

## Characterization of Samples from the Effluent Treatment Facility Evaporator Waste Concentrate Tank

by

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## **Characterization of Samples from the Effluent Treatment Facility Evaporator Waste Concentrate Tank (U)**

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### Summary

During October 1997, the ETF Evaporator Waste Concentrate Tank No.2 was discovered to contain a significant accumulation of solid deposits. SRTC performed destructive and non-destructive examination of solid samples from the tank. The results of these tests indicate that the solids contain mixtures of sodium oxalate (65%), the sulfide enclathrated sodium aluminosilicate (30%), and iron oxide (5%).

### Introduction

The Effluent Treatment Facility (ETF) collects, treats, and discharges radioactive wastewater from various High Level Waste and Nuclear Material Storage and Stabilization facilities at the Savannah River Site. The ETF treatment involves a series of processes which include pH adjustment, sub-micron filtration, ion exchange and evaporation. Decontaminated liquids are determined to meet the regulatory requirements proceed to discharge at a permitted outfall. Concentrated liquids containing the majority of the radionuclide components transfer to Saltstone for final disposal in grout.

During a recent liquid transfer from the ETF Waste Concentrate Tank No.2 to Saltstone, a discrepancy in the total volume of liquid occurred. The Waste Concentrate Tank has a capacity of 1850 gallons and is 84 inches tall. Once the tank reaches capacity, operators pH adjust the liquid. Inspection of the Waste Concentrate Tank No. 2 by the High Level Waste Surveillance, Monitoring and Remote Technology group revealed significant solid deposits. The Savannah River Technology Center was requested<sup>1</sup> to analyze a sample of these solids to determine the chemical constituents.

### Experimental

Approximately 52.4 grams (ETF-001) were collected from the tank and transferred to SRTC for chemical analysis. The sample consisted of a dozen or more large pieces. The pieces appeared reddish in color and were moist. Personnel removed representative samples and submitted for

Scanning Electron Microscopy<sup>2</sup> and X-ray Diffraction analysis.<sup>3</sup> Following a sodium peroxide fusion dissolution,<sup>4</sup> personnel analyzed the liquid for soluble metals by Inductively Coupled Plasma - Emission Spectroscopy.<sup>5</sup>

Laboratory notebook WSRC-NB-97-62 contains data obtained during these tests and the procedures used. Personnel used routine analytical protocol for the samples in this report.<sup>6</sup>

Experimental Results

Figure 1 shows the X-ray Diffraction spectrum obtained for a ground sample of the solids from the ETF Waste Concentrate Tank No. 2. The spectral peaks correlate with those spectral peaks from reference spectra of sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ), sodium aluminosulfate sulfide ( $\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}\text{S}\cdot 4\text{H}_2\text{O}$ ) and hermatite (iron oxide,  $\text{Fe}_2\text{O}_3$ ). Comparison of these reference spectra with the spectrum of the sample shows excellent agreement in both intensity and diffraction angle,  $2\theta$ . All observed diffraction peaks correlate to the reference spectra. The sodium aluminosulfate observed in the EFT Waste Concentrate Tank No. 2 comes from the same chemical family as the material recently discovered in the 242-16H Evaporator Gravity Drain Line.<sup>7</sup>

