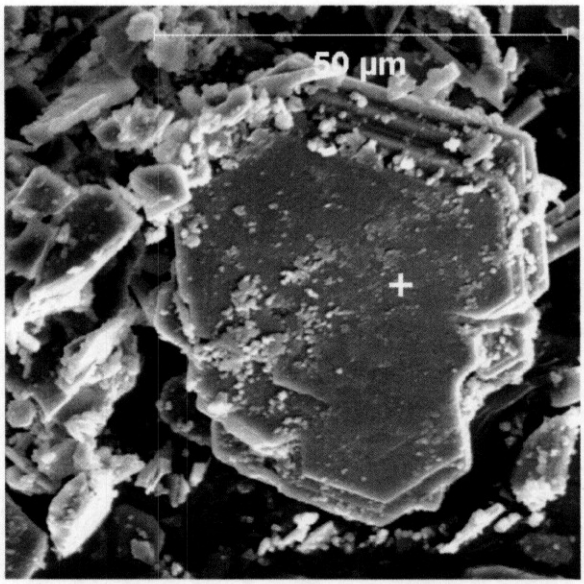

Figure 3.2.2-5. High magnification secondary electron image of BY9Evap5 sample.

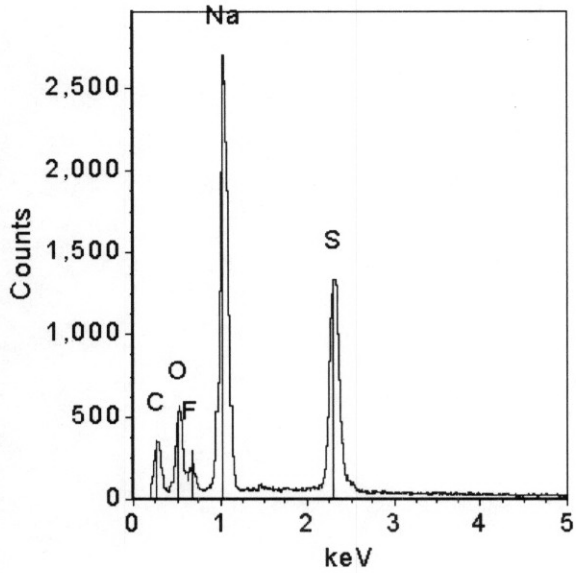

Figure 3.2.2-6. EDS Spectrum from spot marked with + in Figure 3.2.2-5, 15Kv, 30% spot.

3.2.3 Other Occurrences of Na_3FSO_4

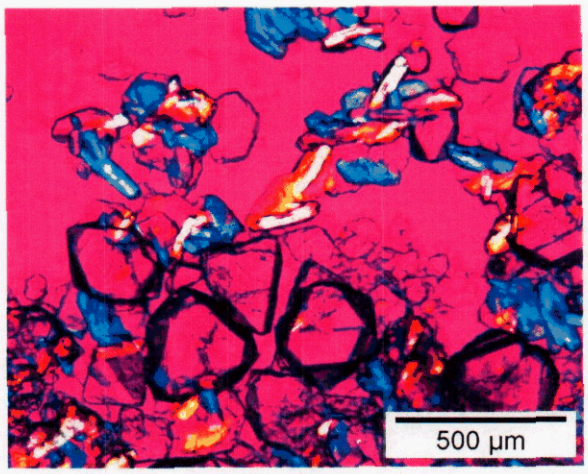

Figure 3.2.3-1. Mixture of Na_3FSO_4 and $Na_7F(PO_4)_2 \cdot 19H_2O$ (compare Figure 3.1.3-1); colorful blue/orange plates of Na_3FSO_4 and larger, high-contrast isotropic $Na_7F(PO_4)_2 \cdot 19H_2O$ crystals formed by mixing equal amounts of dissolved Tank BY-109 saltcake and dissolved Tank U-107 saltcake.

Crossed polars with Red I compensator, 4x objective.

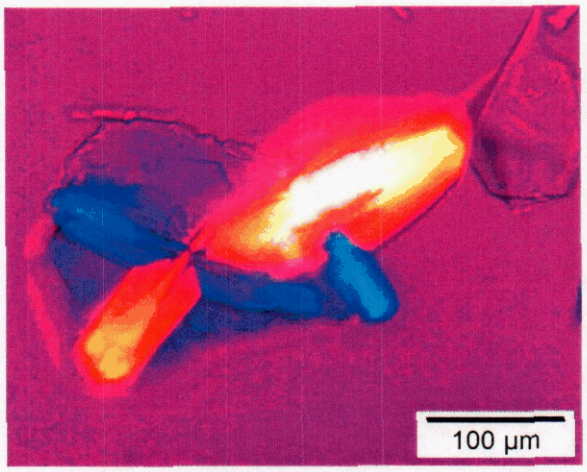

Figure 3.2.3-2. Na_3FSO_4 twinned plates from same sample as Figure 3.2.3-1, viewed at higher magnification.

Crossed polars with Red I compensator, 16x objective.

Figure 3.2.3-3. SEM photo of Na_3FSO_4 crystals from Tank BY-102; original Polaroid[5] photo taken with JEOL[6] 35CF SEM instrument.

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[5] Polariod is a trademark of the Polaroid Corporation, Cambridge, Massachusetts.

[6] JEOL is a trademark of the JEOL Ltd. Corporation, Tokyo, Japan.

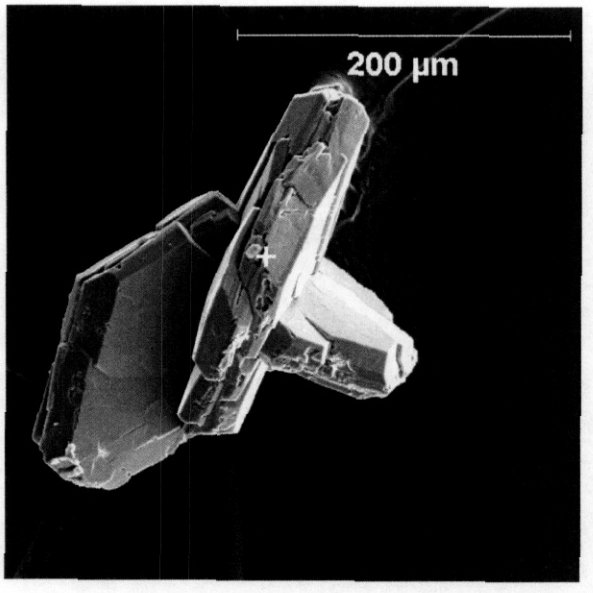

Figure 3.2.3-4. High magnification secondary electron image of Na_3FSO_4 crystals formed from a mixture of dissolved saltcake from Tanks BY-109 and U-107.

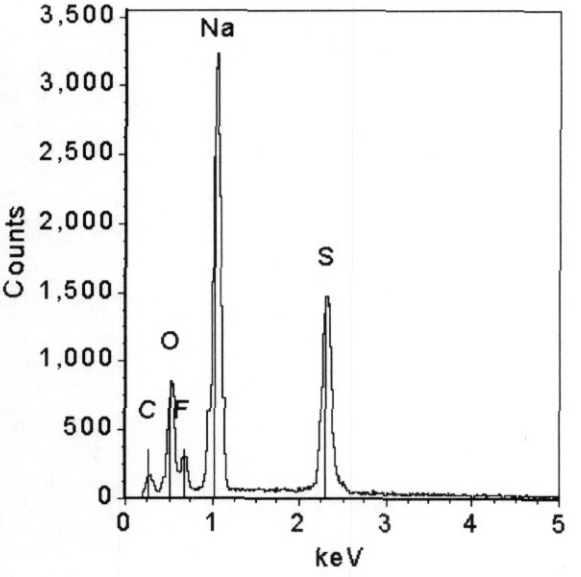

Figure 3.2.3-5. EDS spectrum from spot marked with + in Figure 3.2.3-4, 15Kv, 30% spot.

3.3 NaF

Name: Sodium fluoride	**ICDD PDF#:** 36-1455
Chemical Formula: NaF	**Crystal System:** Cubic
Refractive Index: n = 1.325	**Mineral Name:** Villiaumite

Due to its propensity to form double salts with phosphate and sulfate, the sodium fluoride single salt is uncommon (but not unknown) in Hanford Site tank waste. The photographs of the standard (Figures 3.3.1-1 through 3.3.1-8) are fairly representative of examples that have been observed in tank waste.

Crystals of NaF are easily confused with crystals of $Na_7F(PO_4)_2 \cdot 19H_2O$ with the PLM, as both salts are isotropic and often octahedral in shape. They are distinguishable by the Becke line test, because the refractive index of NaF is invariably lower than the refractive index of the fluid surrounding the crystals, while the situation is reversed for the double salt crystals.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

3.3.1 NaF Standard

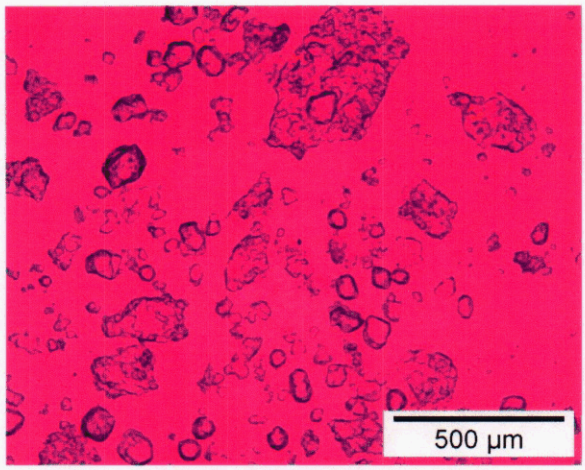

Figure 3.3.1-1. NaF standard.

Isotropic; mixture of near-perfect octahedral crystals and irregular agglomerates.

Crossed polars with Red I compensator, 4x objective.

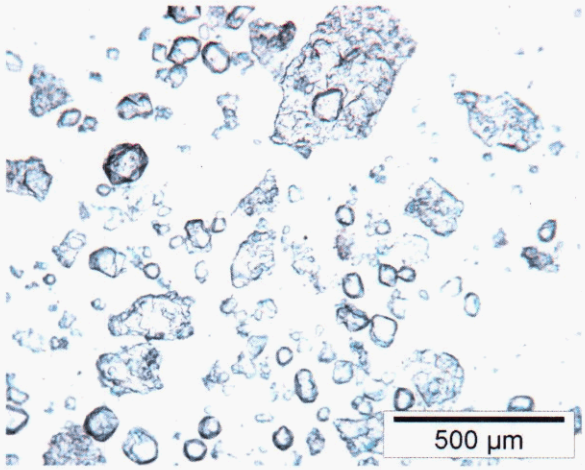

Figure 3.3.1-2. NaF standard.

Same field of view as Figure 3.3.1-2; Becke line goes out as stage is moved away from objective ($n_{xtal} < n_{liq}$), opposite of the more common isotropic crystals in tank waste, $Na_7F(PO_4)_2 \cdot 19H_2O$.

Uncrossed polars, 4x objective.

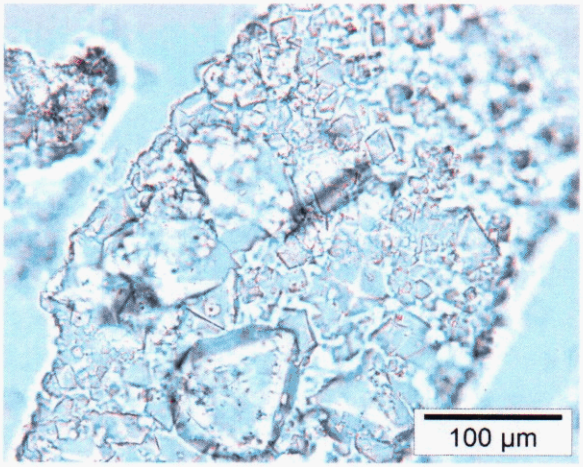

Figure 3.3.1-3. NaF standard.

Close-up of agglomerate near top of Figure 3.3.1-2; agglomerates appear to be made of very tiny octahedra and cubes.

Uncrossed polars, 16x objective.

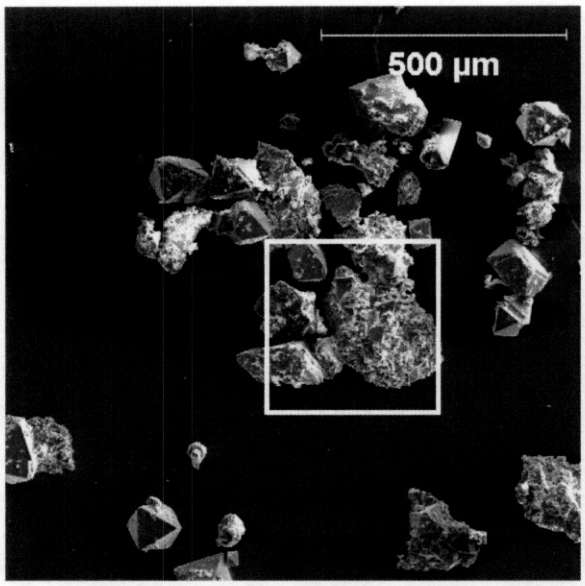

Figure 3.3.1-4. Low magnification secondary electron image of NaF standard.

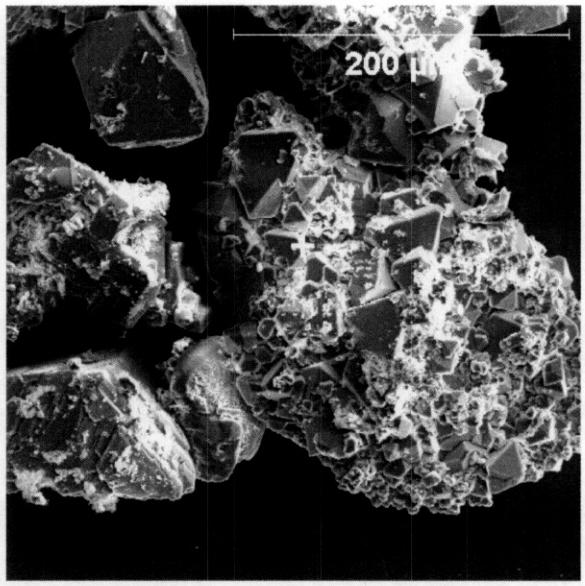

Figure 3.3.1-5. High magnification secondary electron image of NaF standard.

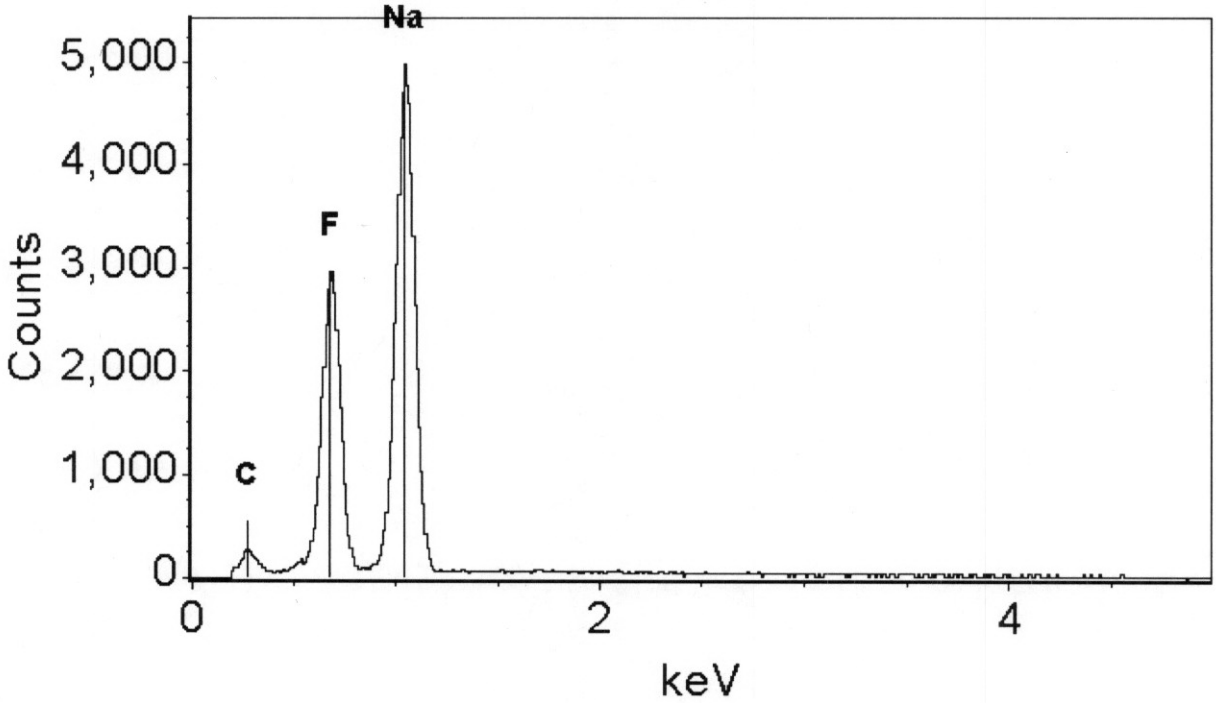

Figure 3.3.1-6. EDS spectrum from spot marked with + in Figure 3.3.1-5, 15Kv, 30% spot.

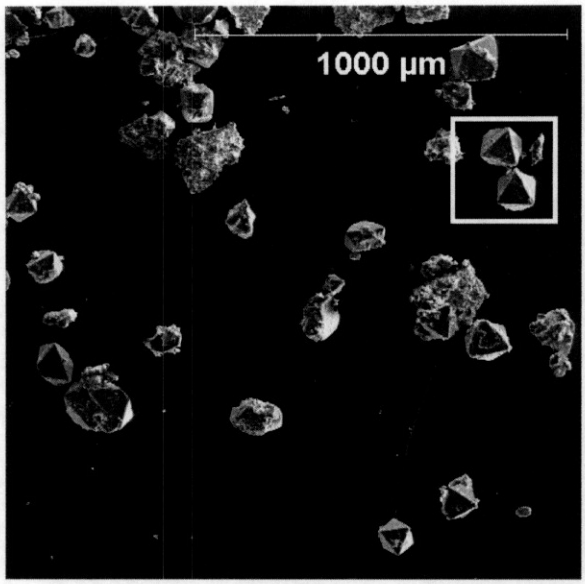

Figure 3.3.1-7. Low magnification secondary electron image of NaF standard.