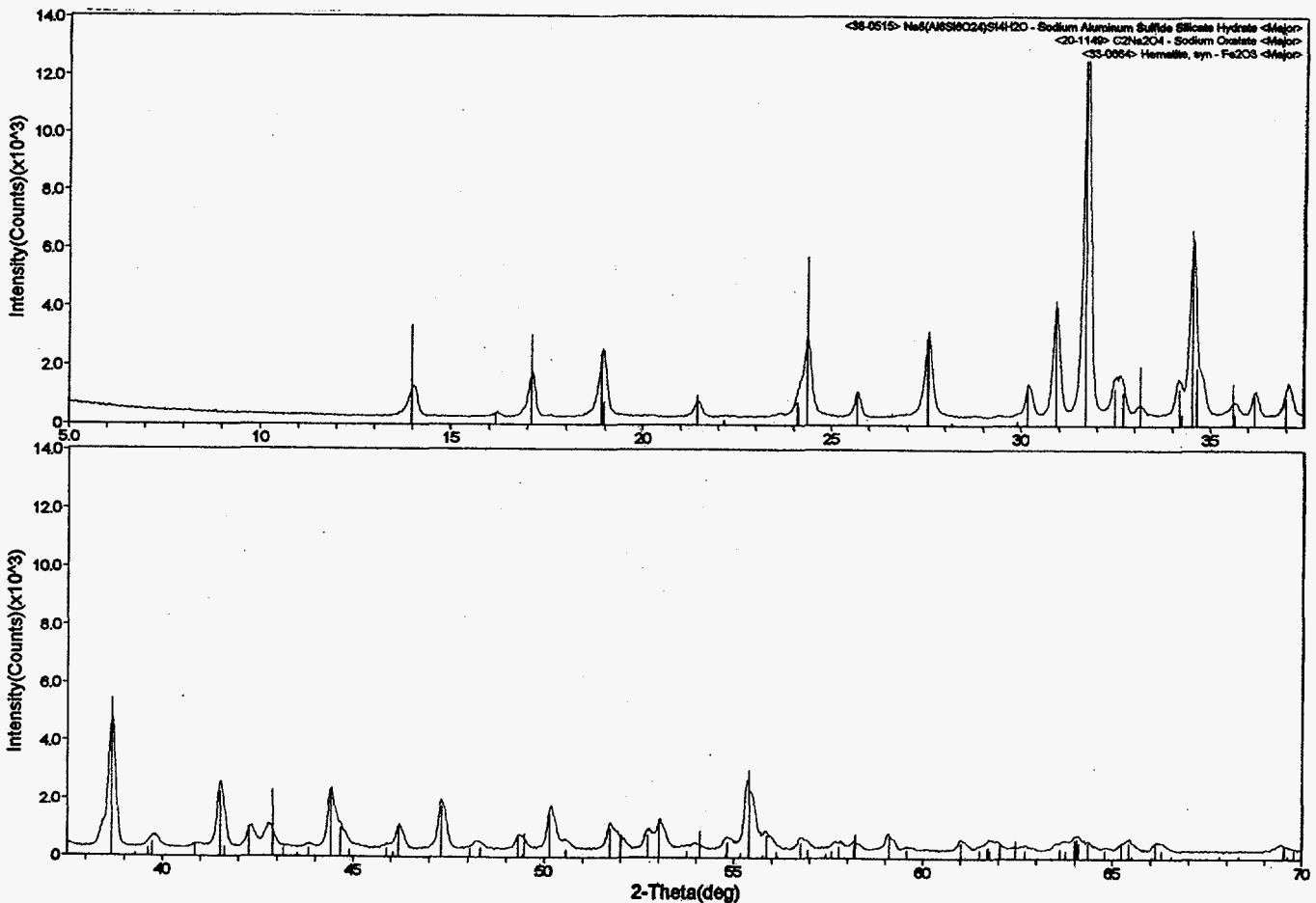


Figure 1. X-ray Diffraction Spectrum of ETF Waste Concentrate Solids

Although the reference spectrum for the sulfide enclathrated sodium aluminosilicate best matched the sample spectrum, the presence of this compound appears unlikely given the lack of sulfide in the ETF process. The peak pattern observed in the X-ray spectra primarily reflects the structure of the aluminosilicate anion. Only slight spectral differences exist between the sulfide, hydroxy, or nitrate compounds. The moisture content of this sample might cause the nitrate or hydroxy spectra to appear more like that of the sulfide.

Figures 2, 3, and 4 show the Scanning Electron Microscopy (SEM) spectra obtained from different regions of the sample (ETF-001). In Figure 2, a single major peak exists for sodium with minor peaks for aluminum, silicon, iron, and nickel. This spectrum resembles that of sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) with very low scattering cross sections for the carbon and oxygen atoms. The contained Scanning Electron Microscopy unit is unable to detect these low atomic weight elements.