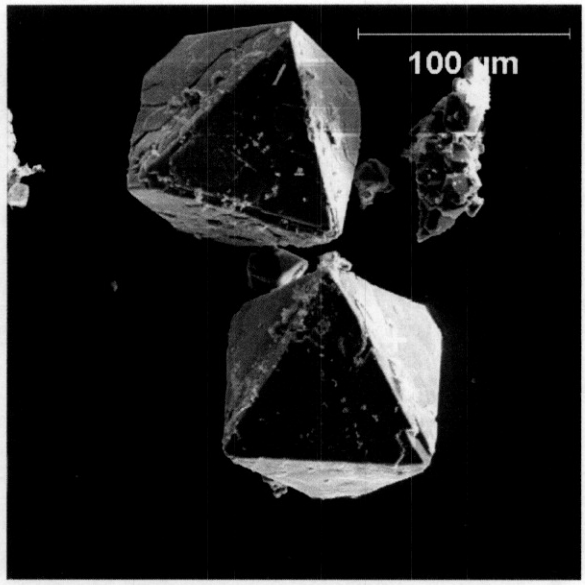

Figure 3.3.1-8. High magnification secondary electron image of NaF standard.

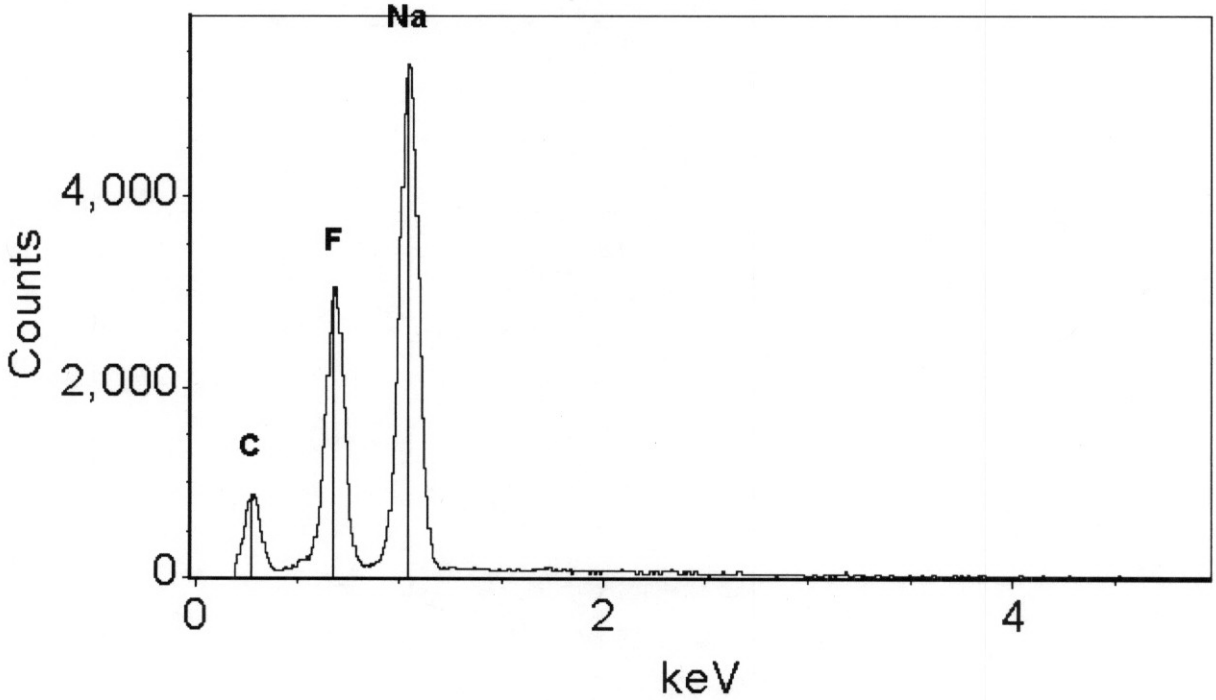

Figure 3.3.1-9. EDS spectrum from spot marked with + in Figure 3.3.1-8, 15Kv, 30% spot.

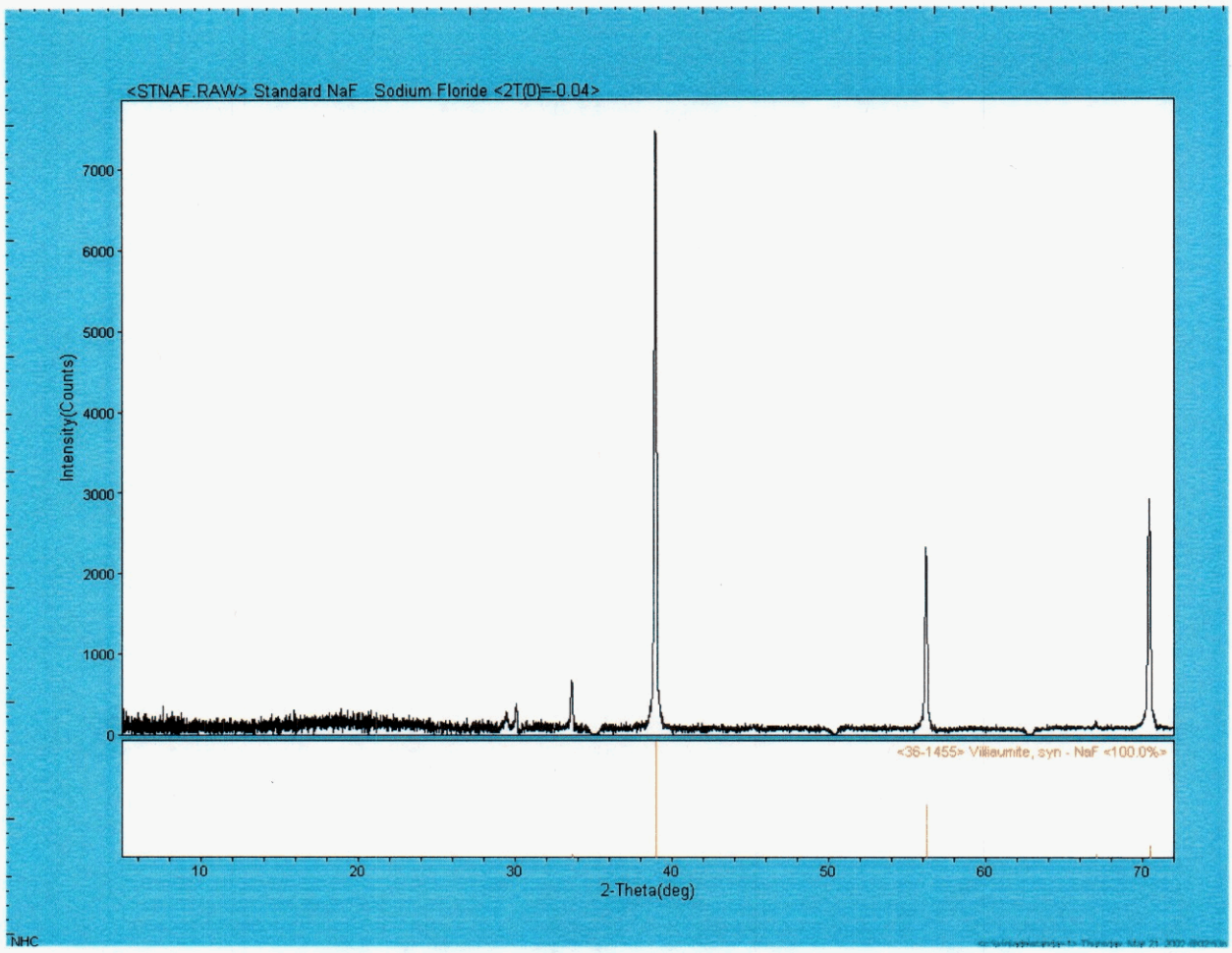

Figure 3.3.1-10. XRD spectrum of NaF standard.

3.3.2 Occurrence of NaF in Tank BY-109 Saltcake

The SEM and XRD agreed on the presence of NaF in one sample (BY9Dil2). The XRD identified NaF in BY9Orig, BY9Dil1, BY9Dil3, BY9Dil6 and BY9Evap6, while the SEM detected NaF in BY9Dil2, BY9Dil4 and BY9Dil5. This phase was not detected by PLM. The lack of agreement among the methods is due to the small size and the low concentration of this phase in the samples.

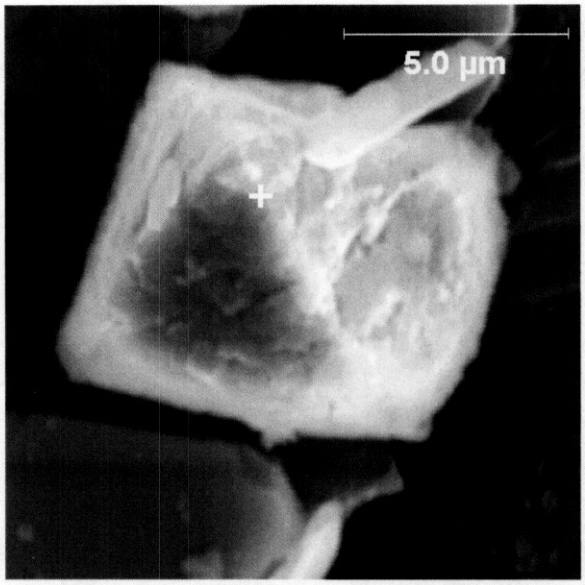

Figure 3.3.2-1. High magnification secondary electron image of BY9Dil2 sample.

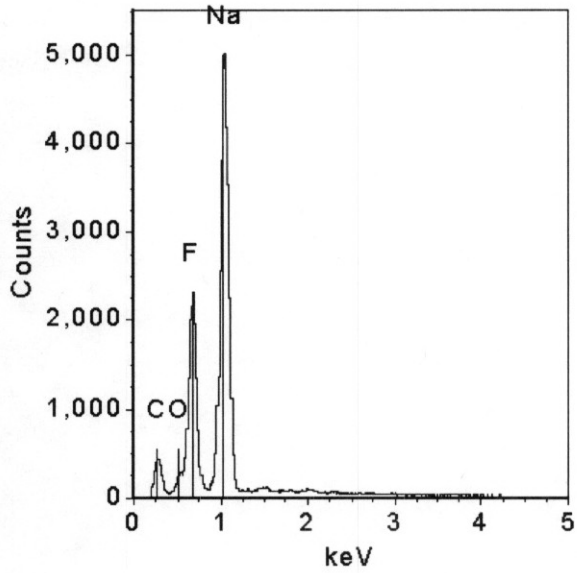

Figure 3.3.2-2. EDS spectrum from spot marked with + in Figure 3.3.2-1, 15Kv, 30% spot.

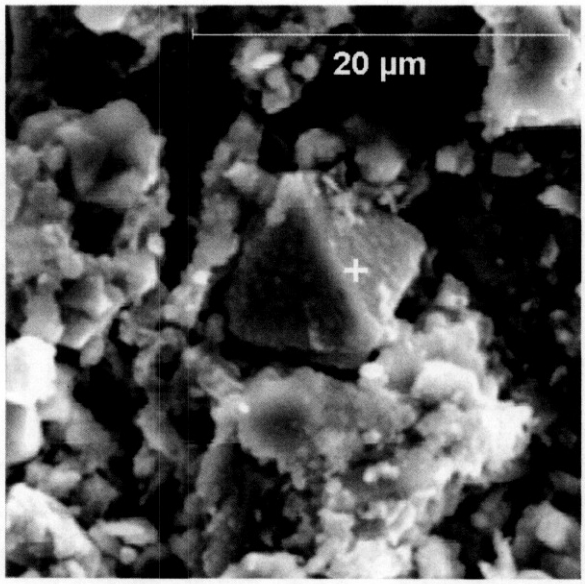

Figure 3.3.2-3. High magnification secondary electron image of BY9Dil4 sample.

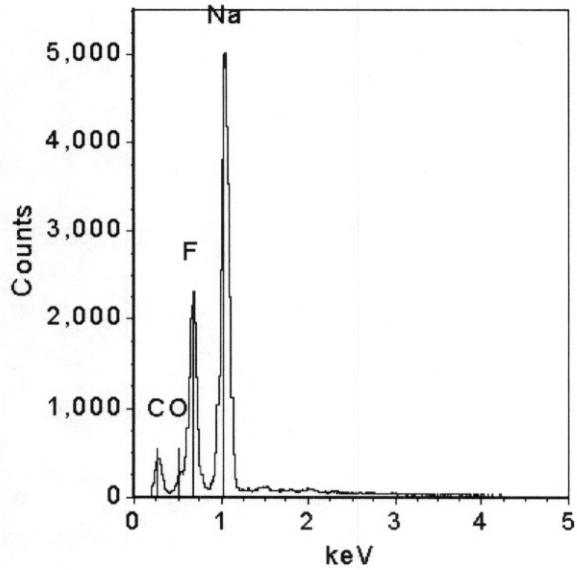

Figure 3.3.2-4. EDS spectrum from spot marked with + in Figure 3.3.2-3, 15Kv, 30% spot.

3.3.3 Other Occurrences of NaF

No images are available to show examples of NaF in other tanks. Sodium fluoride is not a common solid phase in Hanford Site tank waste, though it is known to be present in abundance in Tanks AW-103 and AW-105, which contain neutralized Zirflex decladding waste.

3.4 $Na_2C_2O_4$

Name: Sodium oxalate	**ICDD PDF#:** 20-1149
Chemical Formula: $Na_2C_2O_4$	**Crystal System:** Monoclinic
Refractive Index: $n_x = 1.415$, $n_y = 1.524$, $n_z = 1.592$	**Mineral Name:** Natroxalate

Sodium oxalate is a common component of Hanford Site tank waste. It is often present as a result of decomposition of larger organic molecules, either during the initial waste processing (e.g., the PUREX sugar denitration process) or during the years of aging in the waste tanks. It is one of the least soluble of the common sodium salts in tank waste.

The most common crystal habit of sodium oxalate that has been observed in tank waste is the very small needle habit like that shown in Figure 3.4.3-1. Due to the similarities with $Na_2CO_3 \cdot H_2O$ in optical properties, crystal habit, and chemical composition, these two salts are not easy to distinguish from one another, either by PLM or by SEM/EDS. However, the two salts have fairly distinctive XRD patterns, and larger crystals of $Na_2CO_3 \cdot H_2O$ have a relatively unique crystal habit (see Figures 3.5.1-3 and 3.5.3-1).

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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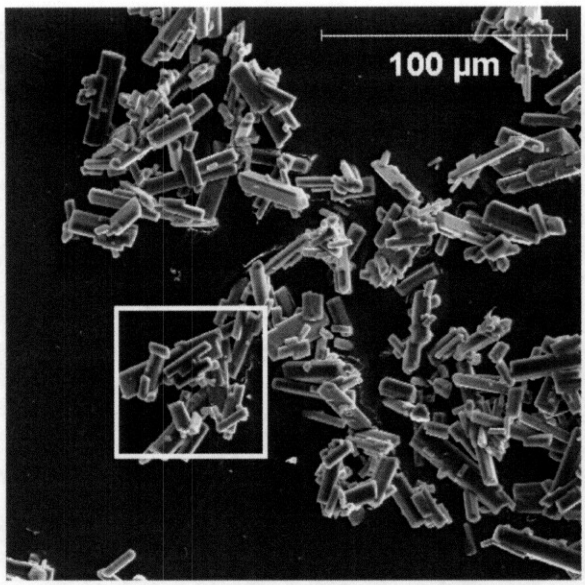

Figure 3.4.1-4. Low magnification secondary electron image of $Na_2C_2O_4$ standard.

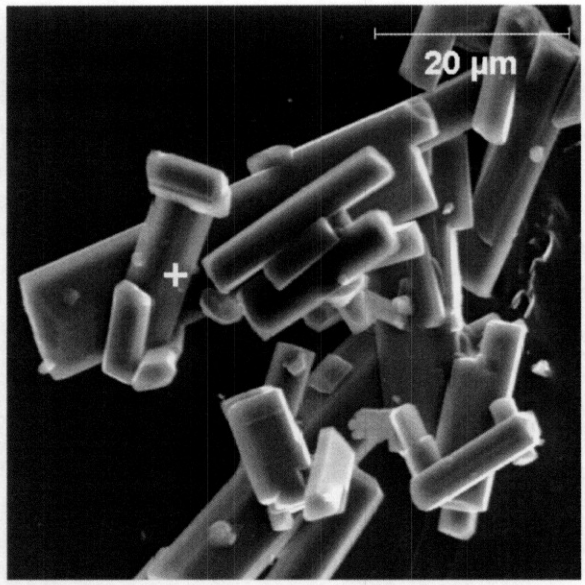

Figure 3.4.1-5. High magnification secondary electron image of $Na_2C_2O_4$ standard.

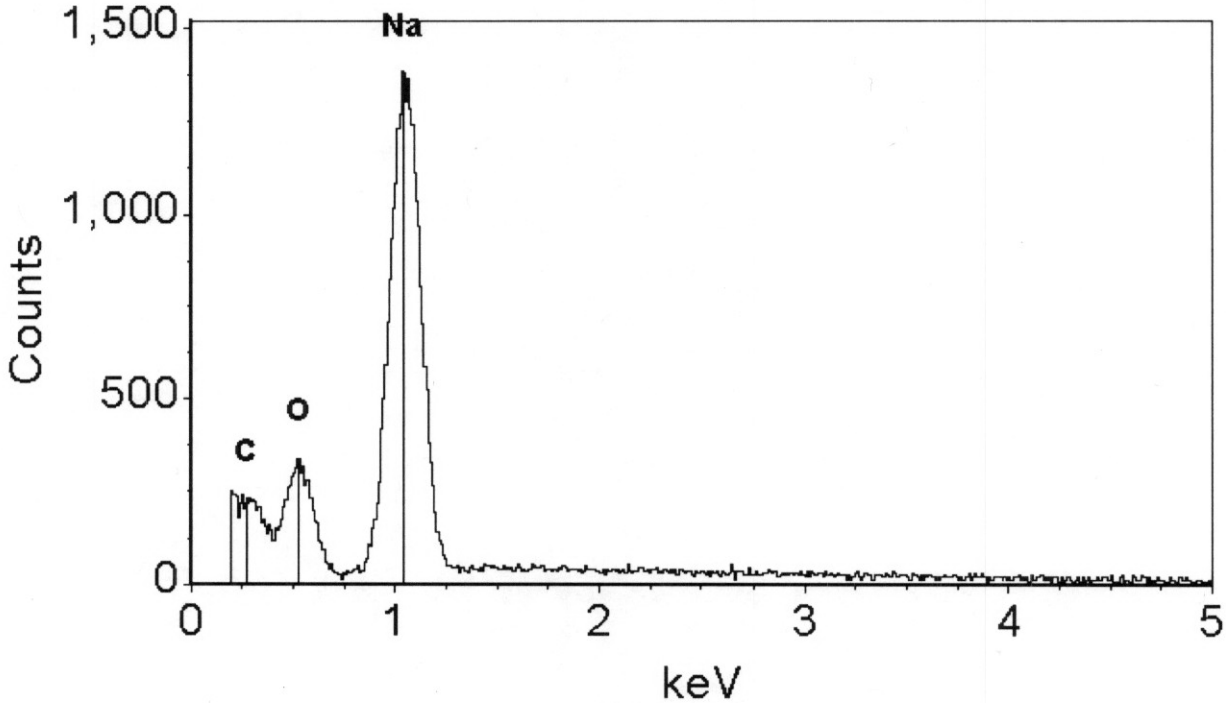

Figure 3.4.1-6. EDS spectrum from spot marked with + in Figure 3.4.1-5, 15Kv, 15% spot.

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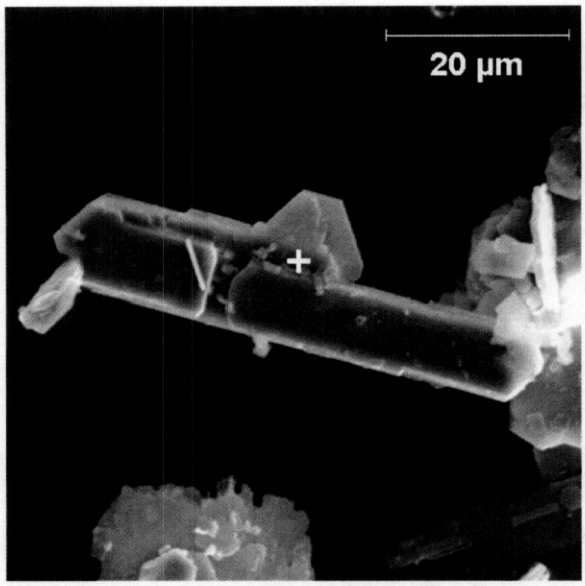

Figure 3.4.2-3. High magnification secondary electron image of BY9Dil1 sample.

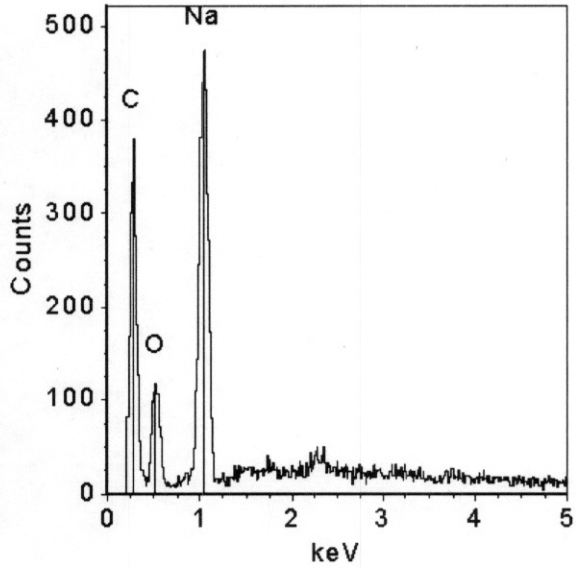

Figure 3.4.2-4. EDS spectrum from spot marked with + in Figure 3.4.2-3, 15Kv, 30% spot.

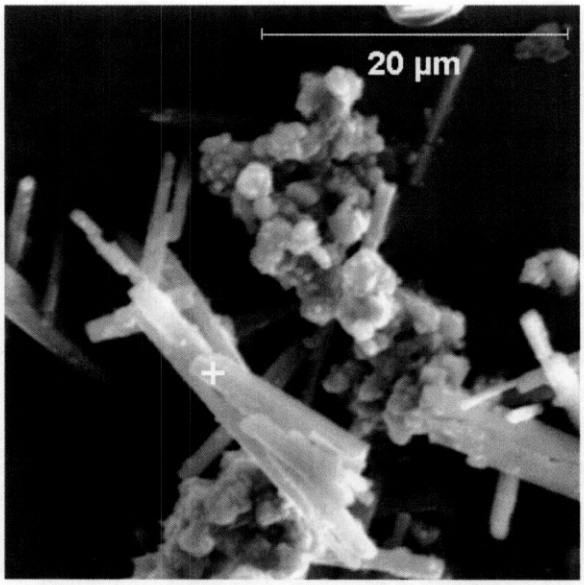

Figure 3.4.2-5. High magnification secondary electron image of BY9Evap2 sample.

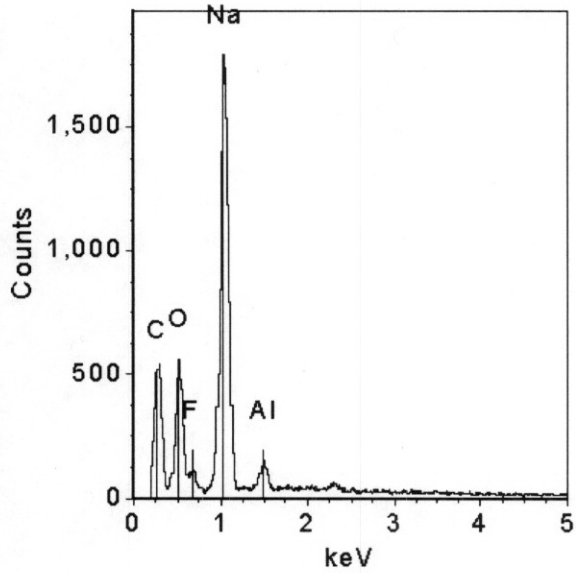

Figure 3.4.2-6. EDS spectrum from spot marked with + in Figure 3.4.2-5, 15Kv, 30% spot.

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Figure 3.4.3-2. High magnification secondary electron image of crystals formed from cooling Tank S-112 liquid from 50 °C to ambient temperature.

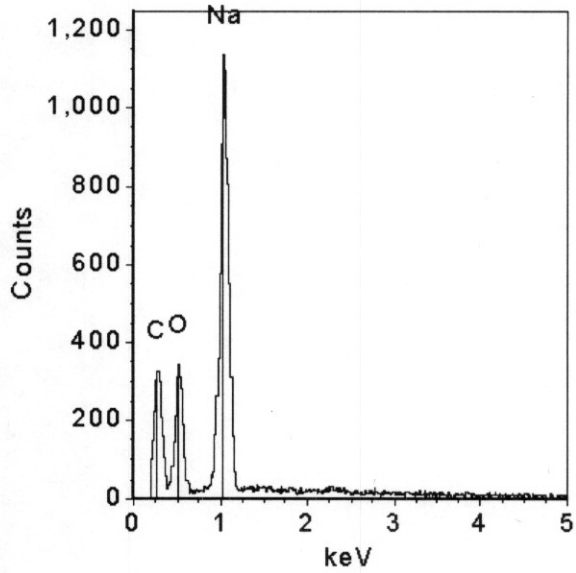

Figure 3.4.3-3. EDS spectrum from spot marked with + in Figure 3.4.3-2, 15Kv, 30% spot.

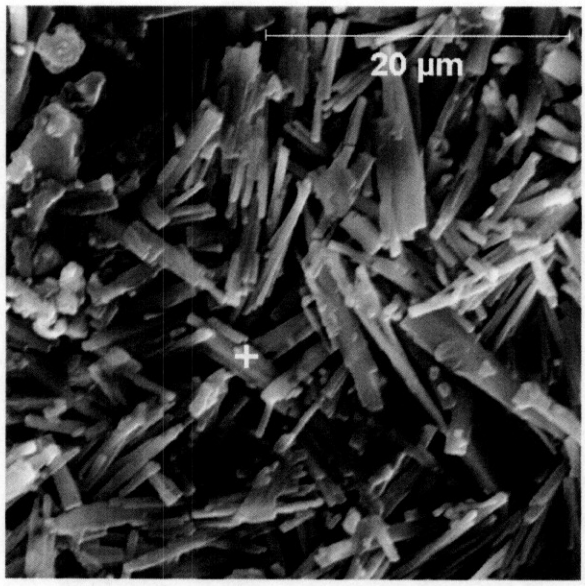

Figure 3.4.3-4. High magnification secondary electron image of crystals formed from a mixture of dissolved saltcake from Tanks S-110 and U-107.

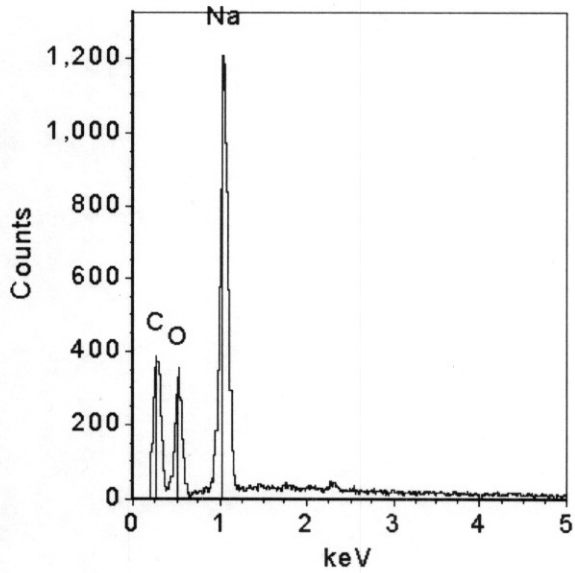

Figure 3.4.3-5. EDS spectrum from spot marked with + in Figure 3.4.3-4, 15Kv, 30% spot.

3.5 $Na_2CO_3 \cdot H_2O$

Phase I

Name: Sodium carbonate monohydrate	**ICDD PDF#:** 08-0448
Chemical Formula: $Na_2CO_3 \cdot H_2O$	**Crystal System:** Orthorhombic
Refractive Index: $n_x = 1.420$, $n_y = 1.507$, $n_z = 1.524$	**Mineral Name:** Thermonatrite

Phase II

Name: Sodium carbonate	**ICDD PDF#:** 18-1208
Chemical Formula: Na_2CO_3	**Crystal System:** Monoclinic
Refractive Index: $n_x = 1.415$, $n_y = 1.535$, $n_z = 1.546$	**Mineral Name:** Natrite

Sodium carbonate is a common salt in Hanford Site tank waste in either the anhydrous or monohydrate form. Higher hydrates of sodium carbonate are not observed in tank waste because of the high ionic strength of the liquid phase.

The diffractogram of the standard (Figure 3.5.1-7) consists of two sodium carbonate phases; the major phase being the monohydrate and a minor of the anhydrous phase. The diffractogram also contains a major contribution from the contaminant $NaNO_3$ and a minor contribution from $NaNO_2$ precipitated from the supernatant liquid. The monohydrate phase also displays a substantial amount of preferred orientation of the crystals.

PLM analysis suggested the presence of $Na_2CO_3 \cdot H_2O$ in the BY9Orig sample. However, that could not be confirmed by XRD, and the SEM analysis performed in this study would have a difficult time discriminating between this phase and the sodium oxalate.

The crystal habit of the standard is not necessarily representative of habit in waste samples; crystal habit tends to be variable for sodium carbonate.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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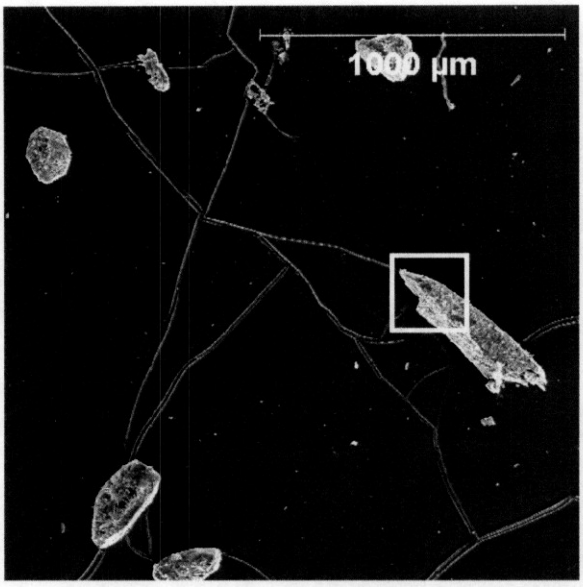

Figure 3.5.1-4. Low magnification secondary electron image of $Na_2CO_3 \cdot H_2O$ standard.

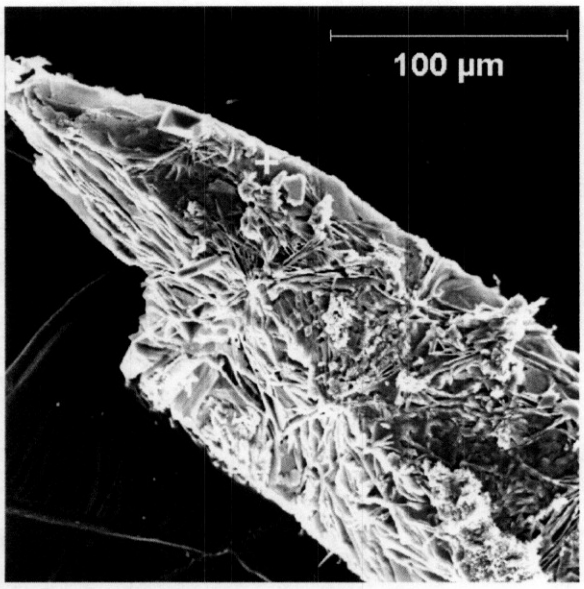

Figure 3.5.1-5. High magnification secondary electron image of $Na_2CO_3 \cdot H_2O$ standard.

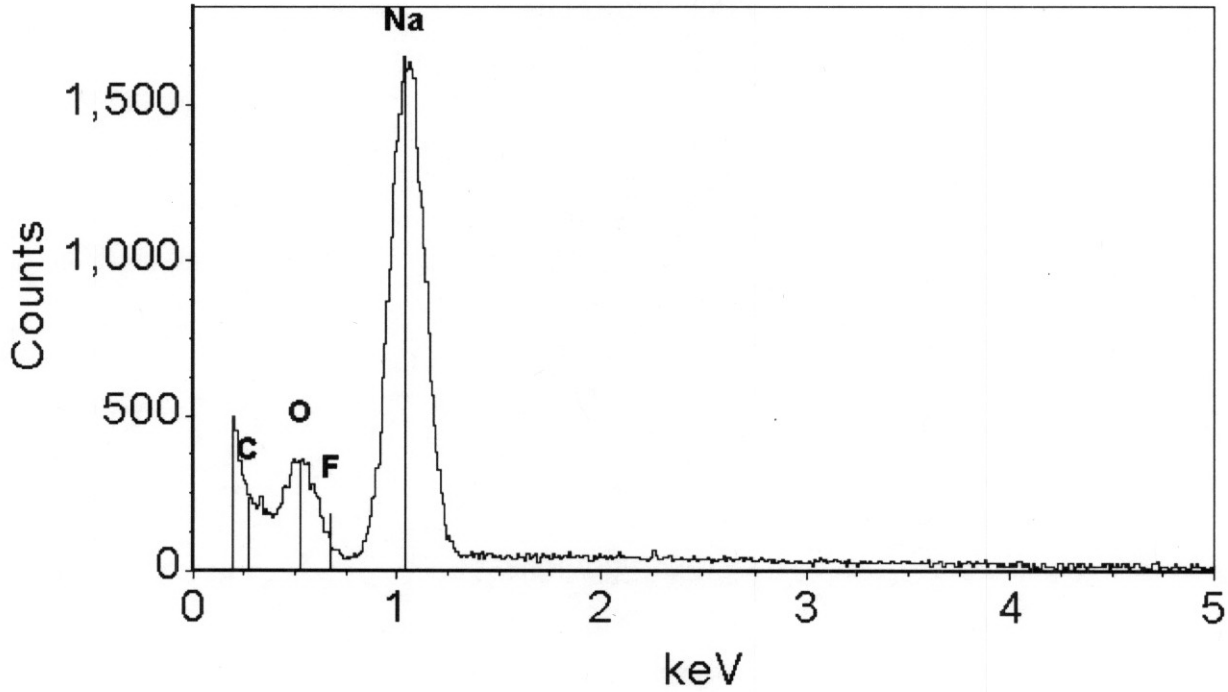

Figure 3.5.1-6. EDS spectrum from spot marked with + in Figure 3.5.1-5, 15Kv, 30% spot.

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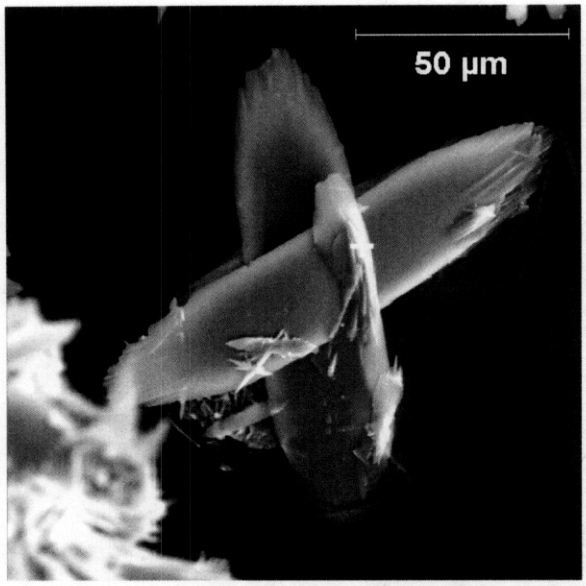

Figure 3.5.3-4. High magnification secondary electron image of crystals formed from the addition of 19.4M NaOH to the supernatant liquid from Tank AN-107.

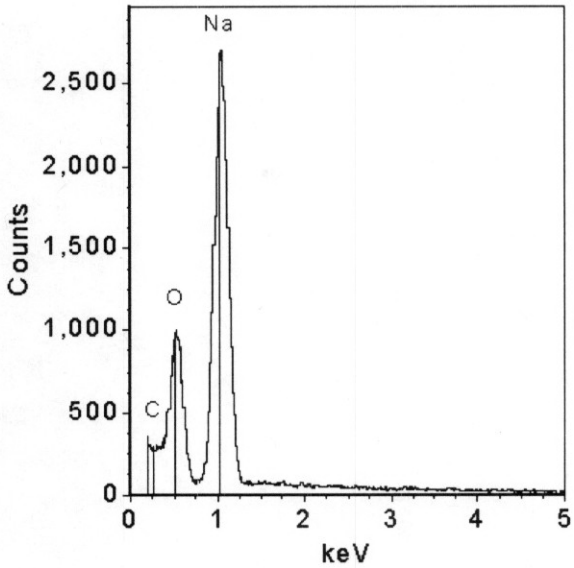

Figure 3.5.3-5. EDS spectrum from spot marked with + in Figure 3.5.3-4, 15Kv, 30% spot.