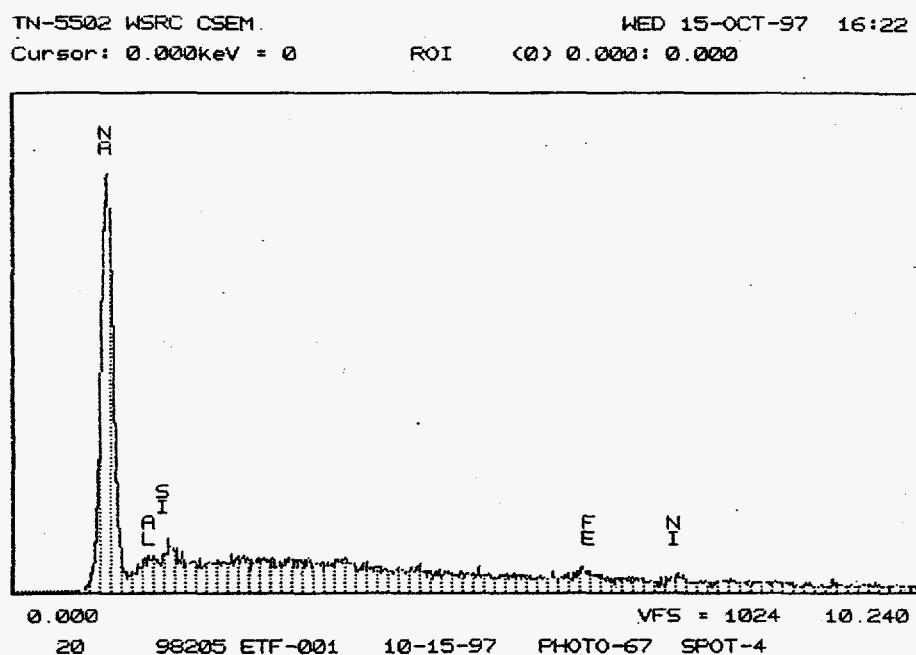


Figure 2. SEM Spectrum of Oxalate-Rich Portion of ETF Solids

Similarly, Figure 3 shows an SEM spectrum very rich in iron indicative of the iron oxide located in the X-ray Diffraction spectrum and consistent with the samples reddish color. Figure 4 shows the SEM spectrum of a portion of the ETF Waste Concentrate sample that is consistent with the sodium aluminosilicate previously found in the H-Area Gravity Drain Line. Minor peaks of other elements occur in each of the spectra corresponding to calcium and nickel. Additional spectra show mixtures of all of the above mentioned materials and stainless steel.