3.6 $Al(OH)_3$ GIBBSITE/BAYERITE

Name: Aluminum hydroxide	**ICDD PDF#:** 33-0018
Chemical Formula: $Al(OH)_3$	**Crystal System:** Monoclinic
Refractive Index: $n_x = 1.566$, $n_y = 1.566$, $n_z = 1.587$	**Mineral Name:** Gibbsite

Name: Aluminum hydroxide	**ICDD PDF#:** 20-0011
Chemical Formula: $Al(OH)_3$	**Crystal System:** Monoclinic
Refractive Index: Not applicable	**Mineral Name:** Bayerite

Name: Aluminum hydroxide	**ICDD PDF#:** Not applicable
Chemical Formula: $Al(OH)_3$	**Crystal System:** Not applicable
Refractive Index: Not applicable	**Mineral Name:** Amorphous aluminum hydroxide

The compound $Al(OH)_3$ 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 $Al(OH)_3$.

Gibbsite is probably the most common form of aluminum hydroxide found in Hanford Site tank waste, especially sludges (Rapko 2000). Its crystals are generally too small to characterize by PLM, but XRD makes identification possible.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages. No standard is available for bayerite. Its presence in BY9Evap3 was detected by XRD analysis.

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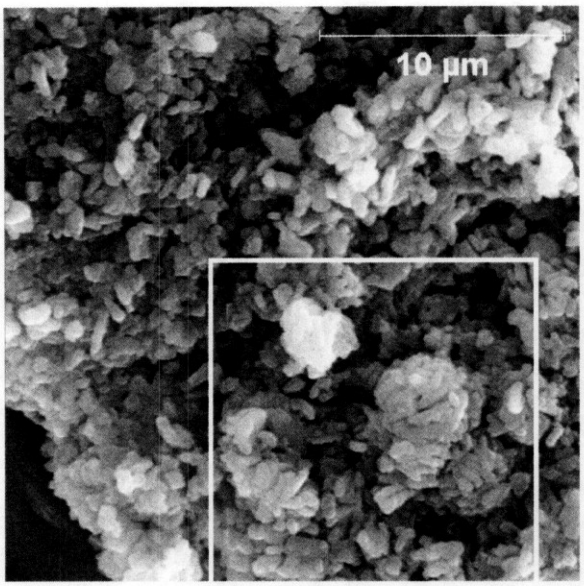

Figure 3.6.1-3. Low magnification secondary electron image of $Al(OH)_3$ standard.

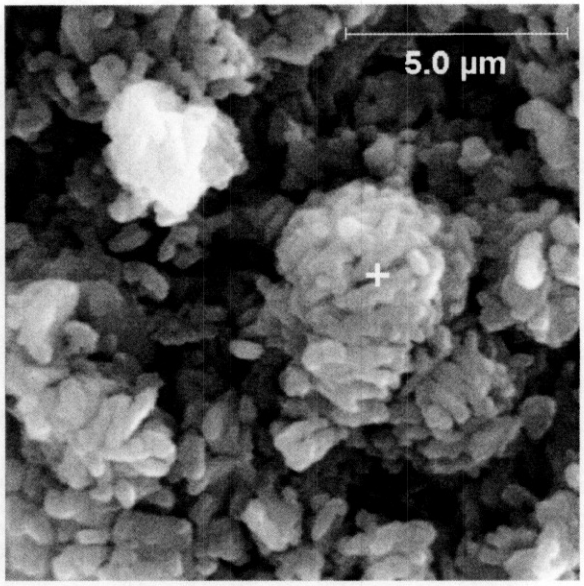

Figure 3.6.1-4. High magnification secondary electron image of $Al(OH)_3$ standard.

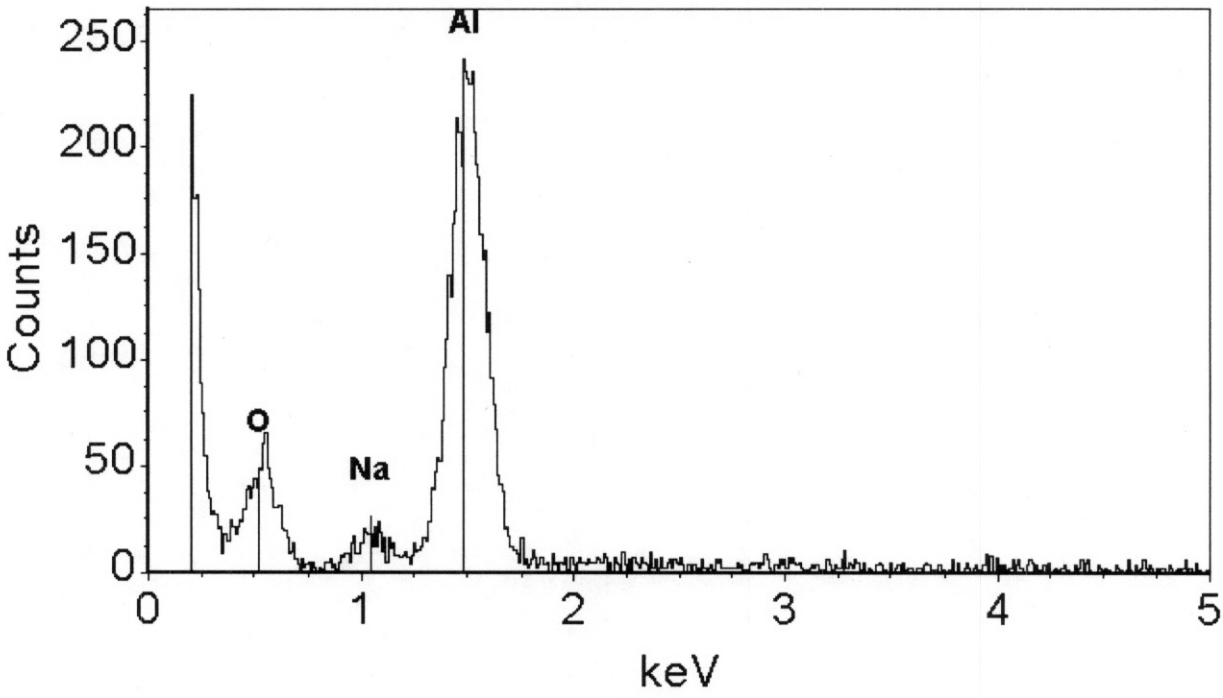

Figure 3.6.1-5. EDS spectrum from spot marked with + in Figure 3.6.1-4, 15Kv, 15% spot.

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3.6.2 Occurrence of $Al(OH)_3$ in Tank BY-109 Saltcake

Aluminum and oxygen-rich phases (typically with associated chromium) were identified as fine-grained aggregates by SEM analysis in samples BY9Dil5, BY9Dil6, BY9Resi and BY9Evap3. XRD identified the presence of bayerite in BY9Evap3 and gibbsite in BY9Resi. Bayerite and gibbsite are crystalline polymorphs of aluminum hydroxide. A standard for the bayerite polymorph was not produced for this study. The difficulty in the identification of aluminum hydroxide phases is due to the fine-grained, perhaps amorphous, nature in the samples. The existence of other elements (notably chromium) in the phase observed in the SEM could also lead to shifts in XRD peaks, further hampering identification.

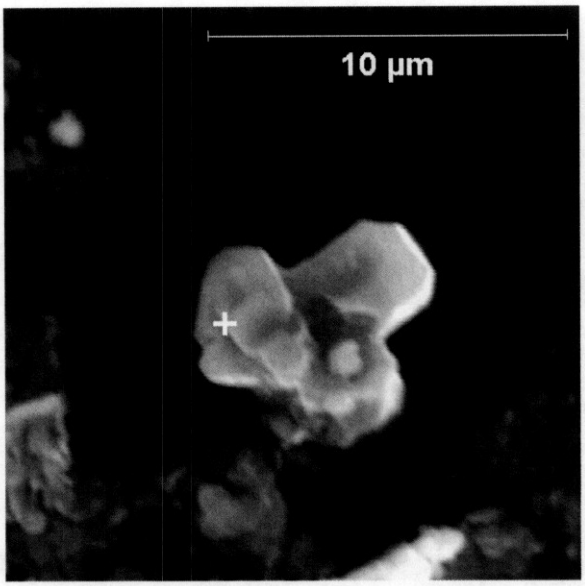

Figure 3.6.2-1. High magnification secondary electron image of BY9Resi sample.

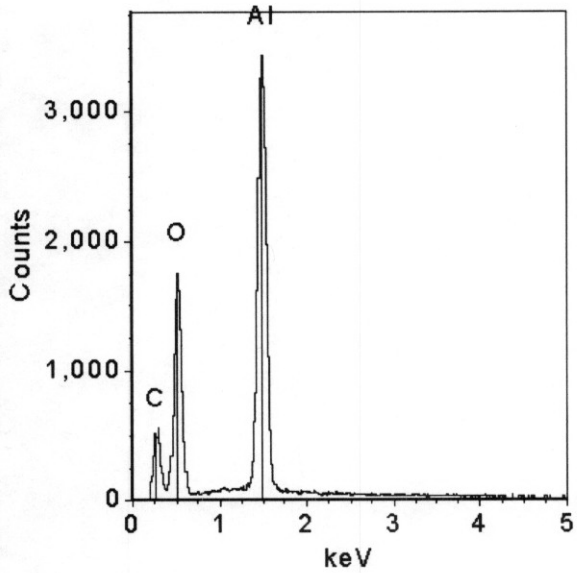

Figure 3.6.2-2. EDS spectrum from spot marked with + in Figure 3.6.2-1, 15Kv, 30% spot.

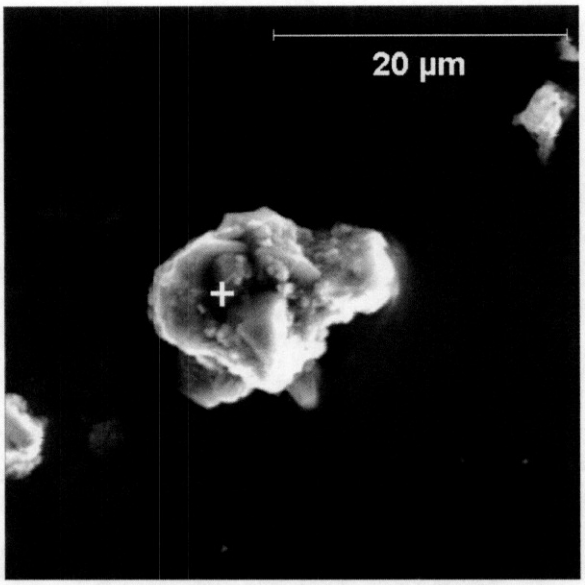

Figure 3.6.2-3. High magnification secondary electron image of BY9Dil5 sample.

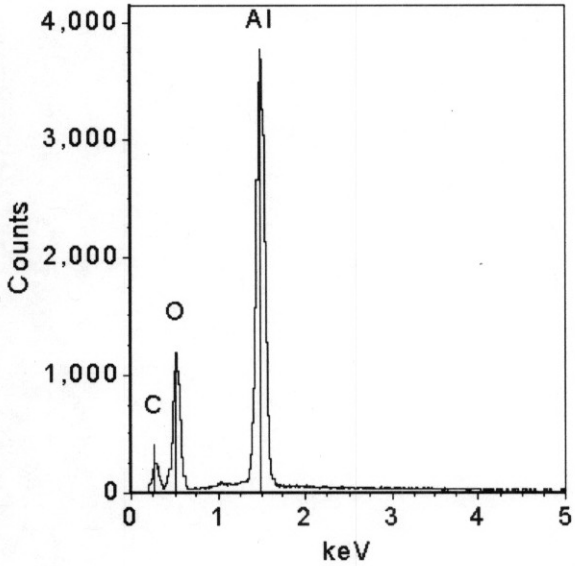

Figure 3.6.2-4. EDS spectrum from spot marked with + in Figure 3.6.2-3, 15Kv, 30% spot.

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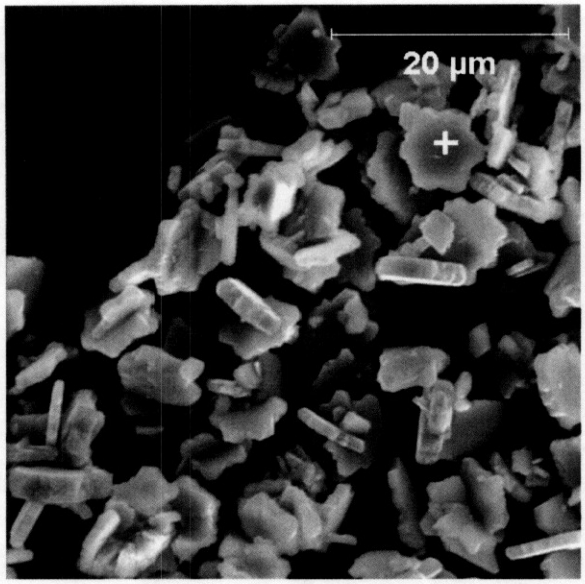

Figure 3.6.3-3. High magnification secondary electron image of Tank SX-101, gibbsite solids formed on cooling of dissolved saltcake from 50 °C to ambient temperature.

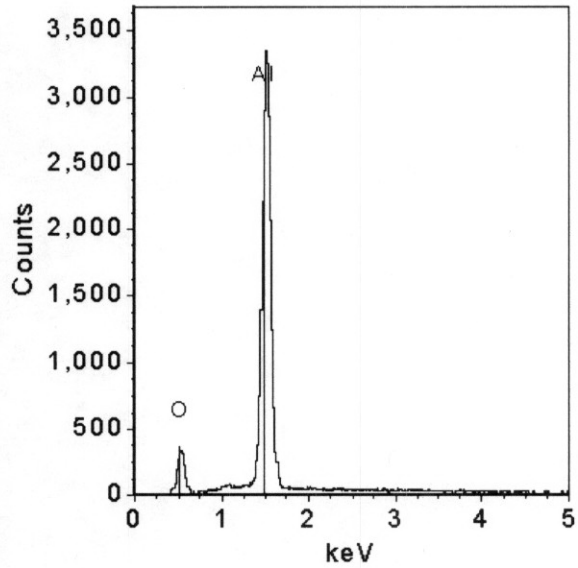

Figure 3.6.3-4. EDS spectrum from spot marked with + in Figure 3.6.3-3, 15 Kv, 30% spot.

3.7 Na_3AlF_6

Name: Sodium aluminum fluoride	**ICDD PDF#:** 25-0772
Chemical Formula: Na_3AlF_6	**Crystal System:** Monoclinic
Refractive Index: n = 1.34	**Mineral Name:** Cryolite

Sodium aluminum fluoride is identified in tank waste materials for the first time in with this report. It was only found in solids precipitating during the evaporation of the solution generated during Tank BY-109 saltcake dissolution.

3.7.1 Na_3AlF_6 Standards

We do not currently have a prepared standard for cryolite. The identification of this phase in the Tank BY-109 evaporating solution is based on the International Centre for Diffraction Data (ICDD) reference x-ray pattern and the EDS chemical composition.

3.7.2 Occurrence of Na_3AlF_6 in Tank BY-109 Saltcake

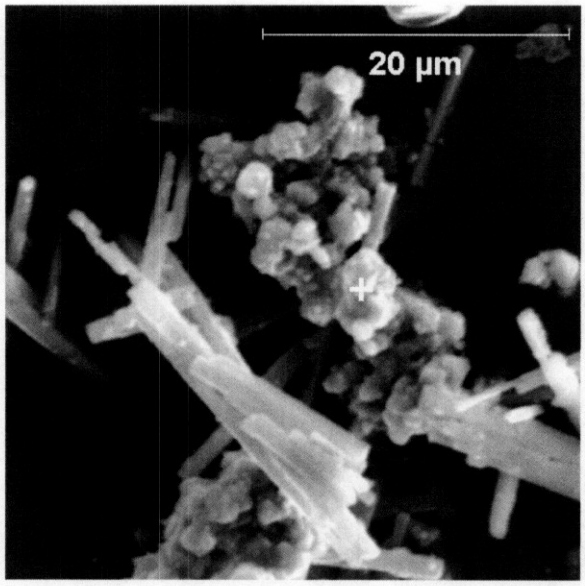

Figure 3.7.2-1. High magnification secondary electron image of BY9Evap2 sample, showing Na_3AlF_6 and $Na_2C_2O_4$ crystals.

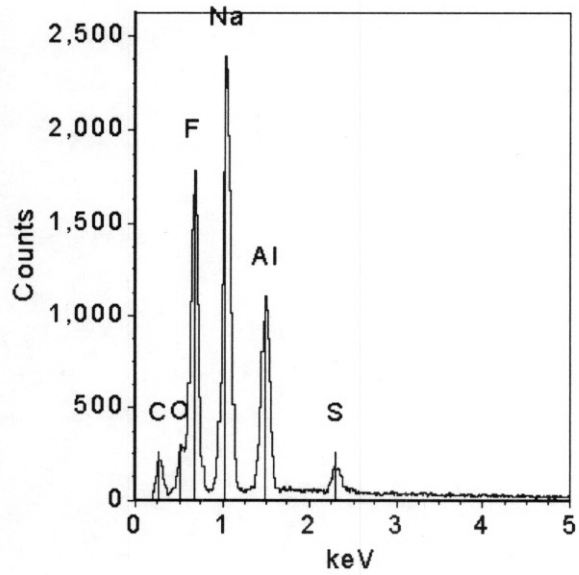

Figure 3.7.2-2. EDS spectrum from spot marked with + in Figure 3.7.2-1, 15Kv, 30% spot.

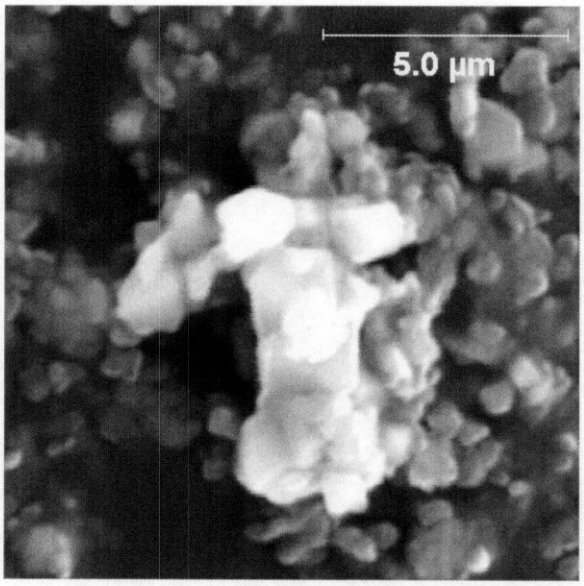

Figure 3.7.2-3. High magnification secondary electron image of BY9Evap3 sample.

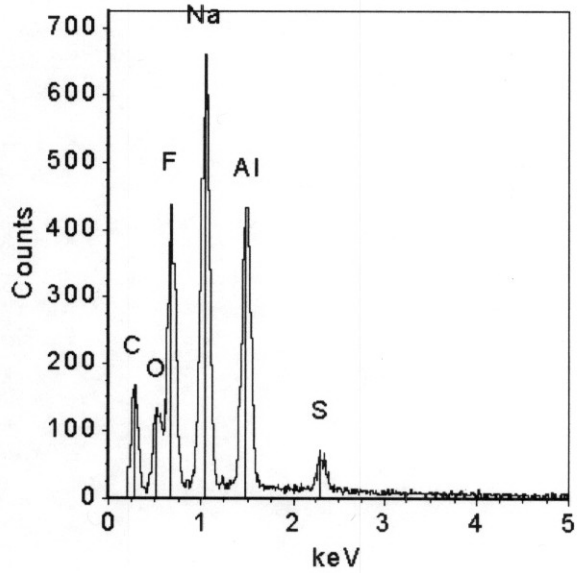

Figure 3.7.2-4. EDS spectrum from spot marked with + in Figure 3.7.2-3 15Kv, 20% spot.

3.7.3 Other Occurrences of Na_3AlF_6

No examples of Na_3AlF_6 in other tanks are available. This is the first time this phase has been reported in Hanford Site tank waste.

3.8 $Na_2U_2O_7$

Name: Sodium diuranate	**ICDD PDF#:** 43-0347
Chemical Formula: $Na_2U_2O_7$	**Crystal System:** Monoclinic
Refractive Index: n ~ 1.84	**Mineral Name:** None

Sodium diuranate was identified as a possible phase in Tank BY-109 using SEM backscattered imaging and obtaining EDS spectra of the brightest particles. The identification of the sodium/uranium-rich particulate as sodium diuranate was confirmed using slow scan XRD patterns of Tank S-112 residues from water and NaOH washing.

The occurrence of this phase is not unexpected, as it has been identified in several sludge tanks (Rapko 2000). It has also been noted in evaporator condenser deposits at Savannah River and in radioactive sludge storage modeling.

3.8.1 $Na_2U_2O_7$ Standards

We do not currently have a prepared standard for sodium diuranate. The identification of this phase in Tank BY-109 is based on the ICDD reference x-ray pattern and the EDS chemical composition.

3.8.2 Occurrence of $Na_2U_2O_7$ in Tank BY-109 Saltcake

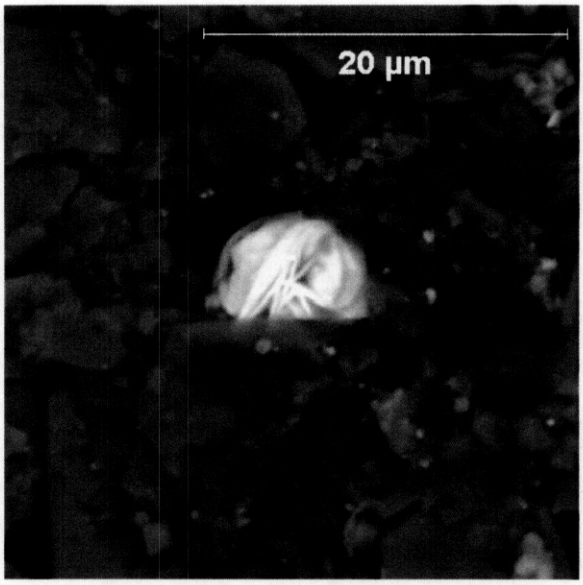

Figure 3.8.2-1. Backscatter electron image of BY9Orig sample.

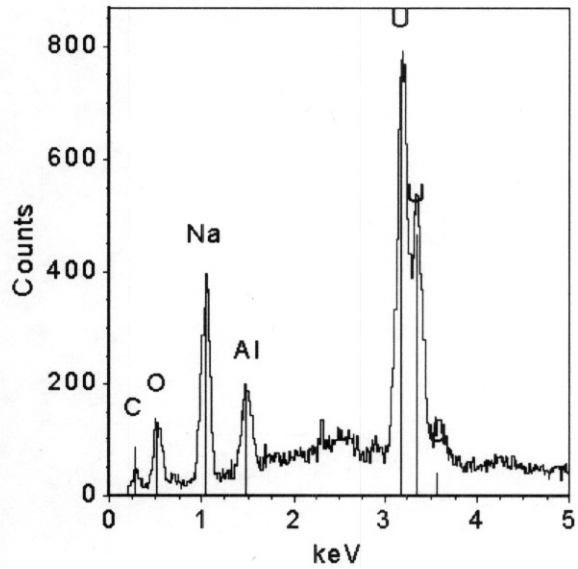

Figure 3.8.2-2. EDS spectrum from spot marked with + in Figure 3.8.2-1, 15Kv, 30% spot.

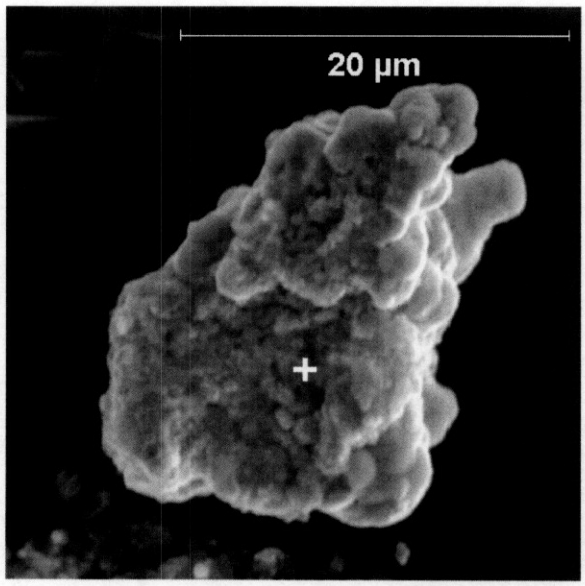

Figure 3.8.2-3. High magnification secondary electron image of BY9Resi sample.

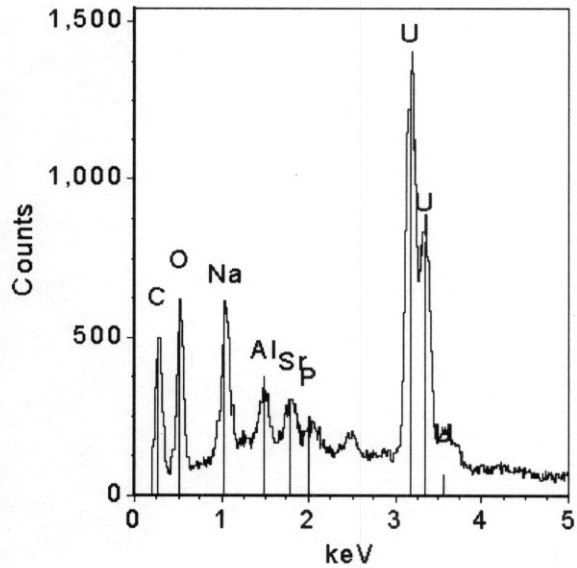

Figure 3.8.2-4. EDS spectrum from spot marked with + in Figure 3.8.2-3, 15Kv, 20% spot.

3.8.3 Other Occurrences of $Na_2U_2O_7$

Sodium diuranate ($Na_2U_2O_7$) was identified in Tank S-112 residues from water and sodium hydroxide washing of saltcake. The identity of the sodium/uranium-rich phses was confirmed by obtaining XRD data at a slow scan rate. This improved the peak signal relative to noise, allowing for a definitive match of the XRD pattern with the ICDD reference spectrum.

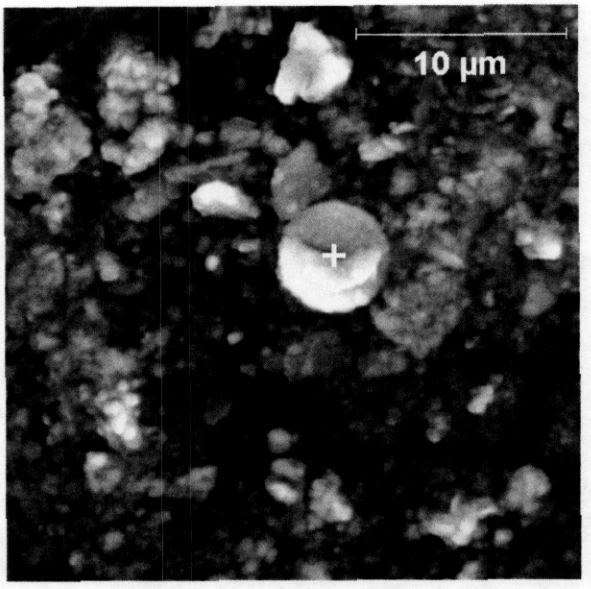

Figure 3.8.3-1. High magnification secondary electron image of small spherule of $Na_2U_2O_7$ from Tank S-112 residue after water and sodium hydroxide washing.

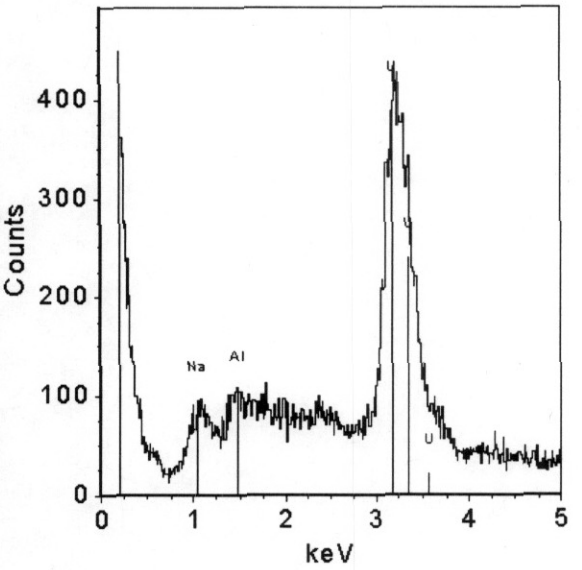

Figure 3.8.3-2. EDS spectrum from spot marked with + in Figure 3.8.3-1, 15Kv, 20% spot.

3.9 OTHER PHASES

3.9.1 Phases

Several other phases have been documented in Tank BY-109 and other waste tanks using the SEM and EDS. A number of these are unique, single particles from single samples. These are not discussed here, since their precise identities and their significance in Hanford tank waste are uncertain. Also, a number of iron-rich particles that are apparently steel and/or rust have also been observed. No pictures of these are included with this report.

In addition, two poorly identified or newly observed phases have been documented. They are described below.

The first of these "novel" phases is a sodium aluminosilicate phase. Particles with this chemical composition were identified by SEM/EDS in BY9Orig, Dil5, Dil6 and Resi 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.

The second novel phase is a distinctive chemical combination of calcium, strontium, chromium and aluminum. This phase has been identified in Tank S-112 water and sodium hydroxide (see Figure 3.9.3-1) digestion residues and probably also occurs in the BY9Resi sample (see Figure 3.9.2-3). 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, with the chemical formula $(Ca,Sr)_3(Cr,Al)_2(OH)_{12}$.

3.9.2 Occurrence in Tank BY-109 Saltcake

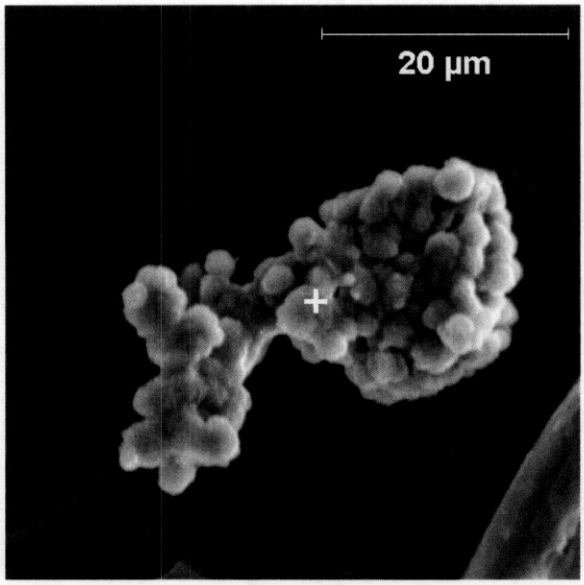

Figure 3.9.2–1. High magnification secondary electron image of agglomerated particles of a sodium aluminosilicate phase in BY9Orig.

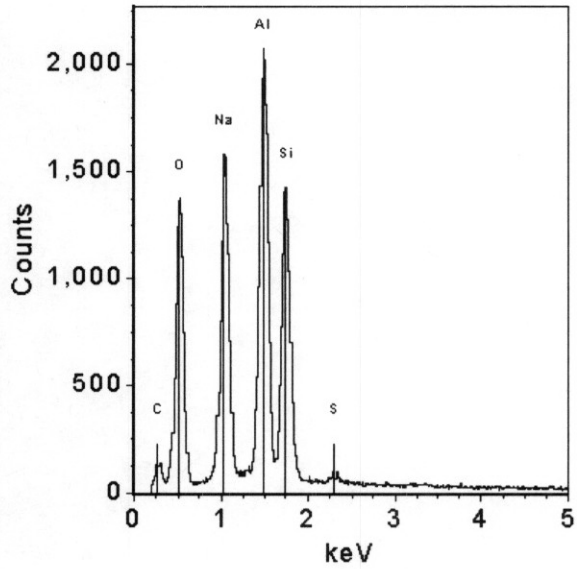

Figure 3.9.2-2. EDS spectrum from spot marked with + in preceding figure, 15Kv, 20% spot.

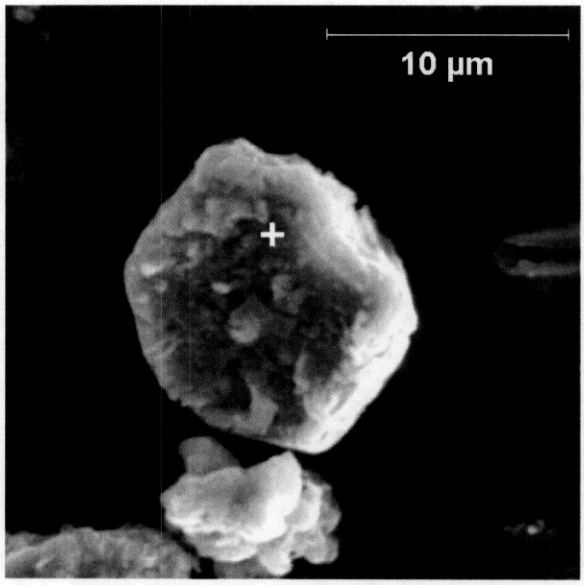

Figure 3.9.2-3. High magnification secondary electron image of $(Ca,Sr)_3(Cr,Al)_2(OH)_{12}$ from BY9Resi.

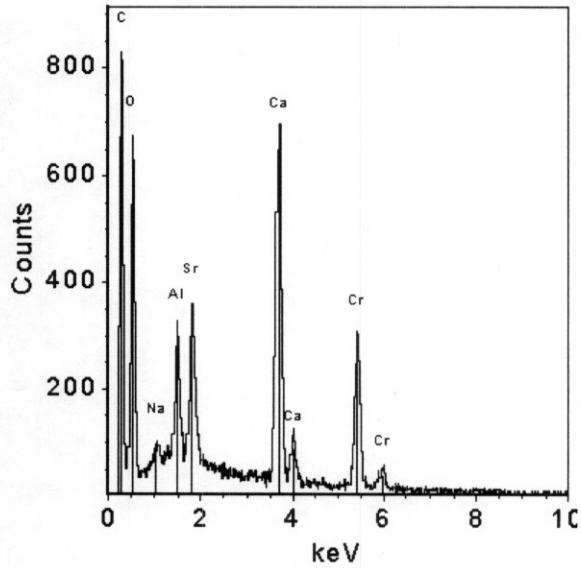

Figure 3.9.2-4. EDS spectrum from spot marked with + in Figure 3.9.2-3, 15Kv, 30% spot.

3.9.3 Other Occurrences

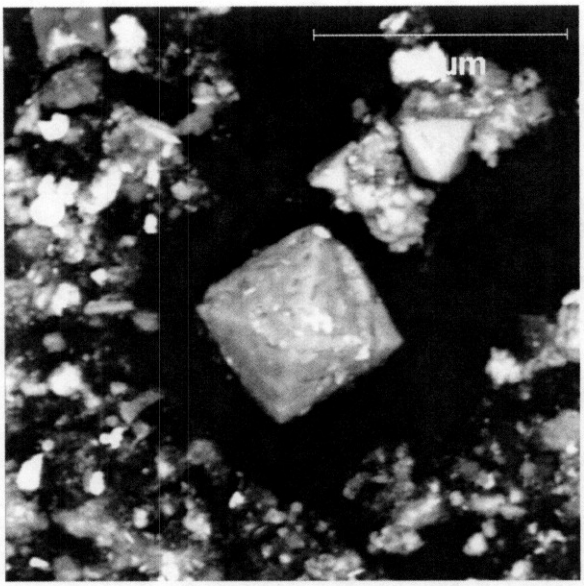

Figure 3.9.3-1. High magnification secondary electron image of water insoluble solids from Tank S-112.

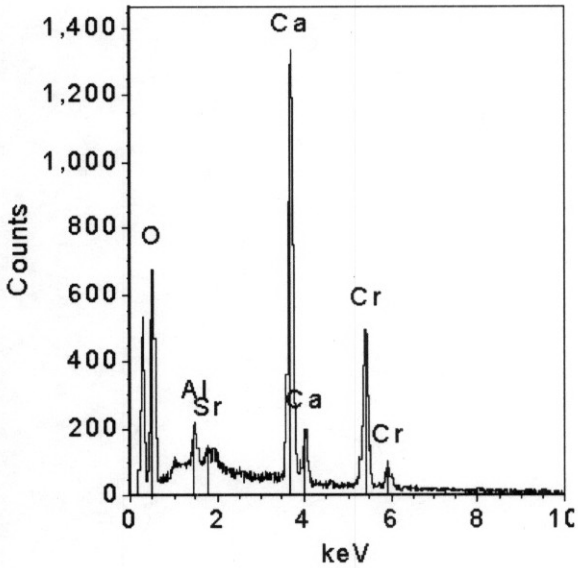

Figure 3.9.3-2. EDS spectrum from spot marked + in Figure 3.9.3-1, 15Kv, 30% spot.