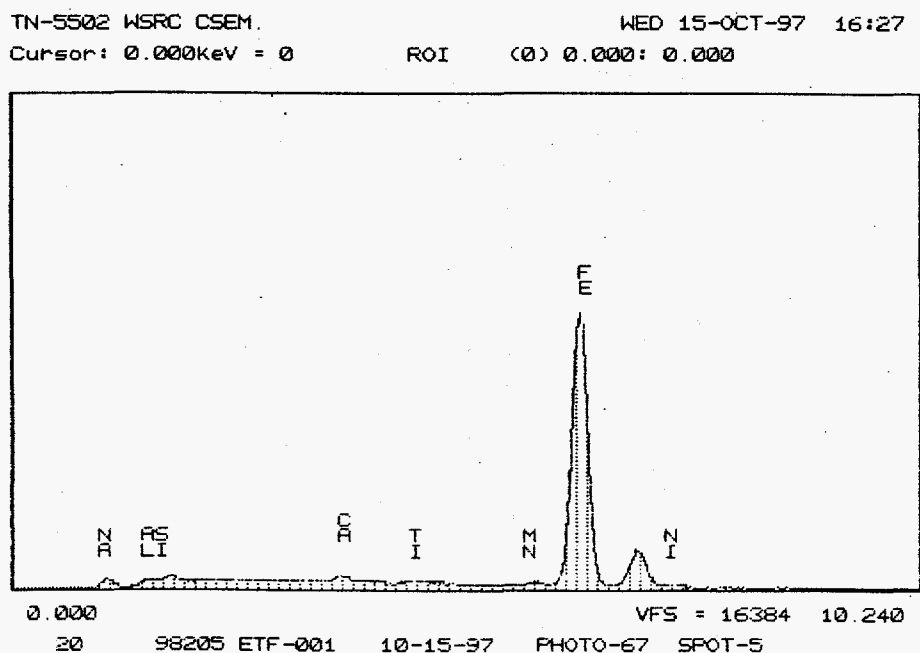


Figure 3. SEM Spectrum of Iron Oxide-Rich Portion of ETF Solids



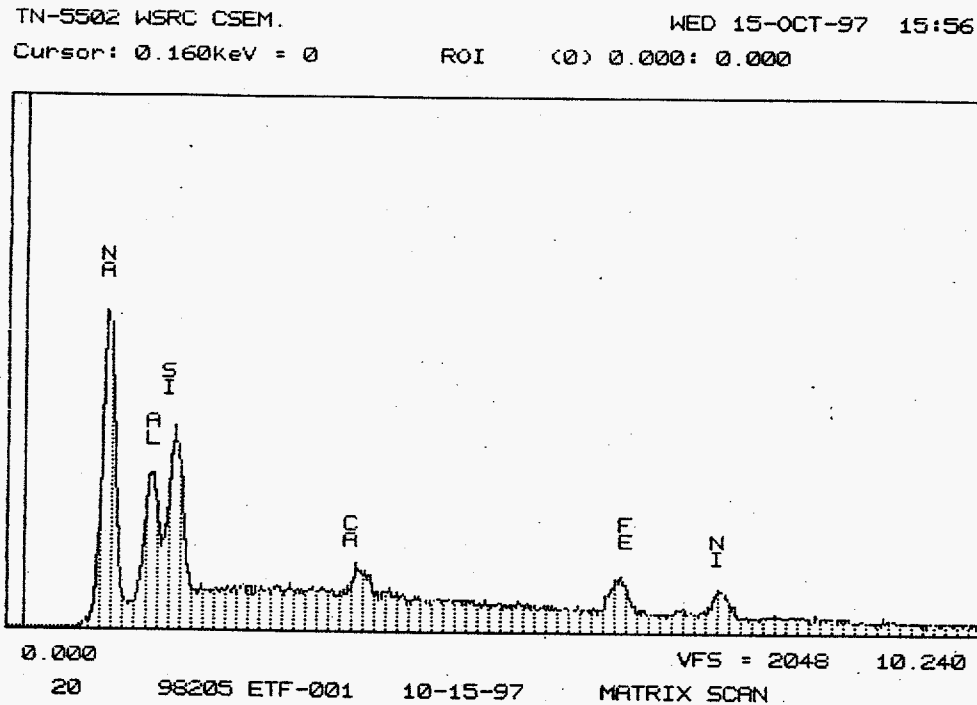


Figure 4. SEM Spectrum of Sodium Aluminosilicate-Rich Portion of the ETF Solids

Personnel performed a sodium peroxide fusion dissolution on a representative portion of solids. This technique involves heating the sample with a quantity of sodium peroxide followed by dissolution in nitric acid. Liquid samples were submitted for analysis by Inductively Coupled Plasma - Emission Spectroscopy (ICP-ES). Table 1 lists the results of these analyses.

Analysis of the ICP-ES data reveals good agreement with the non-destructive analyses. Aluminum and silicon exist in equal molar quantities suggesting agreement with the sodium aluminosilicate molecular formula, i.e., 1:1. Iron is detected in the sample at a percentage roughly in relation to the ratio of aluminosilicate to iron oxide (i.e., Si:Fe = 1:1) observed in the X-ray Diffraction analyses. Magnesium, molybdenum and zinc are the only other constituents

observed in the ICP-ES scan. Sodium, normally detected in the routine ICP-ES scan, is blinded by the sodium peroxide used in the dissolution technique.

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Table 1. ICP-ES Data from ETF Waste Concentrate Tank No.2 Solids

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| Element | Concentration (Wt%) |
|---------|---------------------|
| Al      | 4.16                |
| B       | <0.013              |
| Ba      | <0.0056             |
| Ca      | 0.323               |
| Cd      | <0.003              |
| Co      | <0.008              |
| Cr      | <0.009              |
| Cu      | <0.012              |
| Fe      | 0.6082              |
| La      | <0.023              |
| Li      | <0.008              |
| Mg      | 0.0461              |
| Mn      | 0.018               |
| Mo      | <0.010              |
| Ni      | <0.017              |
| P       | <0.027              |
| Pb      | <0.045              |
| Si      | 4.419               |
| Sn      | <0.014              |
| Sr      | <0.003              |
| Ti      | <0.009              |
| V       | <0.012              |
| Zn      | 0.006               |

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### Conclusions

Samples of solid deposits from the Effluent Treatment Facility Waste Concentrate Tank No. 2 were obtained and delivered to SRTC after a visual inspection showed significant accumulation. Portions of this sample were analyzed by destructive and non-destructive methods to determine the chemical constituents of the solid. Results of these analyses indicate a chemical composition consisted of sodium oxalate (65%), the sulfide enclathrated sodium aluminosilicate (30%), and iron oxide (5%). The recommended method of cleaning would dissolve the sodium oxalate component of the solid deposits in hot water. The insoluble iron oxide and sodium aluminosilicate components could then be filtered.

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Approval:



1-30-98

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Date



2-1-98

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