4.0 RESULTS BY PHASE; PHASES NOT IDENTIFIED IN TANK BY-109

4.1 $NaNO_3$

Name: Sodium nitrate	**ICDD PDF#:** 36-1474
Chemical Formula: $NaNO_3$	**Crystal System:** Trigonal
Refractive Index: $n_o = 1.33$, $n_e = 1.59$	**Mineral Name:** Nitratine

$NaNO_3$ crystals belong to the trigonal crystal system, which is not obvious from the crystal habit. Crystals are typically rhombohedral plates ("rectangular" plates with non-90-degree corners). The optic axis of the crystal extends from one corner of the rhombohedron to the body-diagonally opposite corner, and thus has 3-fold symmetry about the optic axis. This also explains why the crystals undergo symmetrical extinction, an unusual property that serves as a distinguishing characteristic of $NaNO_3$ crystals, along with their unusually high birefringence.

$NaNO_3$ was not observed in the Tank BY-109 saltcake samples. This is probably due to the relatively low concentration of nitrate in this saltcake.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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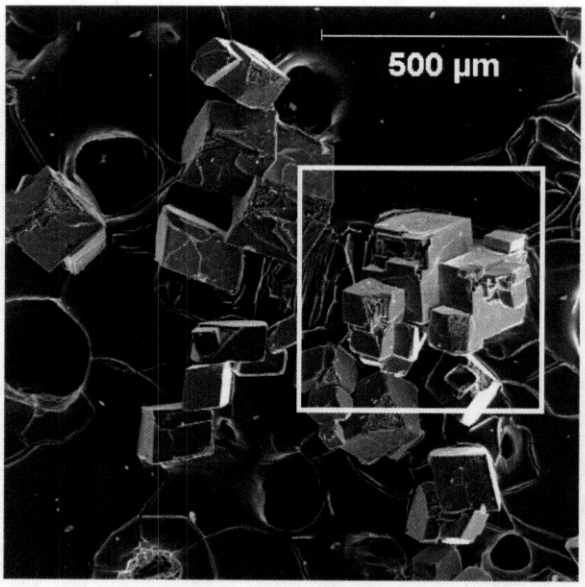

Figure 4.1.1-3. Low magnification secondary electron image of $NaNO_3$ standard.

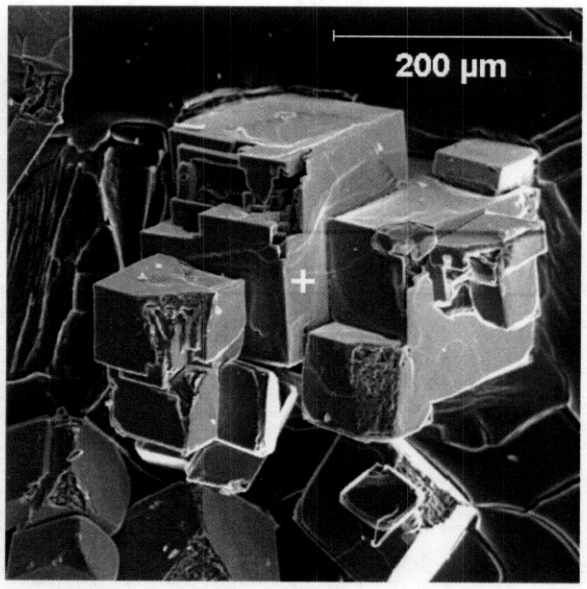

Figure 4.1.1-4. High magnification secondary electron image of $NaNO_3$ standard.

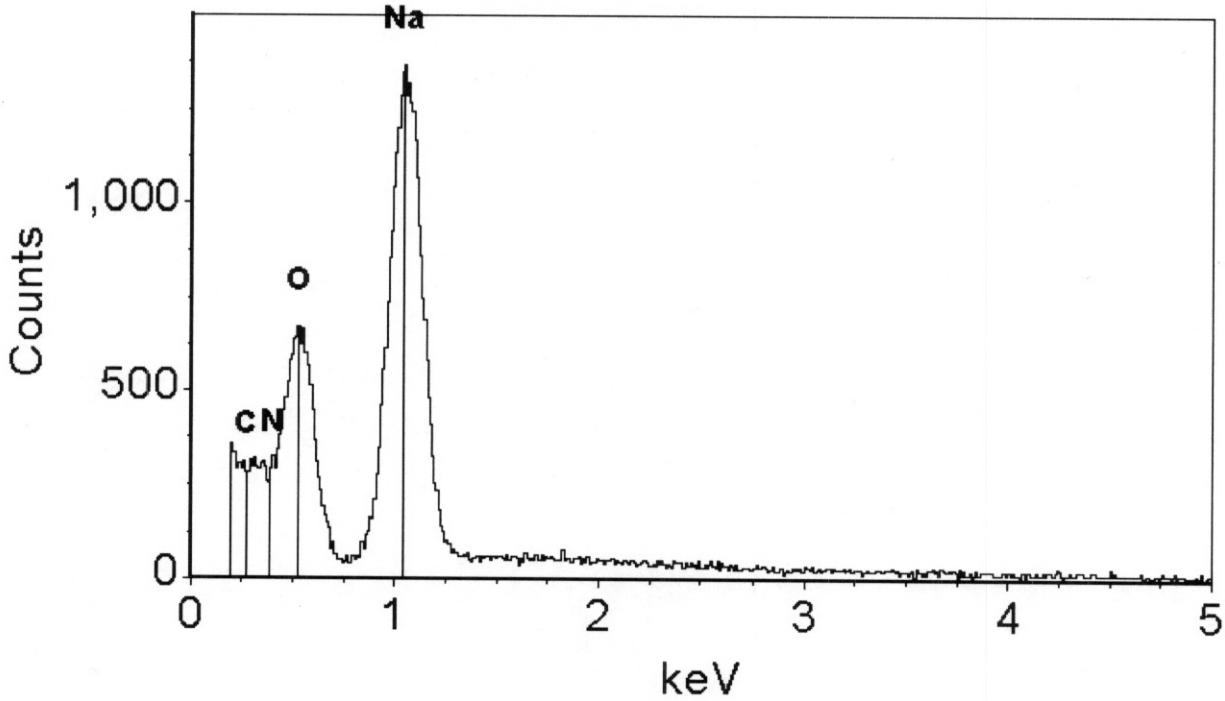

Figure 4.1.1-5. EDS spectrum from spot marked with + in Figure 4.1.1-4, 15Kv, 30% spot.

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4.2 $NaNO_2$

Name: Sodium nitrite	**ICDD PDF#:** 06-0392
Chemical Formula: $NaNO_2$	**Crystal System:** Orthorhombic
Refractive Index: $n_x = 1.35$, $n_y = 1.46$, $n_z = 1.65$	**Mineral Name:** None

Sodium nitrite crystals belong to the orthorhombic crystal *system* (not to be confused with the rhombohedral crystal *habit* exhibited by $NaNO_3$ crystals). Like $NaNO_3$, the crystals have very high birefringence, and show varying contrast depending on orientation relative to the polarizer. Unlike $NaNO_3$, the crystals undergo parallel rather than symmetrical extinction, the surest way of discriminating between the two types of crystals.

Sodium nitrite was not observed in the Tank BY-109 saltcake samples. This is probably due to the relatively low concentration of nitrite in this saltcake.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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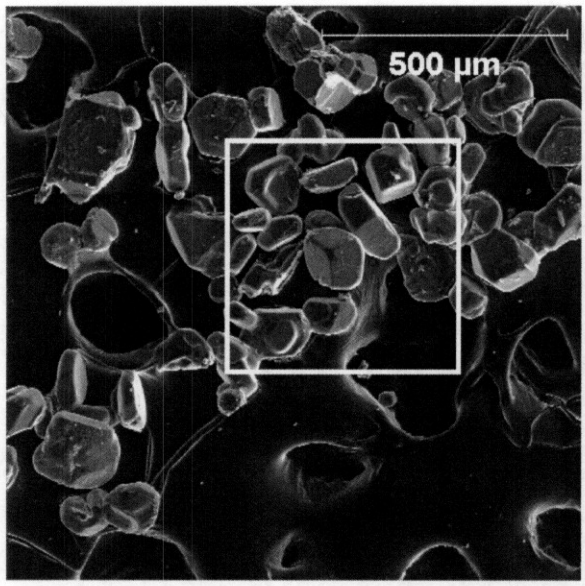

Figure 4.2.1-4. Low magnification secondary electron image of $NaNO_2$ standard.

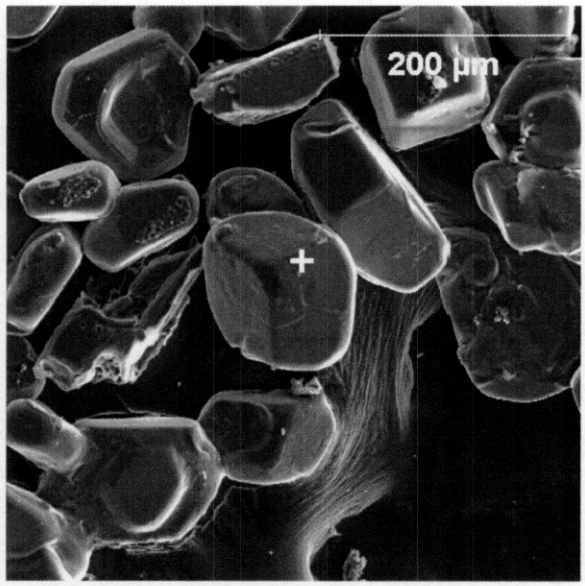

Figure 4.2.1-5. High magnification secondary electron image of $NaNO_2$ standard.

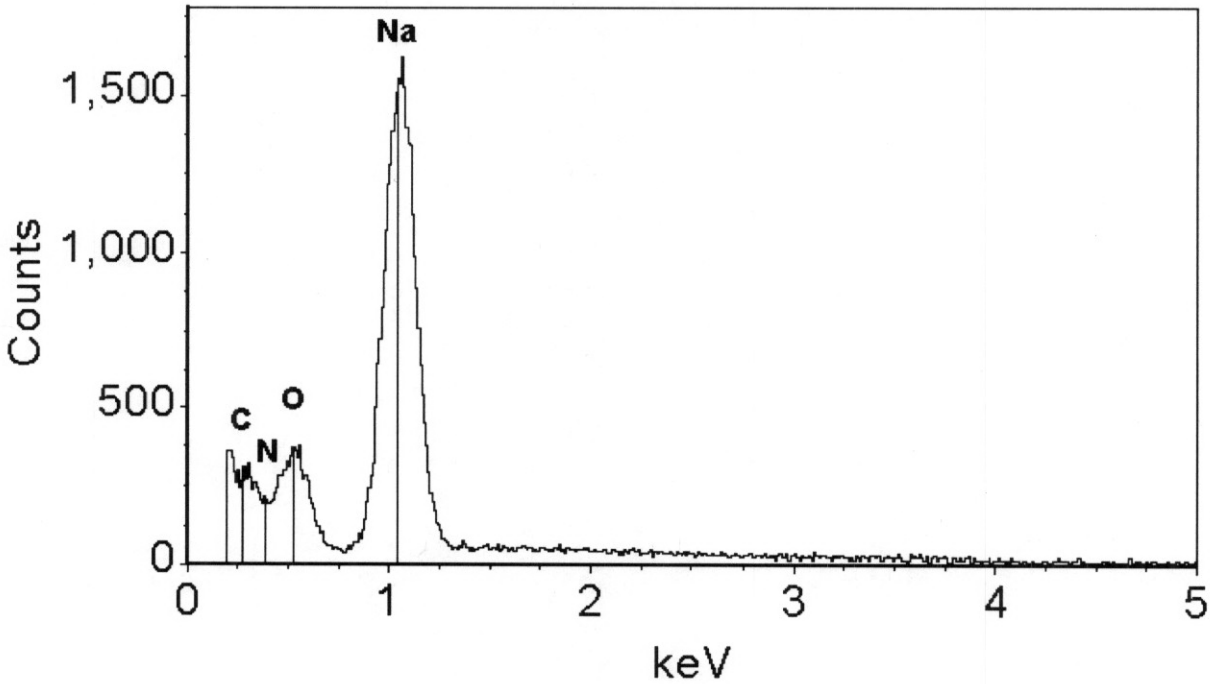

Figure 4.2.1-6. EDS spectrum from spot marked with + in Figure 4.2.1-5, 15Kv, 30% spot.

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4.3 Na_2SO_4 (ANHYDROUS)

Name: Sodium sulfate	**ICDD PDF#:** 37-1465 and 24-1132
Chemical Formula: Na_2SO_4	**Crystal System:** Orthorhombic
Refractive Index: $n_x = 1.471$, $n_y = 1.477$, $n_z = 1.484$	**Mineral Name:** Thenardite

This phase was not observed by PLM, SEM, or XRD in Tank BY-109 saltcake. Due to the elevated fluoride concentration in this saltcake, the sulfate occurs as Na_3FSO_4.

Example PLM and SEM images of sodium sulfate, with EDS and XRD spectra, are shown on the following pages. The diffractogram of the standard appears to be composed of two polymorphs of Na_2SO_4, of which there are reported to be five polymorphs in existence.

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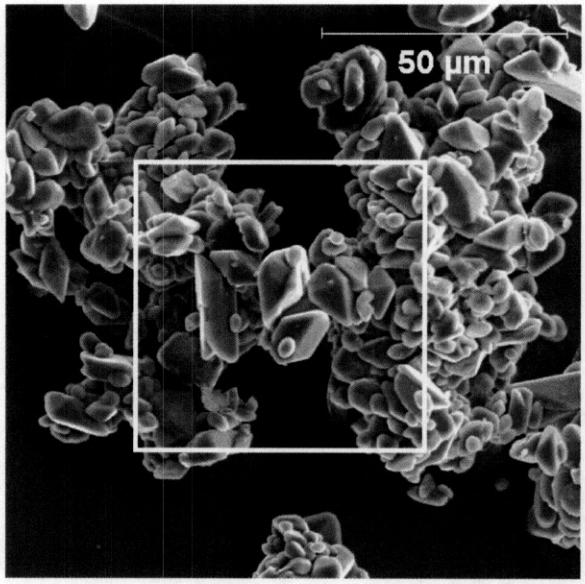

Figure 4.3.1-4. Low magnification secondary electron image of Na_2SO_4 standard.

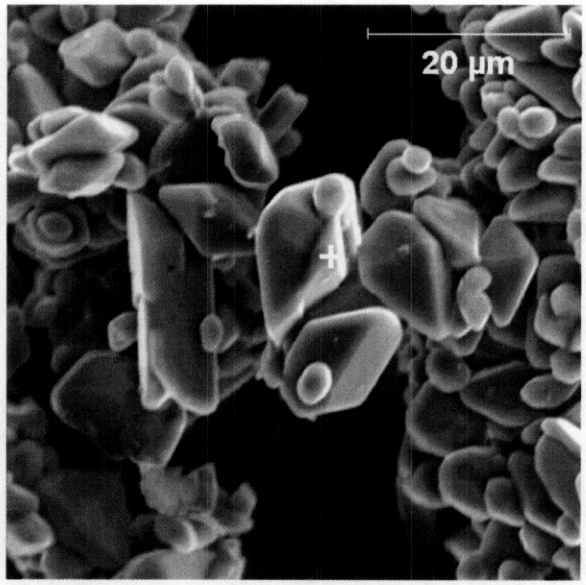

Figure 4.3.1-5. High magnification secondary electron image of Na_2SO_4 standard.

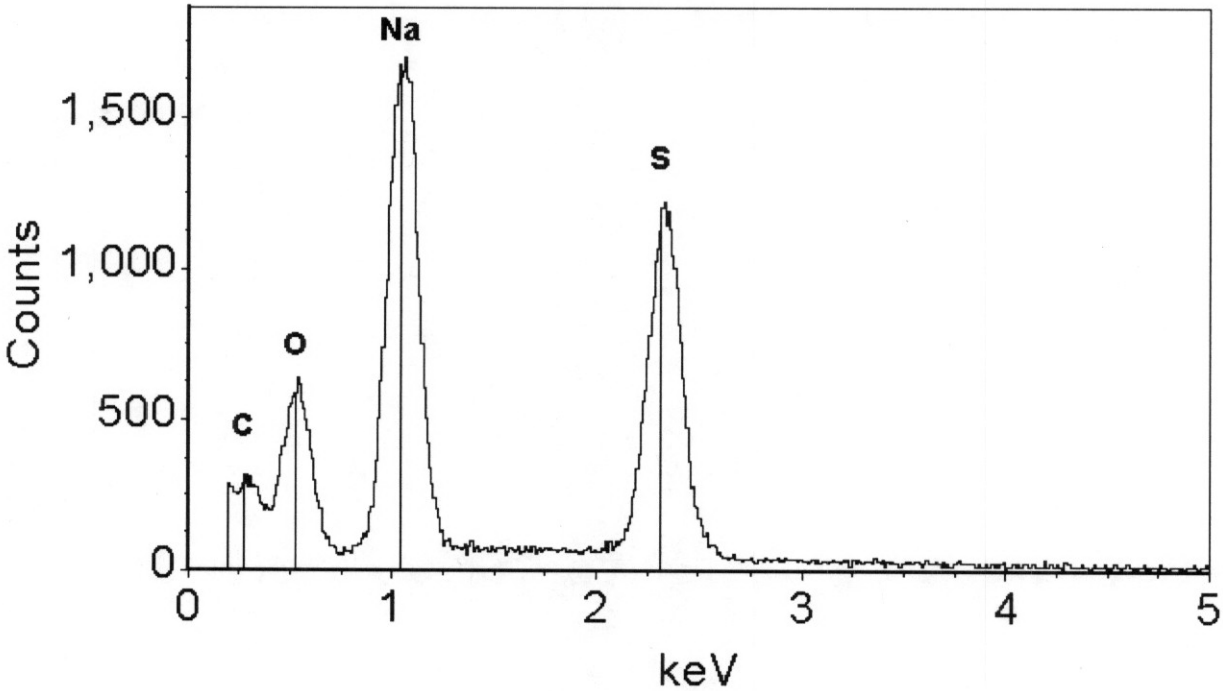

Figure 4.3.1-6. EDS spectrum from spot marked with + in Figure 4.3.1-5, 15Kv, 30% spot.

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4.4 $Na_3PO_4 \cdot 12H_2O$

Name: Sodium phosphate dodecahydrate	**ICDD PDF#:** 10-0189
Chemical Formula: $Na_3PO_4 \cdot 12H_2O \cdot 0.25NaOH$	**Crystal System:** Trigonal
Refractive Index: $n_o = 1.447$, $n_e = 1.452$	**Mineral Name:** None

The sodium phosphate dodecahydrate standard is characterized by long, columnar crystals with aspect ratios (length:width) greater than 10:1. This common crystal habit has been observed in many Hanford Site tank waste samples. Many of the particles on the SEM mount show a pronounced curvature and some surface irregularity that are not visible in the PLM images. The differences in morphology are attributed to partial dehydration of the crystallites in the chamber vacuum. The *CRC Handbook of Chemistry and Physics*, 65th Edition (Weast 1985) lists the dehydration temperature of sodium phosphate dodecahydrate at atmospheric pressure as 73.3 °C.

Sodium phosphate dodecahydrate is often shown with a quarter molecule of associated NaOH, with the chemical formula $Na_3PO_4 \cdot 12H_2O \cdot 0.25NaOH$. For the purposes of this report, the chemical formula of sodium phosphate dodecahydrate is shown as $Na_3PO_4 \cdot 12H_2O$.

In the EDS spectra from this standard, the carbon is due to the carbon coating. A minor amount of sulfur is observed in the first spectrum.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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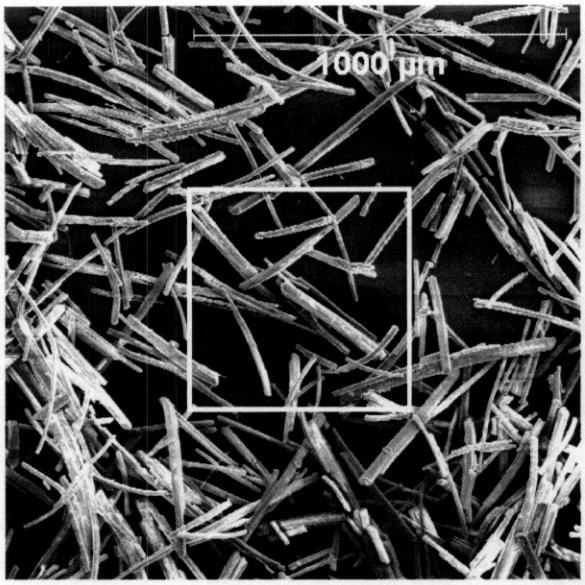

Figure 4.4.1-4. Low magnification secondary electron image of $Na_3PO_4 \cdot 12H_2O$ standard.

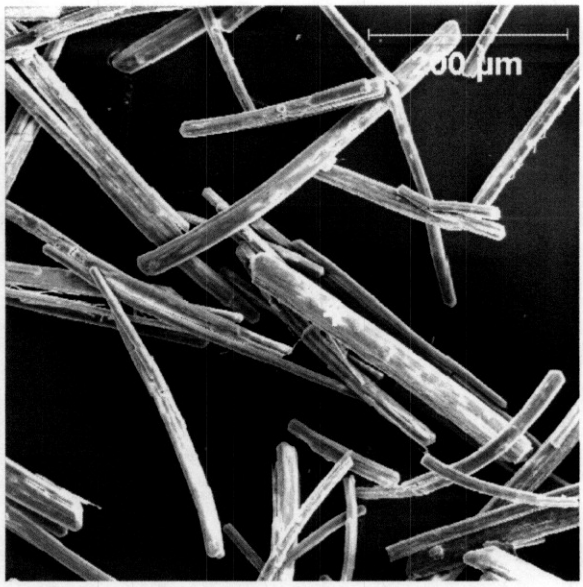

Figure 4.4.1-5. High magnification secondary electron image of $Na_3PO_4 \cdot 12H_2O$ standard.

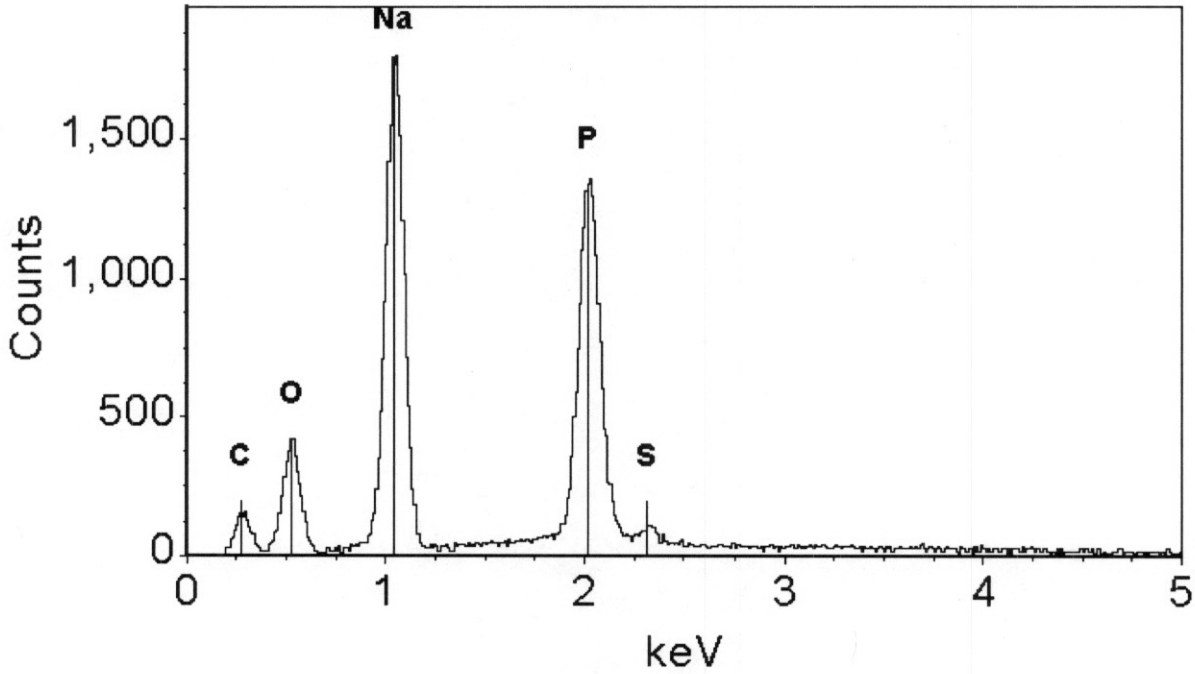

Figure 4.4.1-6. EDS spectrum from spot marked with + in Figure 4.4.1-5, 15Kv, 30% spot.

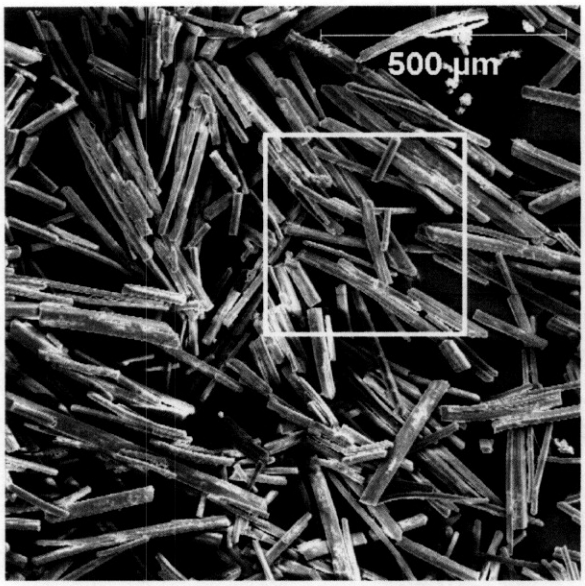

Figure 4.4.1-7. Low magnification secondary electron image of $Na_3PO_4 \cdot 12H_2O$ standard.

Figure 4.4.1-8. High magnification secondary electron image of $Na_3PO_4 \cdot 12H_2O$ standard.

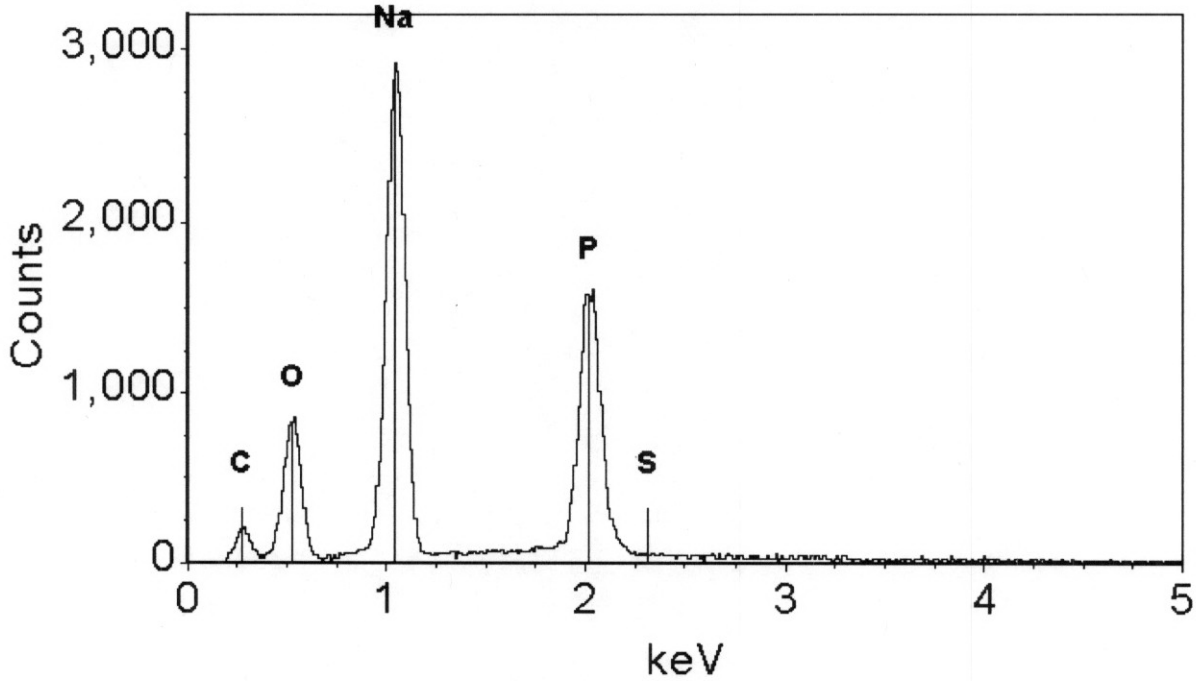

Figure 4.4.1-9. EDS spectrum from spot marked with + in Figure 4.4.1-8, 15Kv, 30% spot.

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4.5 $NaAl(OH)_4$

Name: Sodium aluminate	**ICDD PDF#:** 44-0430 and 41-0638
Chemical Formula: $NaAl(OH)_4$	**Crystal System:** Orthorhombic
Refractive Index: $n_x = 1.566$, $n_y = 1.575$, $n_z = 1.580$	**Mineral Name:** None

Two crystalline forms of sodium aluminate were described as early as 1930 (Fricke and Jucaitis 1930). They were described in the reference as (1) small prisms that tend to form rosettes (see Figure 4.5.1-2), and (2) thin quadratic platelets (see Figure 4.5.1-3). Both forms have been observed in Hanford tank waste and simulants. Slow conversion from the needle form to the quadratic plate form has also been observed in simulants.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages. The diffractogram of the standard best matches a combination of two separate reference patterns of sodium aluminum oxide hydrate. Whether these patterns correspond to the prism and platelet forms described above is unknown. The diffractogram also contains a major contribution from the contaminant $NaNO_3$ precipitated from the supernatant liquid.

Sodium aluminate is also known as sodium aluminum oxide hydrate, as it is shown on the XRD spectra.

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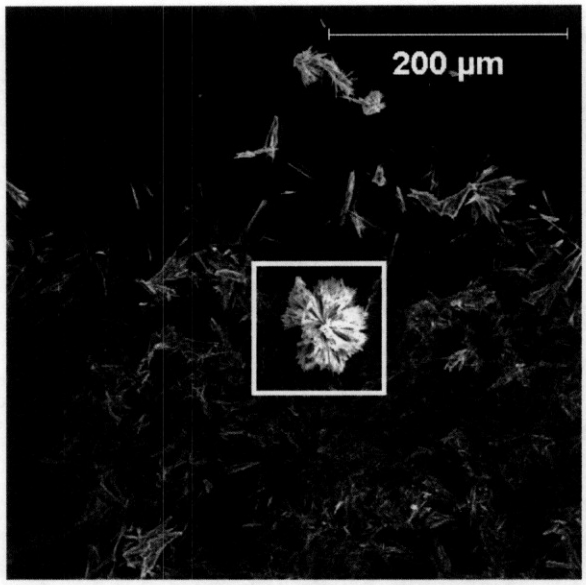

Figure 4.5.1-4. Low magnification secondary electron image of $NaAl(OH)_4$ standard.

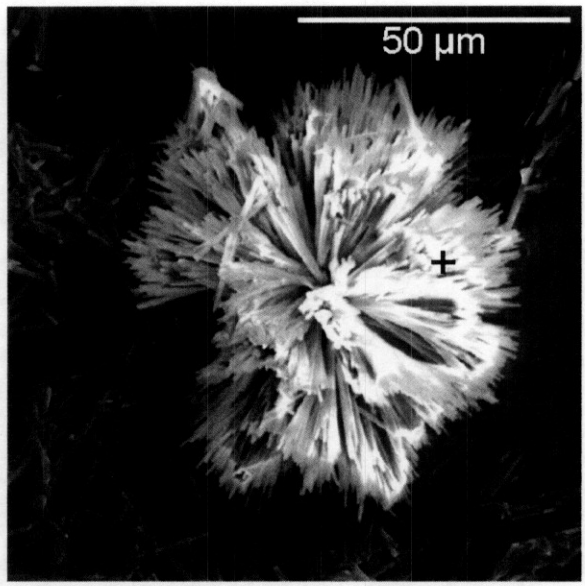

Figure 4.5.1-5. High magnification secondary electron image of $NaAl(OH)_4$ standard.

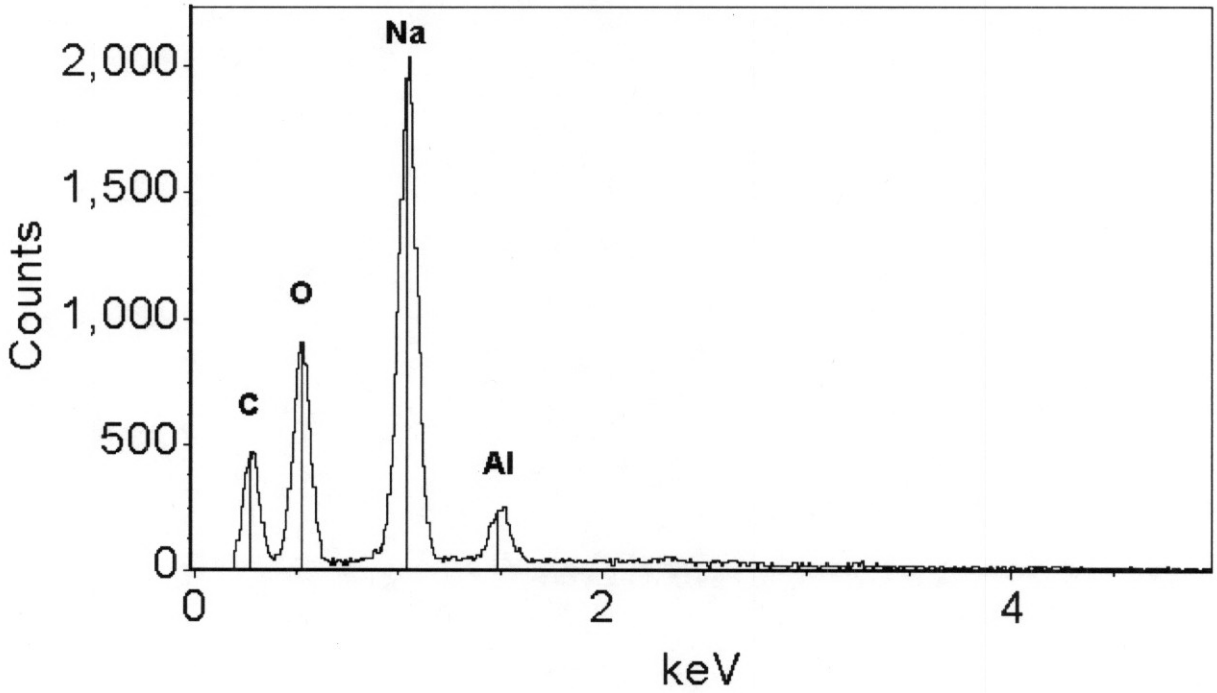

Figure 4.5.1-6. EDS spectrum from spot marked with + in Figure 4.5.1-5, 15Kv, 30% spot.

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4.6 $Na_3NO_3SO_4 \cdot H_2O$

Name: Sodium nitrate sulfate hydrate	**ICDD PDF#:** 23-1408
Chemical Formula: $NA_3NO_3SO_4 \cdot H_2O$	**Crystal System:** Monoclinic
Refractive Index: $n_x = 1.391$, $n_y = 1.481$, $n_z = 1.486$	**Mineral Name:** Darapskite

Sodium nitrate sulfate forms an incongruent double salt of the formula $Na_3NO_3SO_4 \cdot H_2O$. (The $Na:NO_3:SO_4$ mole ratios are different in the solid phase than in the liquid phase in equilibrium with the pure solid. When a small amount of water is added to the dry salt, sodium nitrate preferentially dissolves, converting the solid phase to sodium sulfate.) Crystals are monoclinic with moderate birefringence, which leads to very beautiful low-order interference colors when viewed between crossed polars.

Sodium nitrate sulfate was not identified as a solid phase in Tank BY-109.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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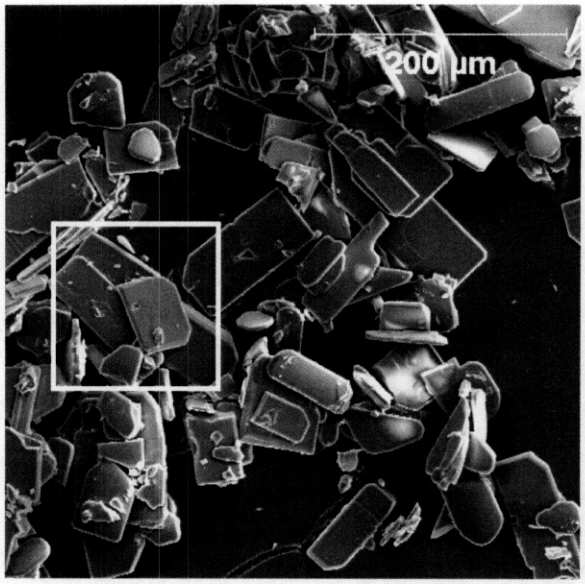

Figure 4.6.1-4. Low magnification secondary electron image of $Na_3NO_3SO_4 \cdot H_2O$ standard.

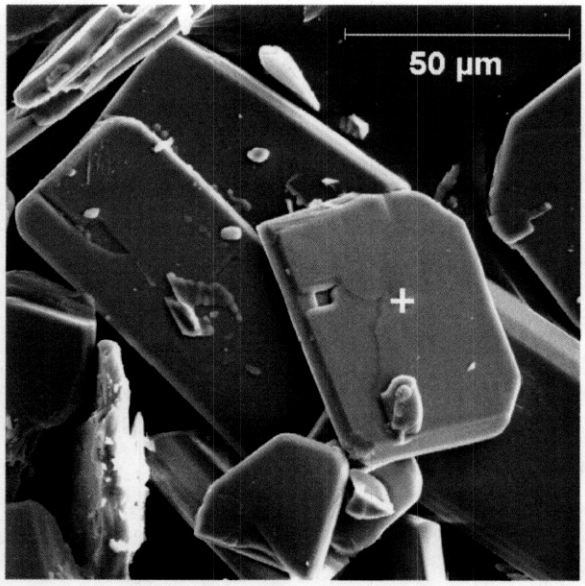

Figure 4.6.1-5. High magnification secondary electron image of $Na_3NO_3SO_4 \cdot H_2O$ standard.

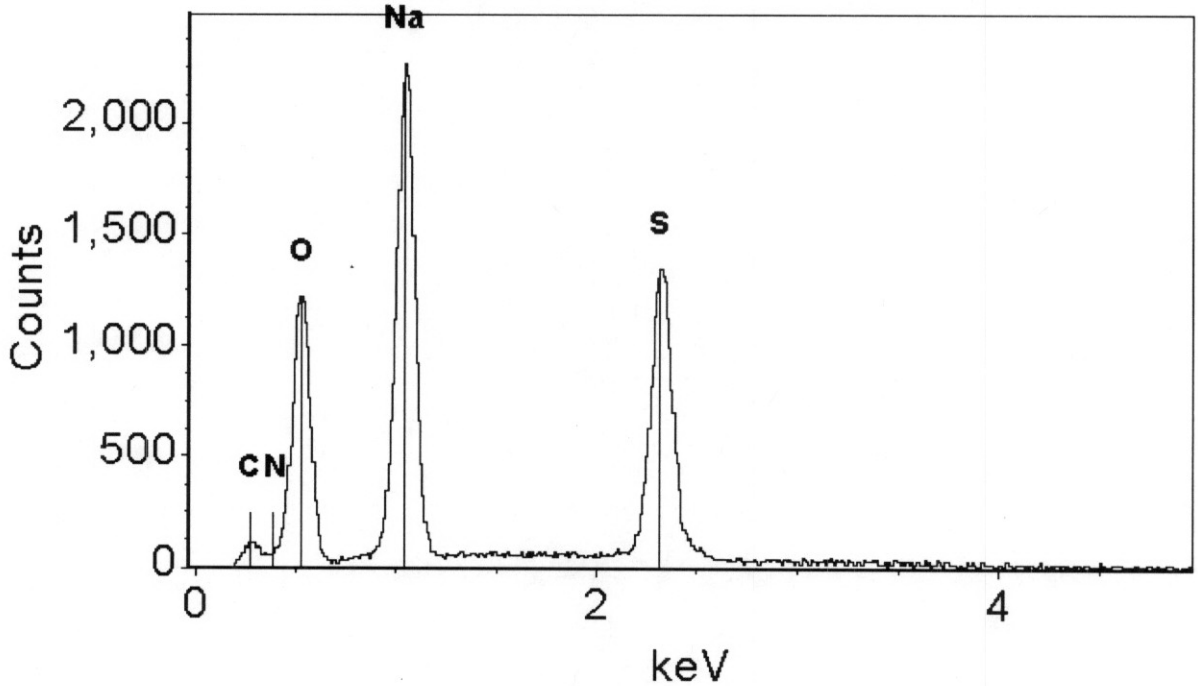

Figure 4.6.1-6. EDS spectrum from spot marked with + in Figure 4.6.1-5, 15Kv, 30% spot.

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4.7 $Na_6CO_3(SO_4)_2$

Name: Sodium carbonate sulfate	**ICDD PDF#:** 24-1139
Chemical Formula: $Na_6CO_3(SO_4)_2$	**Crystal System:** Orthorhombic
Refractive Index: $n_x = 1.450$, $n_y = 1.490$, $n_z = 1.492$	**Mineral Name:** Burkeite

The standard may not be a good representation of burkeite. The major phase present in the diffractogram of the standard is sodium sulfate, Na_2SO_4, which shows strong preferential orientation of the crystals. The sodium carbonate sulfate, $Na_6CO_3(SO_4)_2$, shows up as a minor phase in the diffractogram.

Sodium carbonate sulfate was not identified as a solid phase in Tank BY-109. Although there have not been many definite examples of burkeite found in actual tank waste samples, it is assumed to be a common phase, based on the chemistry of the double salt.

Example PLM and SEM images, with EDS and XRD spectra, are shown on the following pages.

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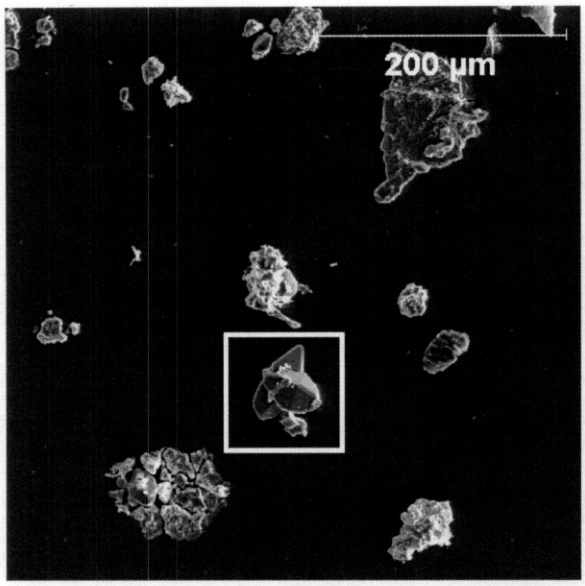

Figure 4.7.1-4. Low magnification secondary electron image of $Na_6CO_3(SO_4)_2$ standard.

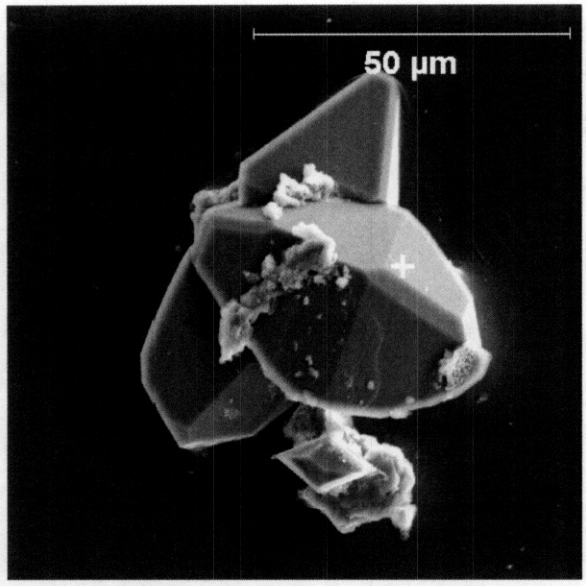

Figure 4.7.1-5. High magnification secondary electron image of $Na_6CO_3(SO_4)_2$ standard.

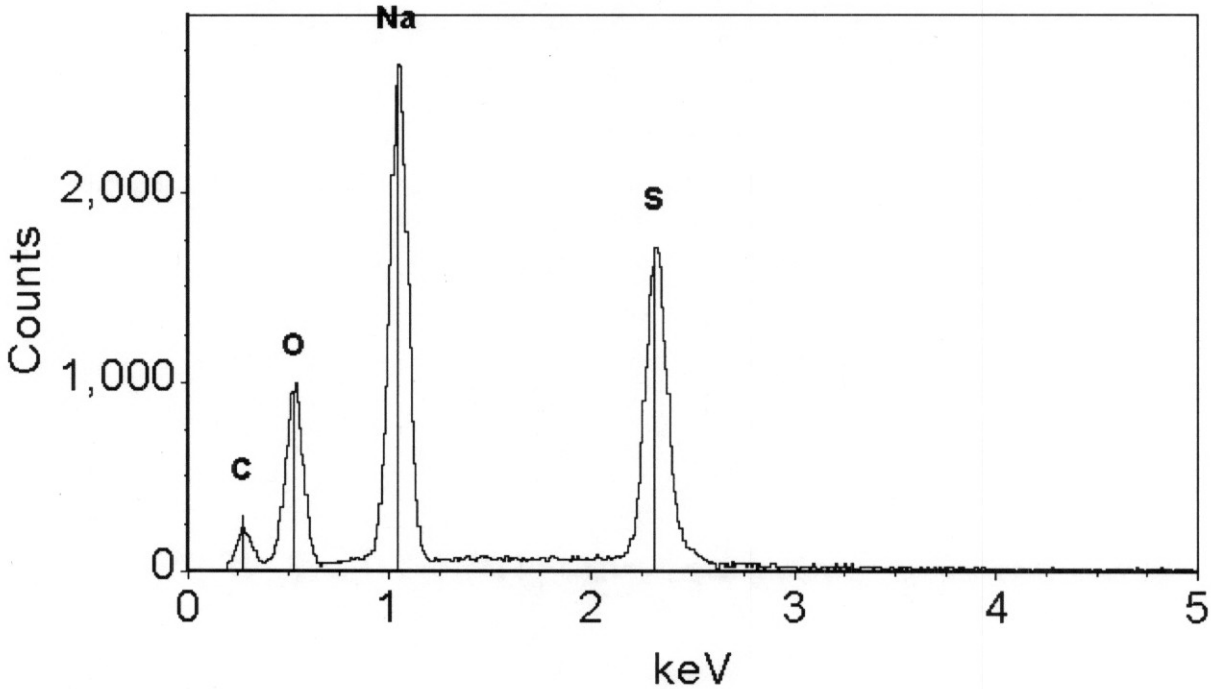

Figure 4.7.1-6. EDS spectrum from spot marked with + in Figure 4.7.1-5, 15Kv, 30% spot.

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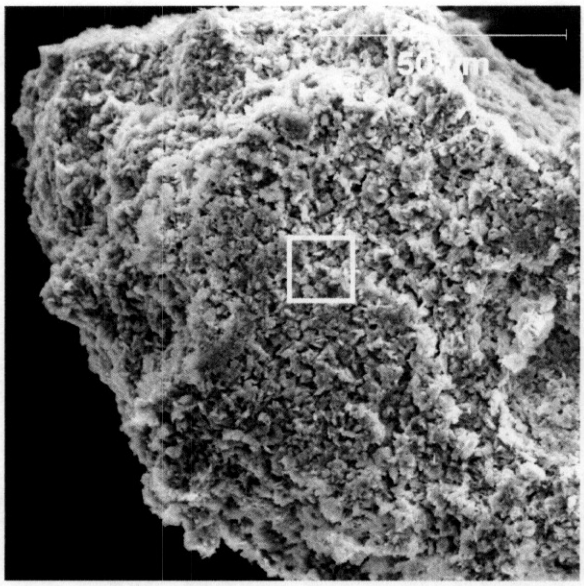

Figure 4.8.1-3. Low magnification secondary electron image of AlO(OH) standard.

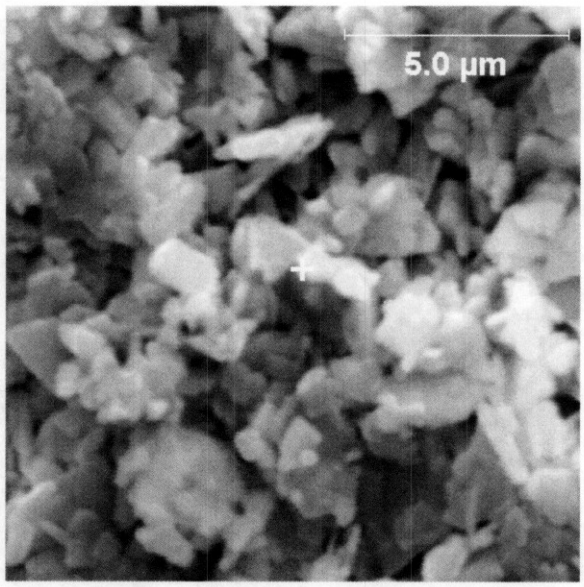

Figure 4.8.1-4. High magnification secondary electron image of AlO(OH) standard.

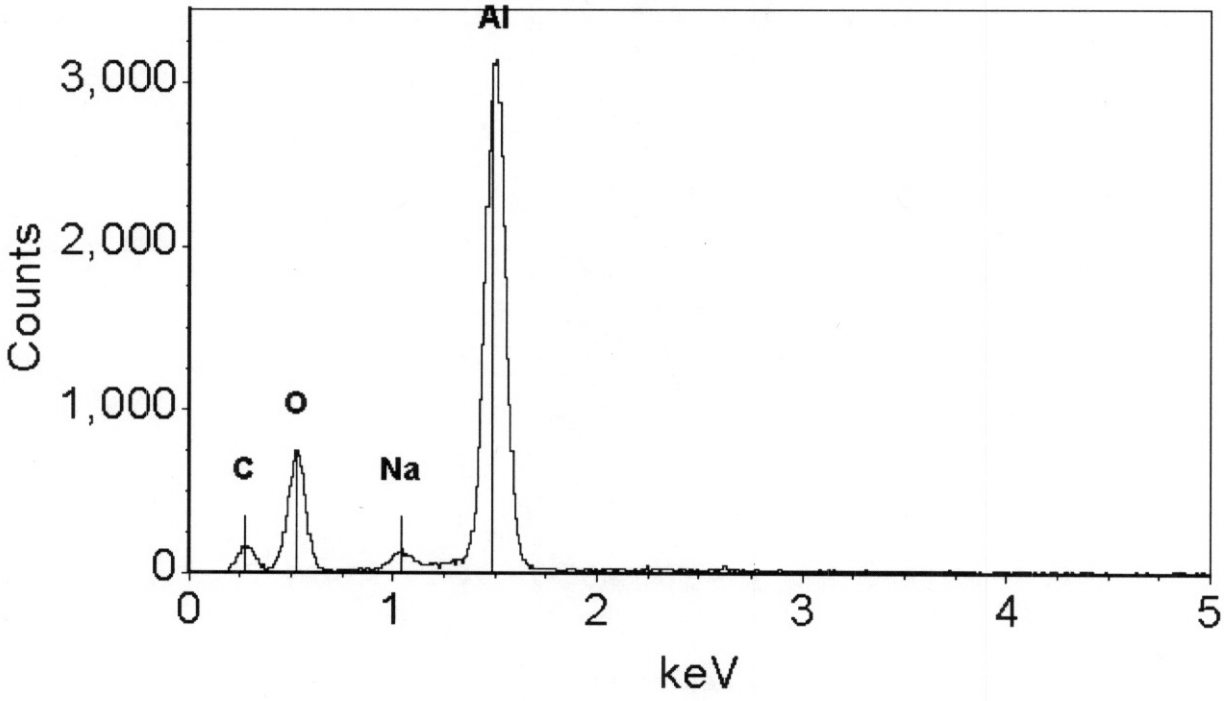

Figure 4.8.1-5. EDS spectrum from spot marked with + in Figure 4.8.1-4, 15Kv, 30% spot.

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5.0 RESULTS BY SAMPLE

Table 5-1 summarizes the observations, by phase, for each Tank BY-109 saltcake sample. PLM and SEM images for each sample are shown in the following sections, along with EDS and XRD spectra.

Table 5-1. Solids Identified in Tank BY-109 Saltcake.

	$Na_7F(PO_4)_2 \cdot 19H_2O$	Na_3FSO_4	$Na_2C_2O_4$	NaF	Al oxide/ hydroxide	Na_3AlF_6 (Cryolite)	Aluminosilicate	Other
Orig	P S X	P S X	P S X	X			P S	Uranium-rich phase, $Na_2CO_3 \cdot H_2O$
Dil1	P X	P S X	P S X	X				Uranium-rich phase
Dil2	X	P S X	P S X	S X				
Dil3		P S X	P S X	X				Uranium-rich phase
Dil4			P S	S				Uranium-rich phase
Dil5				S	S		S	Cr-rich phase, Bi-Pd-rich phase
Dil6				X	S		S	Uranium-rich phase
Resi					S X[1]		S	Uranium-rich phase, Sr/Ca-rich phase
Evap2	P S X		P S X			S X		
Evap3		P			S X[2]	S X		Bayerite
Evap4	P S X	P S X	P S X					
Evap5	P X	P S X	P S X			S		
Evap6	P S X	S X	P S X	X				

[1] $Al(OH)_3$ gibbsite.
[2] $Al(OH)_3$ bayerite.

P = PLM images (polarized light microscopy).
S = SEM/EDS images (scanning electron microscope images and energy dispersive spectrometer spectra).
X = XRD (x-ray diffraction).

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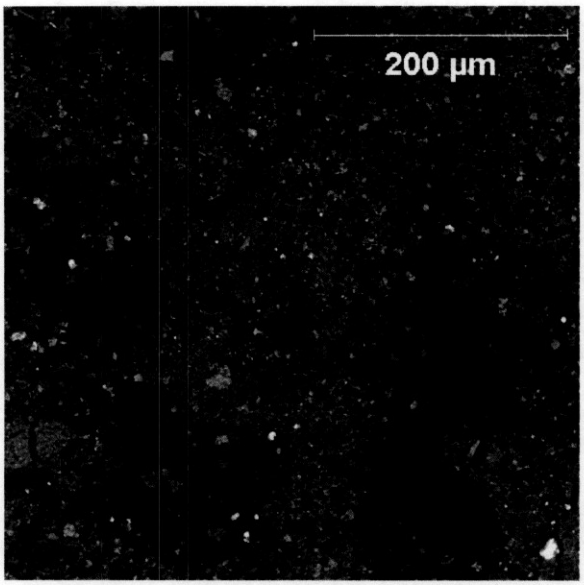

Figure 5.6-3. Backscattered electron image of another field on BY9Dil5 shows more frequent, bright, high atomic weight particles (mostly uranium-rich) than were observed in the original sample (Figure 5.1-3).

XRD analysis was not done on the BY9Dil5 sample. Due to the expected high background, no new phases were expected to appear.

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6.0 ACKNOWLEDGEMENTS

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Mr. John Smith prepared samples for both SEM and XRD analysis, and assisted in the data collection for both instruments. Mr. Wayne Edmonson performed the hotcell dissolution and evaporation work, and prepared samples for PLM analysis.

Ms. Leona Aamot organized and edited the document.

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